Thermo Scientific

TRACE GC Ultra

Gas Chromatograph

Maintenance and Troubleshooting Manual

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Home

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Declaration

Manufacturer: *Thermo Fisher Scientific*

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

- installation
- re-calibration
- changes and repairs

have been carried out by authorized personnel and if:

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

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

Contents

About This Manual

Overview

This manual reports information and operating sequences for the maintenance and the troubleshooting of the TRACE GC Ultra and its components.

This manual is organized as follows:

Chapter 1, *Preventive Maintenance*, gives a schedule for preventive maintenance of your TRACE GC Ultra.

Chapter 2, *Maintaining a Split/Splitless Injector*, reports information and maintenance sequences for the Split/Splitless injector.

Chapter 3, *Maintaining a Large Volume Splitless Injector*, reports information and maintenance sequences for the Large Volume Splitless injector

Chapter 4, *Maintaining a Cold On-Column Injector for Standard Automatic Injections*, reports information and maintenance sequences for the Cold On-Column injector for TriPlus autosampler.

Chapter 5, *Maintaining a Cold On-Column Injector Direct Narrow-Bore*, reports information and maintenance sequences for the Cold On-Column injector for direct injection into capillary column with the TriPlus autosampler.

Chapter 6, *Maintaining a Cold On-Column Injector*, reports information and maintenance sequences for the Cold On-Column injector.

Chapter 7, *Maintaining a Packed Column Injector*, reports information and maintenance sequences for the Packed Column injector.

Chapter 8, *Maintaining a Purged Packed Column Injector*, reports information and maintenance sequences for the Purged Packed Column injector.

Chapter 9, *Maintaining a PTV Injector*, reports information and maintenance sequences for the Programmable Temperature of Vaporization injector.

Chapter 10, *Ensuring Tightness*, reports instructions for identifying and correcting leaks.

Chapter 11, *Maintaining a FID*, reports information and maintenance sequences for the Flame Ionization (FID) detector.

Chapter 12, *Maintaining an Electron Capture Detector*, reports information and maintenance sequences for the Electron Capture (ECD) detector.

Chapter 13, *Maintaining a Nitrogen Phosphorus Detector*, reports information and maintenance sequences for the Nitrogen Phosphorus (NPD) detector.

Chapter 14, *Maintaining a Flame Photometric Detector*, reports information and maintenance sequences for the Flame Photometric (FPD) detector.

Chapter 15, *Maintaining a Photoionization Detector*, reports information and maintenance sequences for the Photoionization (PID) detector.

Chapter 16, *Maintaining a Thermal Conductivity Detector*, reports information and maintenance sequences for the Thermal Conductivity (TCD) detector.

Chapter 17, *Maintaining Detectors in Stack Configuration*, reports information and maintenance sequences for detectors if installed in stack configuration.

Chapter 18, *Analytical Troubleshooting*, is a guide to troubleshoot the instrument when its analytical response indicates a problem.

Appendix A, *Reagents Safety Information*, reports chemical safety information concerning reagents used in the operating sequences.

Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices worldwide. Use the *Reader Survey* in this section to give us feedback on this manual and help us improve the quality of our documentation.

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

Conventions Used in This Manual

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

Bold	Bold text indicates names of windows, menus, dialog boxes, buttons, 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 to indicate text the user should enter with the keyboard.
`Monospace Bold`	Monospace Bold indicates messages or prompts 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**.
 **CAUTION**	This symbol alerts you to an action or procedure that, if performed improperly, could damage the instrument.
 **NOTE**	This symbol alerts you to important information related to the text in the previous paragraph.
 **WARNING!**	This symbol alerts you to an action or procedure that, if performed improperly, 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 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 procedure that must NOT be performed by the user. A Thermo Fisher Scientific authorized Customer Support Engineer must perform this procedure.

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 procedure.

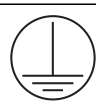
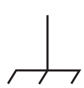
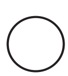
This symbol indicates the user must wear protective shoes when performing the procedure.

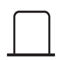
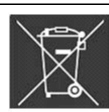
This symbol indicates the user must wear protective clothing when performing the procedure.

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

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.

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)
	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 Equipments (WEEE) placed on the european market after August, 13, 2005.

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.

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 *Performing a Manual System Leak Check* operating procedure on page 66 of the Operating Manual.
- 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 1010[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).

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

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

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 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.

1

Preventive Maintenance

Chapter at a Glance…

Suggested Maintenance Schedule

Preventive maintenance keeps your instruments running at peak performance. The recommended maintenance schedule shown in Table 1-1 on page 23 is based on a GC running multiple samples in an 8-hour day. You may need to adapt the schedule to your situation depending on:

- the number of analyses per day
- how clean the samples are
- the condition of the autosampler syringe needle
- environmental conditions such as dirt and ambient temperature

Septa degrade over time. Different types will degrade at varying rates. Factors affecting septa life include:

- number of samples injected
- injector temperature
- condition of the syringe needle

Our recommendations for septa replacement are based on 200 injections in a Thermo Fisher Scientific standard septum. Septa from other manufacturers may show different rates of degradation.

Make sure you not only perform each scheduled item, but that you record it as well, along with any observation about instrument performance. Your records and notes can be invaluable when trying to trace a service problem.

In addition to the routine and preventive maintenance that you perform, we recommend annual inspections by Thermo Fisher Scientific customer service engineers.
These yearly checkups ensure that your instrument remains in peak condition.

Table 1-1. Standard Maintenance Schedule

Frequency	Task
Daily	Run performance check standard as required by method. Run calibration curve as required by method. Note gas pressure when the weekly pressure check shows it to be below 5000 kPa (50 bar or 750 psig).
Weekly	Change injector liner. Check pressure of gas cylinders. Replace the cylinder when its pressure drops below 3500 kPa (35 bar or 500 psig). Change S/SL septum. Change PTV septum. Change PKD septum. Change PPKD septum. Change OC septum. Perform the Leak Test procedure.
Monthly	Check for leaks from the primary gas supply to the GC.
Semiannually	Regenerate the S/SL inlet vent line trap. Regenerate the PTV inlet vent line trap. Replace FID jet. Clean FPD windows and stainless steel mirror. Clean PID cell and UV lamp window. Replace NPD source and clean collector. Clean or replace ECD collector. Recondition or replace internal and external traps.

Table 1-1. Standard Maintenance Schedule

Annually	Clean FID collector. Replace PID lamp. Regenerate the active carbon filter mounted on the carrier gas line Regenerate the active carbon filter mounted on the split line.
Biannually	Replace FPD photomultiplier. Clean or replace ECD source [1]
As required by local law	Perform wipe test on ECD[1]

1. Only a licensed laboratory can perform this operation

2

Maintaining a Split/Splitless Injector

Chapter at a Glance…

Operating Sequences

Split/Splitless Injector

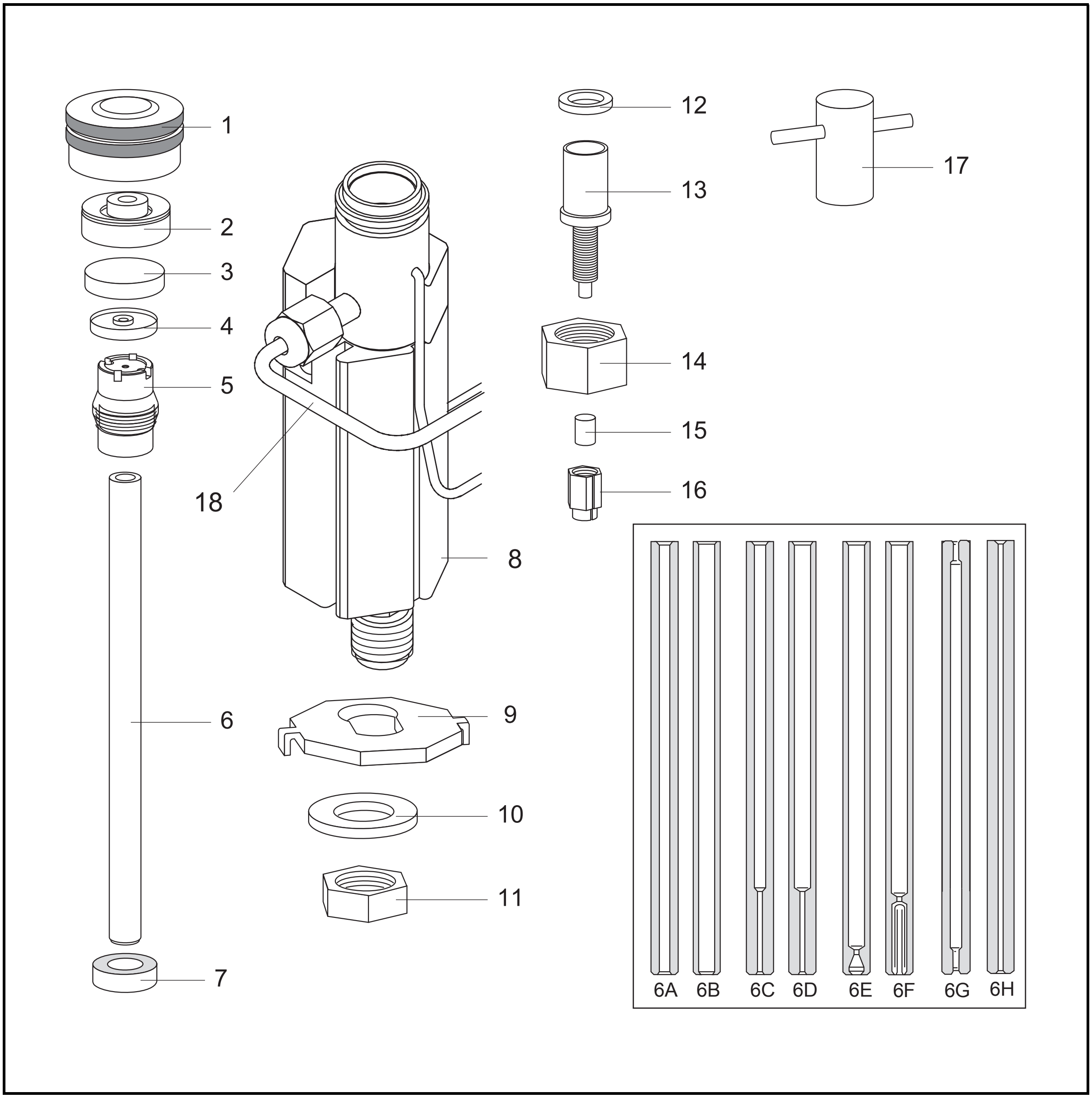


Table 2-1. Split/Splitless Injector parts identification table

No	Description	P/N
1	Septum cap	350 010 50
	Septum cap for HS 2000	347 500 04
2	Septum holder	233 030 15
3	Septum	313 032 11
4	Septum support	350 054 33
5	Liner cap	290 042 88
6A	Liner, split, 3 mm ID	453 200 31
6B	Liner, split, 5 mm ID	453 200 30
6C	Liner, splitless, 3 mm ID	453 200 32
6D	Liner, splitless, 5 mm ID	453 200 33
6E	Liner for wide bore columns	453 003 10
6F	Liner for polar solvents	453 003 20
6G	Liner double gooseneck for nano-volumes injection	45322080
6H	Liner 0.8 mm ID	45352083
7	Liner seal	290 334 06
8	Injector body	
9	Anti-rotation plate	
10	Washer	
11	Retaining nut	
12	Silver seal	290 336 29
13	Terminal fitting for capillary columns	347 054 51
14	Nut for terminal fitting	350 221 25
15	Graphite ferrule for 0.2 mm ID column	290 134 89
	Graphite ferrule for 0.25 mm ID column	290 134 88
	Graphite ferrule for 0.32 mm ID column	290 134 87
	Graphite ferrule for 0.53 mm ID column	290 134 86
16	Fixing nut for column	350 324 23

Table 2-1. Split/Splitless Injector parts identification table

17	Liner cap removal tool	205 070 10
18	Kit Assy Split line 1/8-in.	190 507 00

Split/Splitless Injector Maintenance

The S/SL (Split/Splitless) injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel.
In order to operate at peak performances, the injector requires maintenance from the user. This maintenance includes:

- the replacement of the septum
- the cleaning or replacement of the liner
- the replacement of the split line tubing

When replacing the septum

The septum should be replaced at least after every 100-200 injections, or every time a problem related to septum damage or wear occurs (refer to Chapter 19 *Analytical Troubleshooting* for more information). It is a good practice to change the septum with a new one every time the liner is replaced.

CAUTION **The Injector temperature, when high, it makes a prematurely "aging" of the septum. It is good practice to replace the septum of the S/SL injector every week independently on the number of injections done.**

When cleaning or replacing the liner

The liner must be replaced periodically, depending on the number of injections performed and the characteristics of the samples injected. Typical symptoms will indicate that the liner must be replaced (refer to Chapter 19 *Analytical Troubleshooting*). The most common is the appearance of tailing peaks in the chromatogram, particularly for polar compounds.

It is good practice to replace the septum every time the liner is replaced.

You can replace the liner with a new one or clean the liner and reinstall it. Table 2-2, *S/SL available liners*, shows the available types of liners and the indications for the correct choice.

This operating sequence describes how to:

- Remove the liner and the septum
- Clean and reinstall the liner

Table 2-2. S/SL available liners

Application	P/N
Split injections, 3 mm	453 200 31
Split injections, 5 mm	453 200 30
Splitless injections, 3 mm	453 200 32
Splitless injections, 5 mm	453 200 33
Liner for direct injections into wide bore column	453 003 10
Laminar cup liner for split injections at high split flow rates or for the most polar solvents.	453 003 20

If the glass liner breaks inside the injector

When replacing or removing a glass liner, it might break inside the injector. In this case the broken parts of the liner must be removed from the injector, including the glass splinters that might fall into the lower part of the vaporization chamber. Refer to *Replace a Broken Liner* Operating Sequence on page 40 for instructions.

When replacing the split line tubing

Replace the split line tubing when strong contamination or block has been verifyied. The Kit assy split line 1/8” (PN 190 507 00) is required.

OPERATING SEQUENCE

Replace the Septum

Materials needed

- Non metallic sharp tool
- Septum (P/N 313 032 11)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

OVEN		
Temp	50	50 <
Initial time		2
Ramp 1		10.0

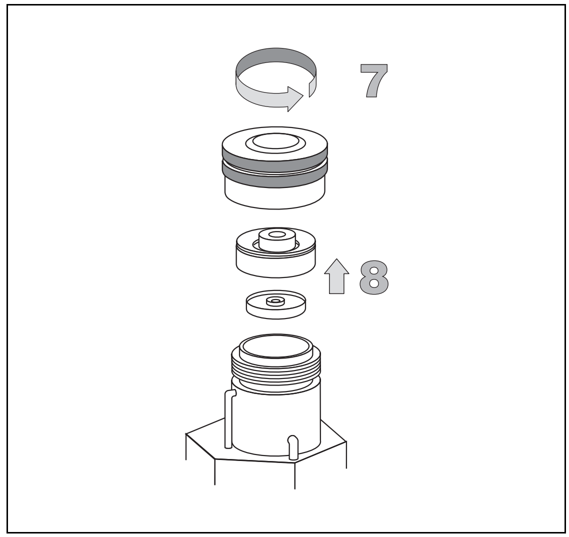
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

RIGHT INLET (S/SL)		
Temp	250	250 <
Pressure	100	100
Mode		split

5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

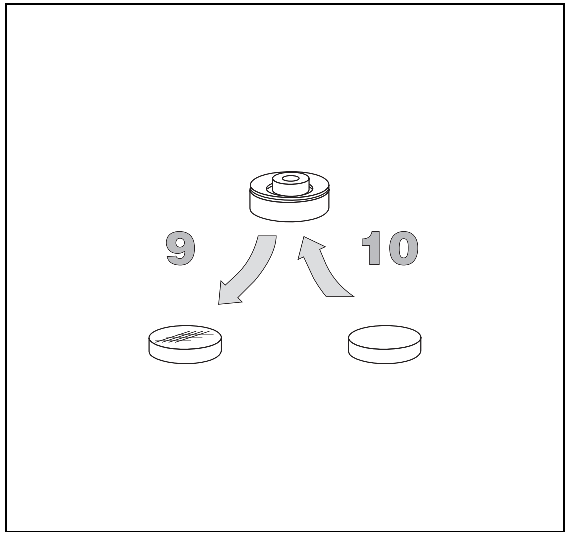
```
      RIGHT CARRIER (S/SL)
Pressure             30.0   30.0
Col. flow            3.00        <
Lin. Veloc.               (60.9)
```

7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



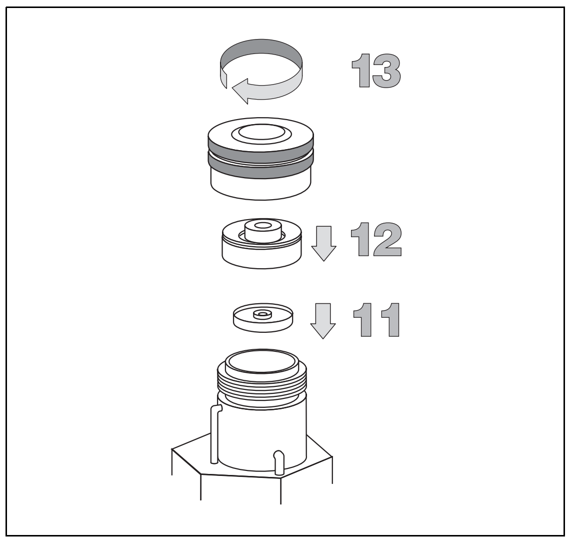
9. Remove the septum from the septum holder (use non-metallic tools)
10. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



11. Clean the septum support from possible fragments left by the septum and reinsert it into the injector
12. Place the septum holder on the top of the septum support.
13. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



14. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
15. Press **OVEN**, scroll to `Temp` and press **ON**.

16. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
17. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Clean or Replace the Liner

Materials needed

- Liner cap removal tool (P/N 205 070 10)
- Glass liner (see Table 2-2, on page 29)
- Graphite seal (P/N 290 334 06)
- Ultrasonic bath
- Methanol/acetone mixture (1:1)
- Non metallic sharp tool
- Septum (P/N 313 032 11)
- Tweezers

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

PRECAUTIONS

WARNING! When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                 OVEN
Temp                      50       50 <
Initial time                        2
Ramp 1                           10.0
```

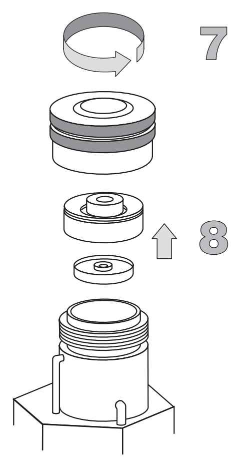
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

```
         RIGHT INLET (S/SL)
Temp                    250    250 <
Pressure                100    100
Mode                         split
```

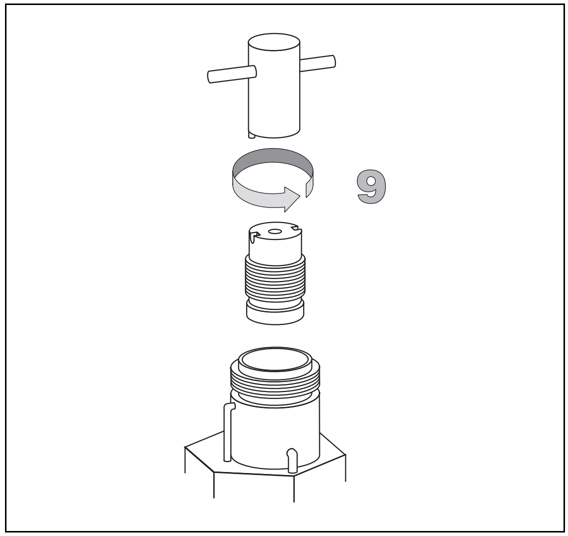
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
        RIGHT CARRIER (S/SL)
Pressure               30.0   30.0
Col. flow              3.00          <
Lin. Veloc.                 (60.9)
```

7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



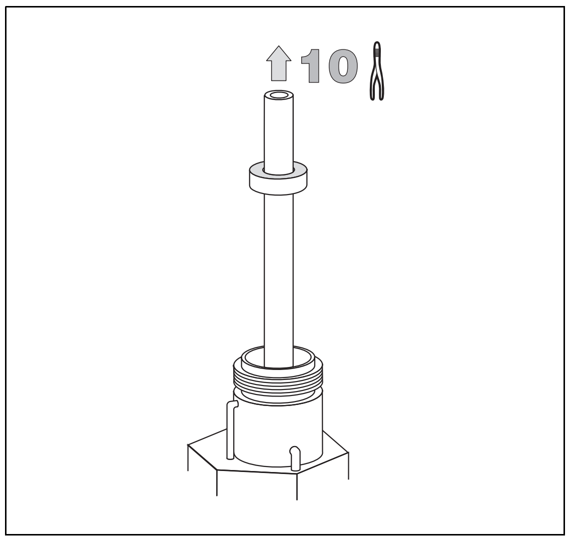
9. Use the liner cap wrench provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the liner with the graphite seal.

CAUTION **Be careful not to break the glass liner inside the injector when removing it. Glass splinters might fall into the lower part of the vaporization chamber.**

If the liner breaks, refer to the *Replace a Broken Liner* Operating Sequence on page 40.



11. If you are going to use a new liner, go to step 14. If you are going to clean the liner, put the dirty liner into an ultrasonic bath filled with a methanol/acetone mixture (1:1) and sonicate it for about half an hour.

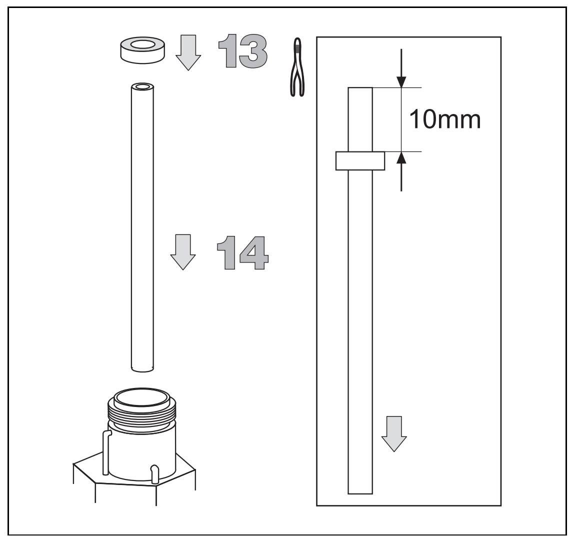
12. Using tweezers, remove the liner from the bath and dry it with compressed clean air.

NOTE For trace analysis, you should pre-treat the liner with a suitable silylating reagent prior to re-inserting it into the injector.

PRECAUTIONS

13. Holding the new (or cleaned) liner with tweezers, place a graphite seal over the liner, making sure to leave a distance of about 10 mm between the seal and the liner end.

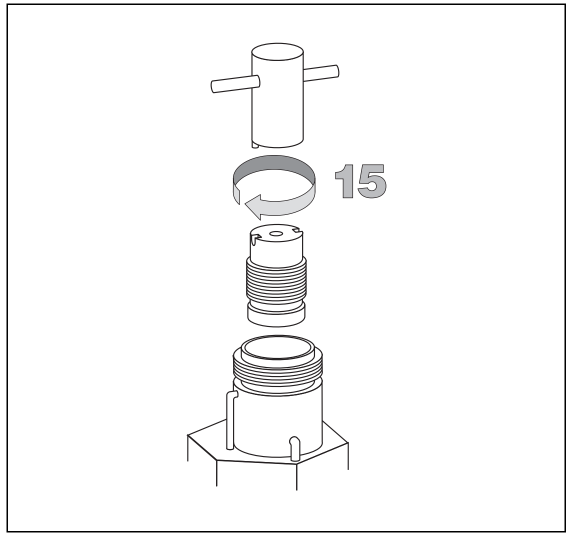
14. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting.



CAUTION **Make sure you insert the bevelled end of the liner towards the bottom of the injector.**

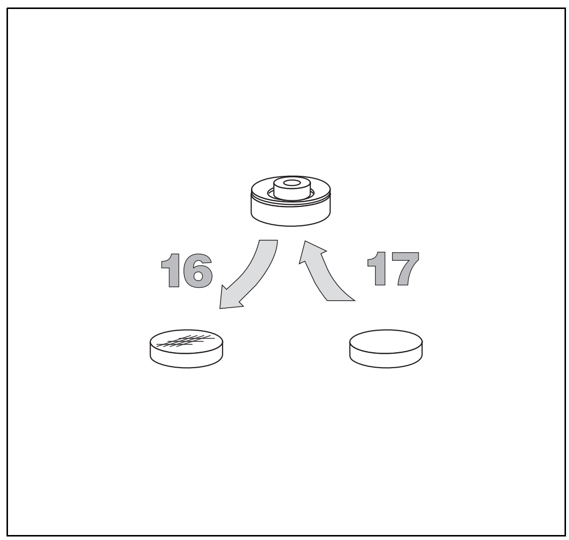
CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

15. Tighten the liner cap using the liner cap wrench provided with the GC.



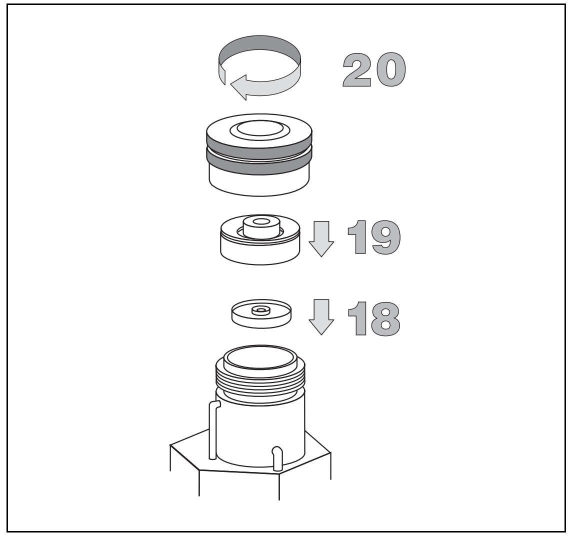
16. Remove the septum from the septum holder (use non-metallic tools).
17. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



18. Clean the septum support from possible fragments left by the septum and reinsert it into the injector
19. Place the septum holder on the top of the septum support.
20. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



21. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
22. Press **OVEN**, scroll to `Temp` and press **ON**.
23. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
24. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace a Broken Liner

Materials needed

- Liner cap removal tool (P/N 205 070 10)
- Glass liner (see Table 2-2 on page 29)
- Tweezers

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                 OVEN
Temp                 50       50 <
Initial time                   2
Ramp 1                      10.0
```

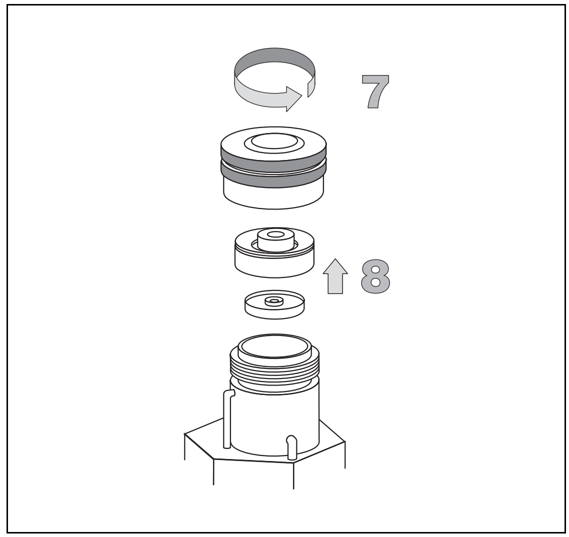
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

```
        RIGHT INLET (S/SL)
Temp                250      250 <
Pressure            100      100
Mode                       split
```

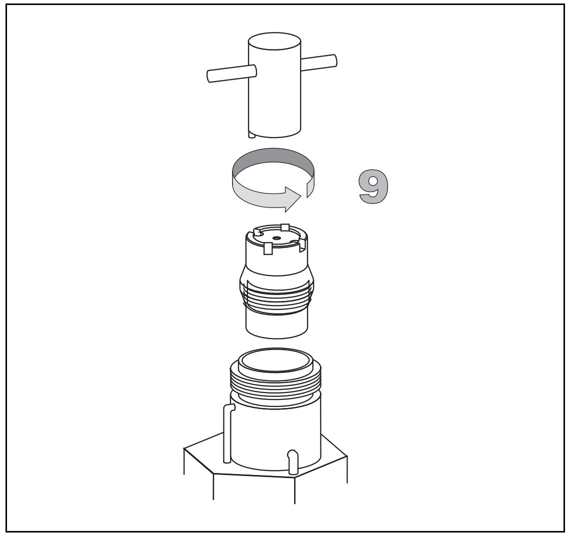
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (S/SL)
Pressure             30.0   30.0
Col. flow            3.00         <
Lin. Veloc.                (60.9)
```

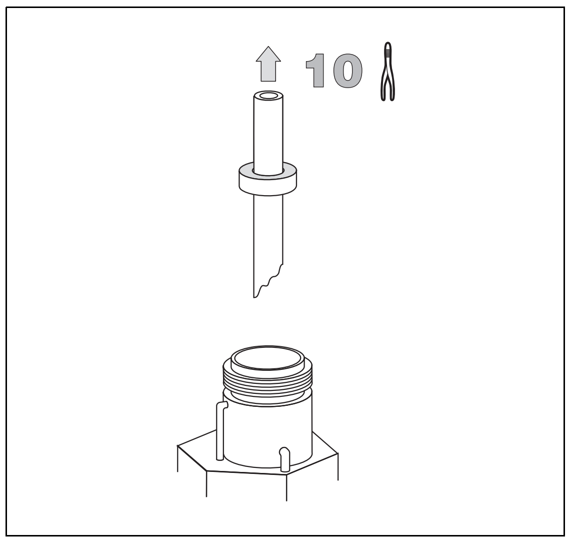
7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



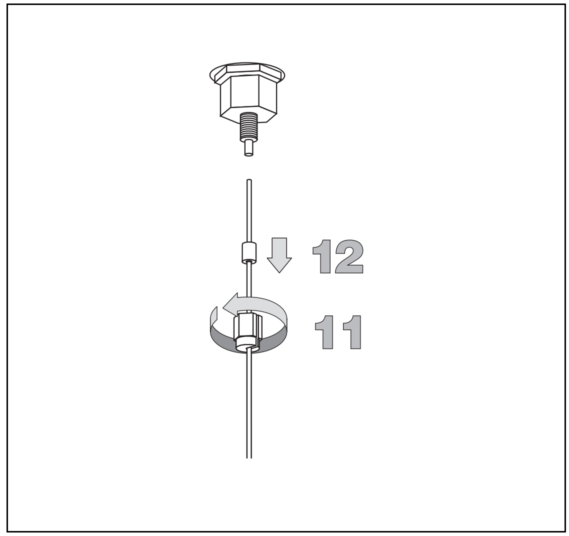
9. Use the liner cap wrench provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the upper part of the broken liner (with the graphite seal) from the injector.



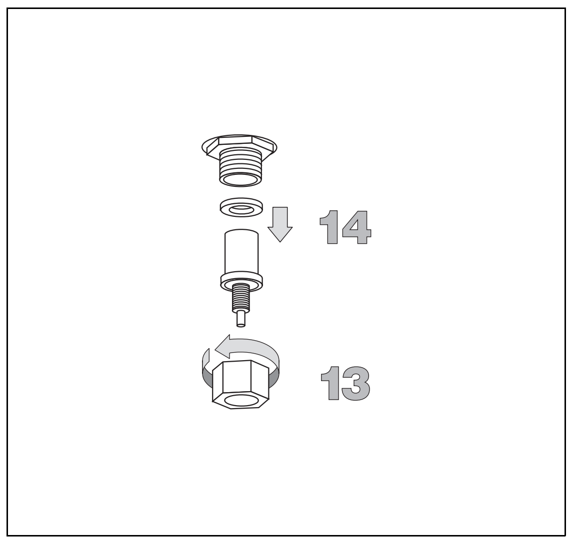
11. Unscrew the nut that retains the analytical column.
12. Remove the analytical column with its ferrule.



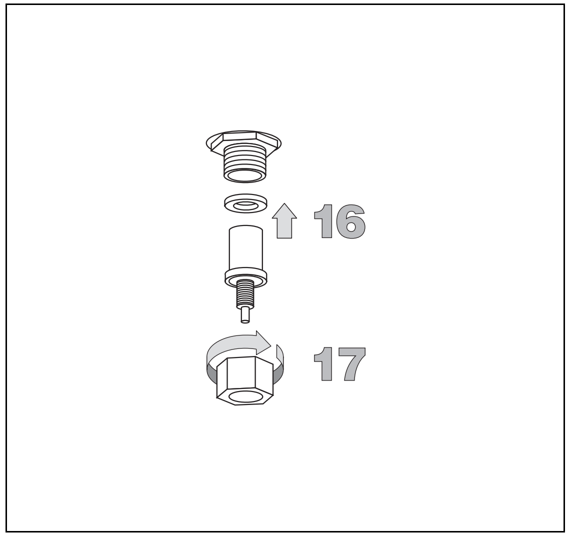
13. Unscrew the retaining nut at the bottom of the injector.
14. Remove the terminal fitting with its silver seal.

CAUTION **Glass splinters from the broken liner will fall from the injector.**

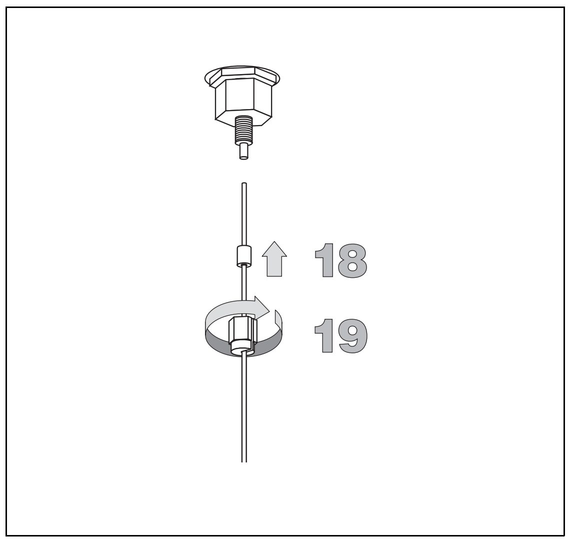
15. With the aid of a pipe cleaner, remove the possible glass fragments from the vaporization chamber.



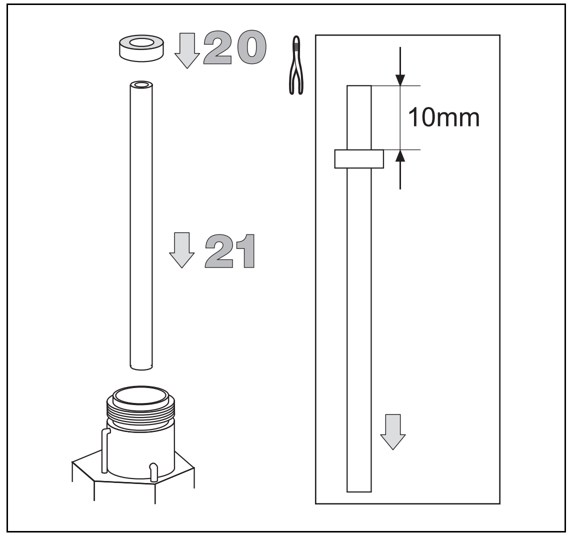
16. Reinsert the silver seal and the terminal fitting.
17. Tighten the nut that retains the terminal fitting.



18. Insert the analytical column with its ferrule into the bottom of the injector in its previous position (for instructions, refer to the TRACE™ GC *Operating Manual*).
19. Tighten the M4 retaining nut to hold the column in place.

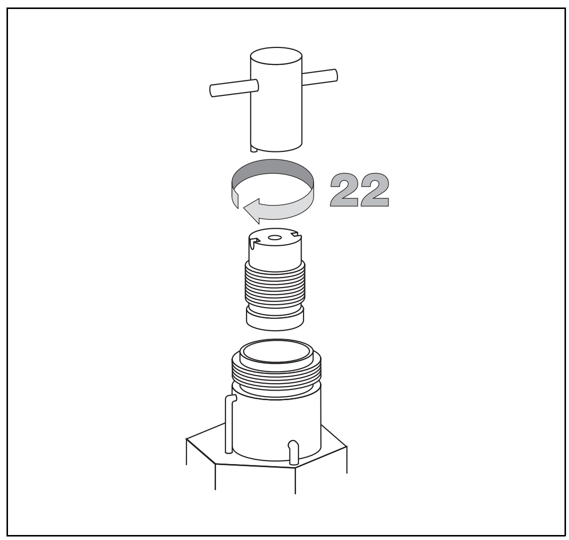


20. Holding the new liner with tweezers, place a graphite seal over the liner, making sure to leave a distance of about 10 mm between the seal and the liner end.
21. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting. Make sure you insert the bevelled end of the liner towards the bottom of the injector.



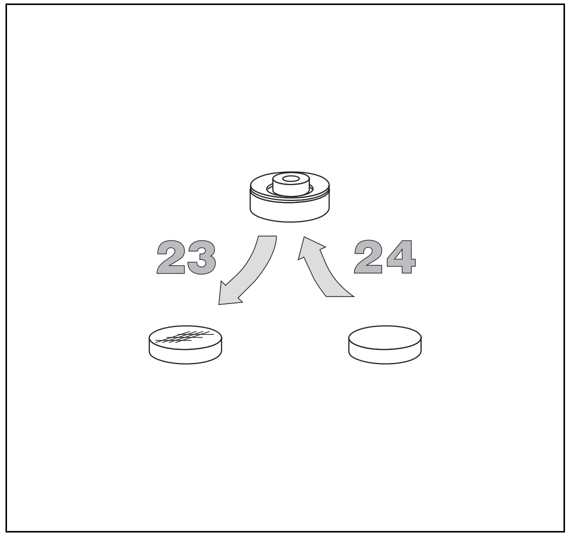
CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

22. Tighten the liner cap using the liner cap wrench provided with the GC.



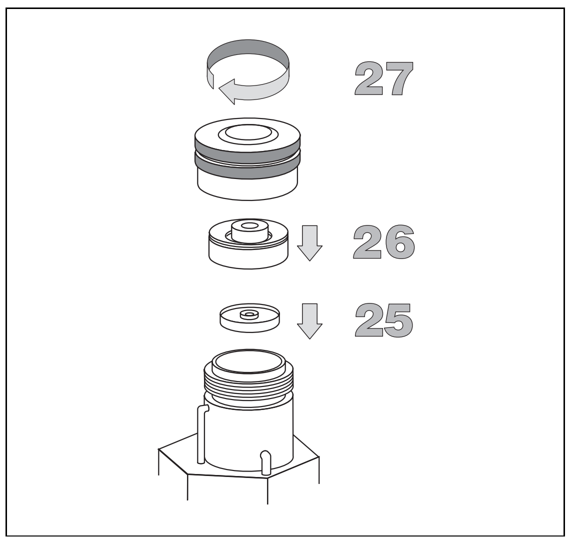
23. Remove the septum from the septum holder (use non-metallic tools).
24. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



25. Clean the septum support from possible fragments left by the septum and reinsert it into the injector
26. Place the septum holder on the top of the septum support.
27. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



28. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
29. Press **OVEN**, scroll to `Temp` and press **ON**.

30. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.

31. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace the Split Line Tubing

Materials needed

- 7/16” wrenches
- Two 8-10 mm wrences
- Kit Assy Split line 1/8” (PN 190 507 00)
  - Split line tubing 1/8”
  - Brass front/back ferrule for 1/8” tubing
  - 5/16” brass nut
  - 1.6 mm ID brass ferrule
  - M6x0.75 mm (6MB) nut

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
              OVEN
Temp                  50      50 <
Initial time                   2
Ramp 1                      10.0
```

3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.

```
        RIGHT INLET (S/SL)
Temp                 250     250 <
Pressure             100     100
Mode                       split
```

4. Scroll to `Temp` and press **OFF**.

5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.

6. Scroll to `Pressure` and press **OFF**.

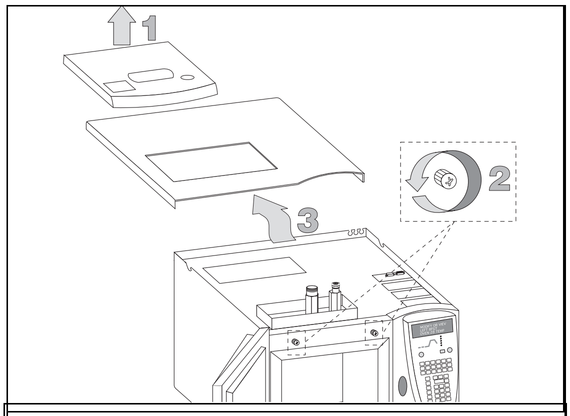
RIGHT CARRIER (S/SL)			
Pressure	30.0	30.0	
Col. flow	3.00		<
Lin. Veloc.		(60.9)	

7. Remove the GC top cover.

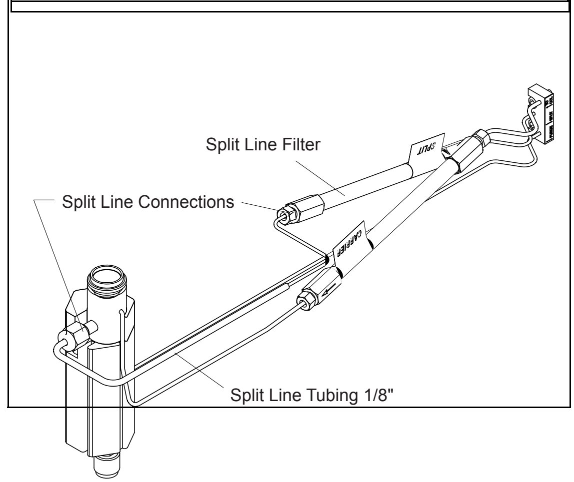
   a. (1) Lift the detector cover off the GC top cover.

   b. (2) Open the oven door and unscrew the two top cover fastening screws.

   c. (3) Push the cover back about 1 cm and lift it up and off the GC.



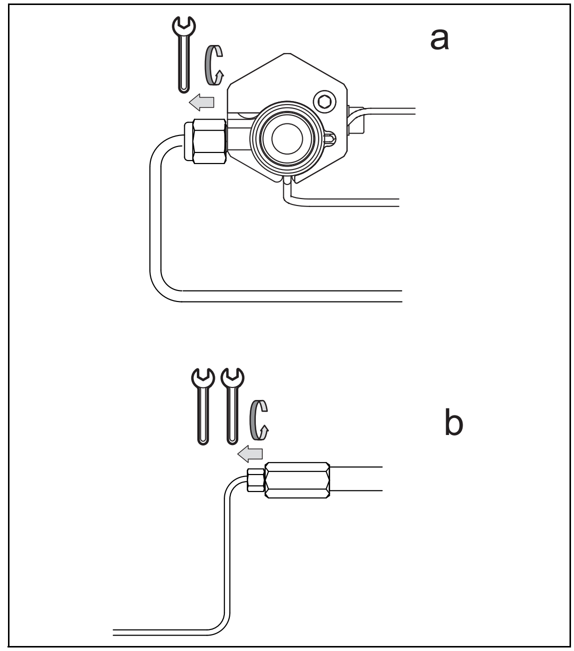
8. Look for the split line tubing connections.



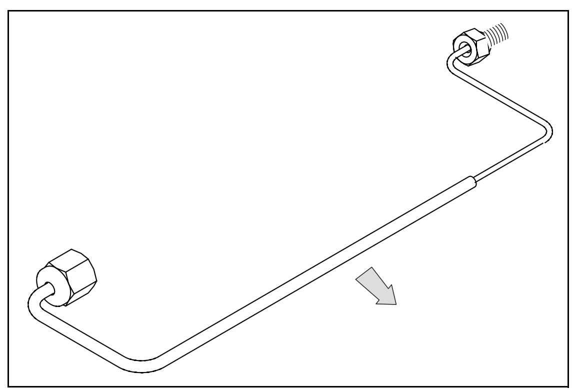
9. Disconnect the split line tubing from the injector body and from the split line filter.

   a. Using the 7/16” wrench, unscrew and disconnect the split line tubing from the injection body.

   b. Using two 8-10 mm wrenches, unscrew and disconnect the split line tubing from the split line filter.



10. Remove the split line tubing from the GC.



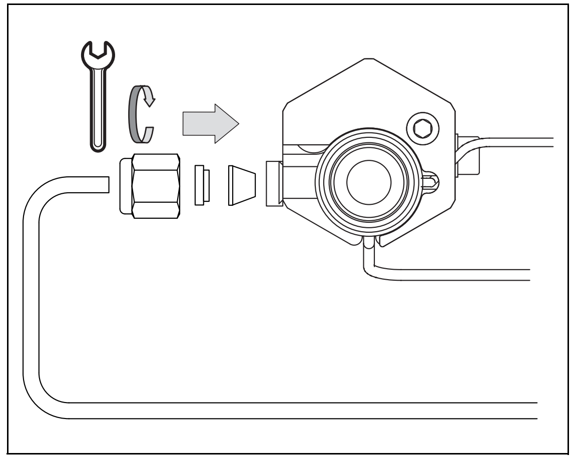
11. Connect the new split line tubing to the injector body.

    a. On the end of the new split line tubing having the greater diameter, slide the 5/16” brass nut and the brass front/back ferrule.

    b. Fully insert the tubing into the fitting on the injector body and rotate the nut finger-tight.

    c. Mark the nut at the 12 o’clock position.

    d. While holding the fitting body steady, tighten the nut tree-quarters turn to the 9 o’clock position.



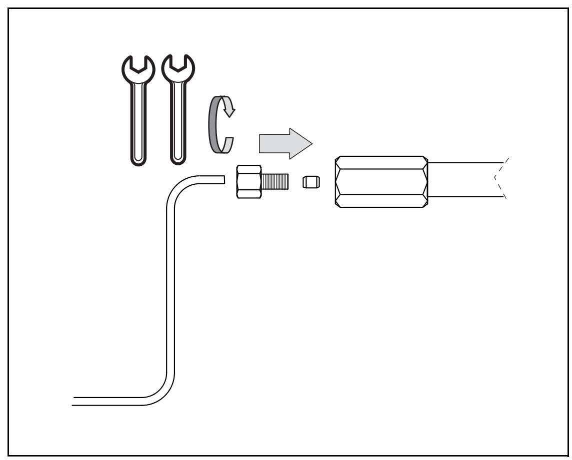
12. Connect the new split line tubing to the split line filter

    a. On the end of the new split line tubing having the lower diameter, slide the nut 6MB nut and the 1.6 mm ID brass ferrule.

    b. Insert the tubing into the outlet of the split line filter.

    c. Finger-tighten the 6 MB nut until it start to grip the filter.

    d. Use two 8-10 mm wrench to tighten the nut. Use no more pressure than is necessary to obtain a good seal.



13. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.

14. Press **OVEN**, scroll to `Temp` and press **ON**.

15. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
16. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.If leaks are detected, check the critical connections with an electronic leak detector to locate the possible leaks.
17. When the injector is free of leaks, reinstall the GC top cover.
18. Set the normal injector and GC working condition.

3

Maintaining a Large Volume Splitless Injector

Chapter at a Glance...

Operating Sequences

Large Volume Splitless Injector

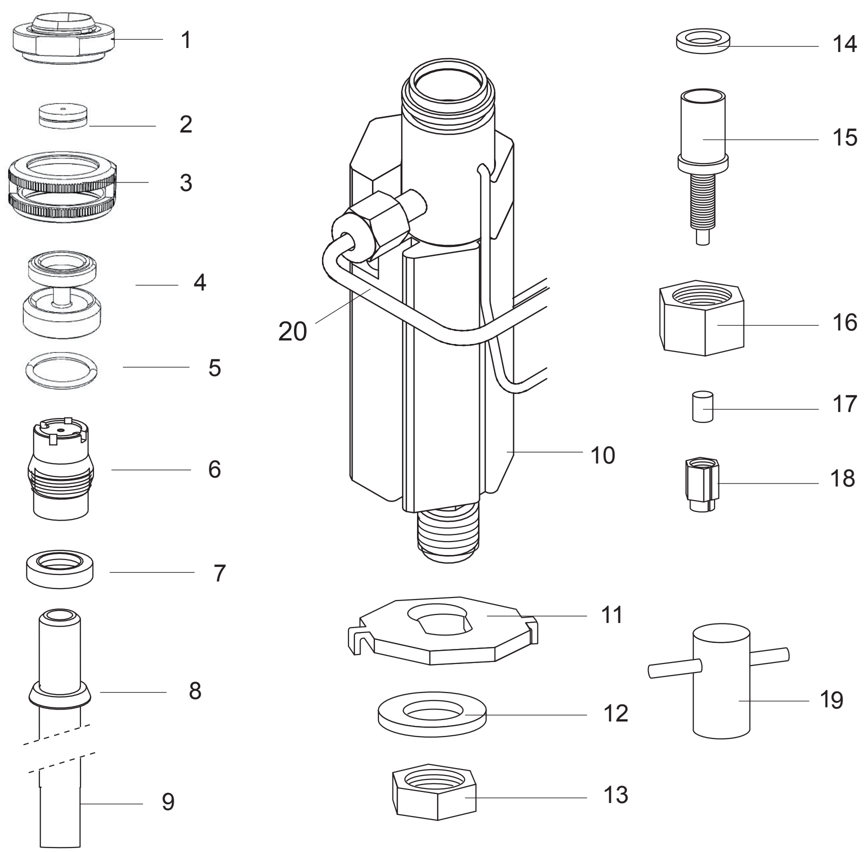


Table 3-1. Large Volume Splitless Injector spare parts

	Description	Qty.	P/N
1	Septum Cap		3500 13 25
2	11-mm OD BTO Septum with center guide	10	313 032 35
3	Ring Nut		350 013 24
4	Septum Holder		247 520 26
5	O-ring		290 013 17
6	Liner Cap		290 042 90
7	Metal Ring (already installed on the liner; see 9A and 9B)		---
8	Vespel liner seal (already installed on the liner; see 9A and 9B)		---
9A	Glass liner packed with deactivated glass wool	2	453 020 65
9B	Deactivated laminar liner (optional)	2	453 220 67
10	Injector body		
11	Anti-rotation plate		
12	Washer		
13	Retaining nut		
14	Silver seal	10	290 336 29
15	Terminal fitting for cap. columns		347 054 51
16	Nut for terminal fitting	2	350 221 25
17	Graphite ferrule for 0.2 mm ID column	2	290 134 89
17	Graphite ferrule for 0.25 mm ID column	2	290 134 88
17	Graphite ferrule for 0.32 mm ID column	2	290 134 87
17	Graphite ferrule for 0.53 mm ID column	2	290 134 86
18	Fixing nut for column	5	350 324 23
19	Liner cap removing tool		205 070 10
20	Kit Assy Split line 1/16”		190 507 10

Large Volume Splitless Injector Maintenance

The LVSL (Large Volume Splitless) injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel.
In order to operate at peak performances, the injector requires periodic maintenance from the user. This maintenance includes:

- the replacement of the septum
- the cleaning or replacement of the liner
- the replacement of the split line tubing

When replacing the septum

The septum should be replaced at least after every 200 injections, or every time a problem related to septum damage or wear occurs (refer to Chapter 19, *Analytical Troubleshooting* for more information).
It is a good practice to change the septum with a new one every time the liner is replaced.

When cleaning or replacing the liner

The liner must be replaced periodically, depending on the number of injections performed and the characteristics of the samples injected. Typical symptoms will indicate that the liner must be replaced (refer to Chapter 19, *Analytical Troubleshooting*). The most common is the appearance of tailing peaks in the chromatogram, particularly for polar compounds.

It is good practice to replace the septum every time the liner is replaced.

You can replace the liner with a new one or clean the liner and reinstall it. Table 3-2, *LVSL available liners*, shows the available types of liners and the indications for the correct choice.

This operating sequence describes how to:

- Remove the liner and the septum
- Clean and reinstall the liner

Table 3-2. LVSL available liners

Application	P/N
Glass liner packed with deactivated glass wool	453 220 65
Deactivated laminar liner (optional)	453 220 67

If the glass liner breaks inside the injector

When replacing or removing a glass liner, it might break inside the injector. In this case the broken parts of the liner must be removed from the injector, including the glass splinters that might fall into the lower part of the vaporization chamber. Refer to *Replace a Broken Liner* Operating Sequence on page 67 for instructions.

When replacing the split line tubing

Replace the split line tubing when strong contamination or block has been verifyied. The Kit Assy Split line (PN 190 507 10) is required.

OPERATING SEQUENCE

Replace the Septum

Materials needed

- Non metallic sharp tool
- Septum (P/N 313 032 41)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

OVEN		
Temp	50	50 <
Initial time		2
Ramp 1		10.0

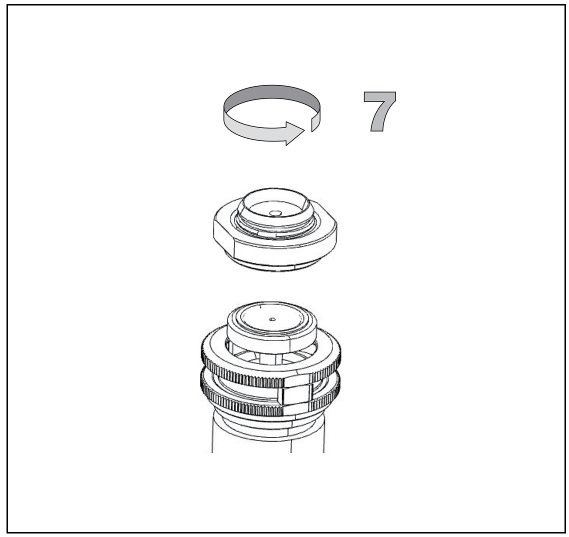
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

RIGHT INLET (S/SL)		
Temp	250	250 <
Pressure	100	100
Mode		split

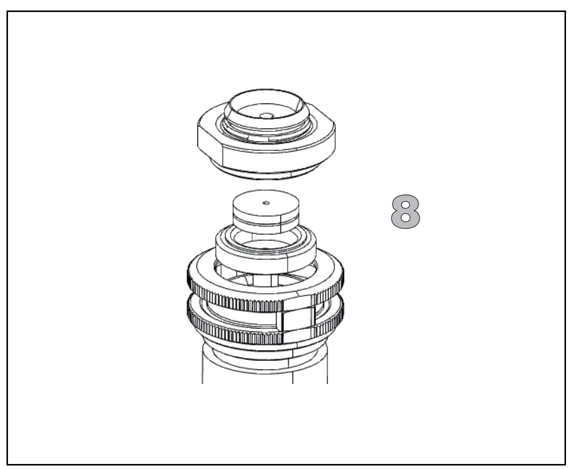
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (S/SL)
Pressure             30.0   30.0
Col. flow            3.00         <
Lin. Veloc.               (60.9)
```

7. Unscrew the injector cap.



8. Remove the septum from the septum holder (use non-metallic tools)



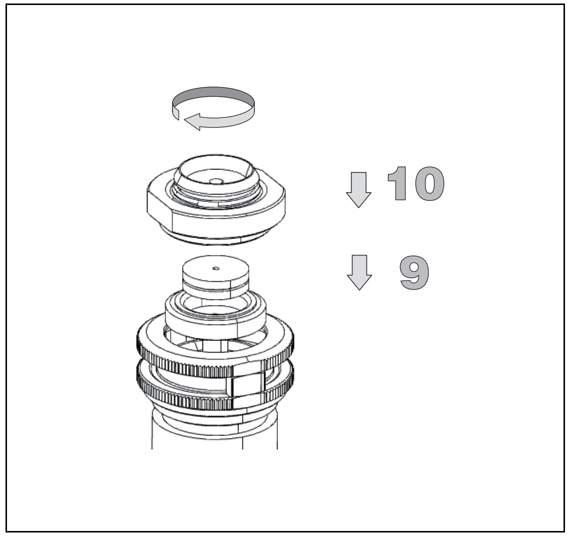
9. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**

10. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**

11. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
12. Press **OVEN**, scroll to `Temp` and press **ON**.
13. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
14. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.



OPERATING SEQUENCE

Clean or Replace the Liner

Materials needed

- Liner cap removal tool (P/N 205 070 10)
- Glass liner (see Table 3-2, on page 57)
- Ultrasonic bath
- Methanol/acetone mixture (1:1)
- Non metallic sharp tool
- Septum (P/N 313 032 41)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                OVEN
Temp                 50      50 <
Initial time                  2
Ramp 1                     10.0
```

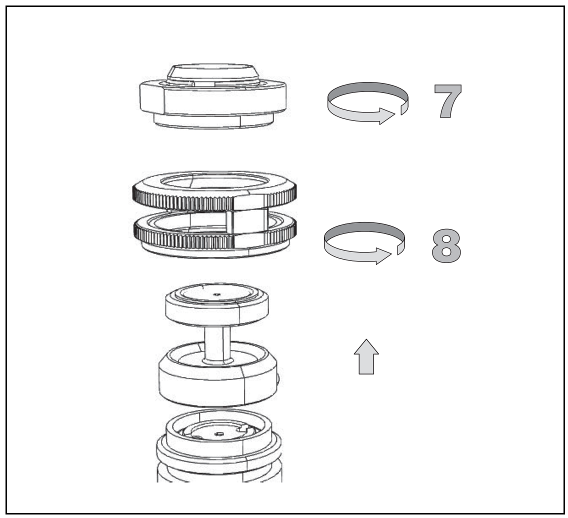
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

```
      RIGHT INLET (S/SL)
Temp                    250    250 <
Pressure                100    100
Mode                         split
```

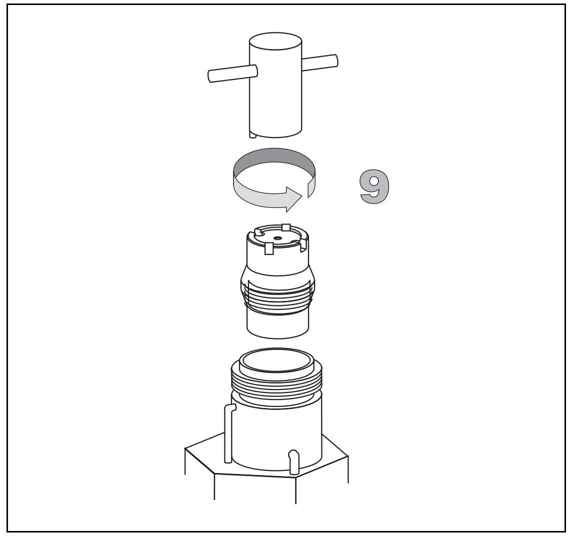
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
     RIGHT CARRIER (S/SL)
Pressure               30.0   30.0
Col. flow              3.00        <
Lin. Veloc.                 (60.9)
```

7. Unscrew the injector cap.
8. Unscrew the ring nut and remove the septum holder with the septum from the inlet port.



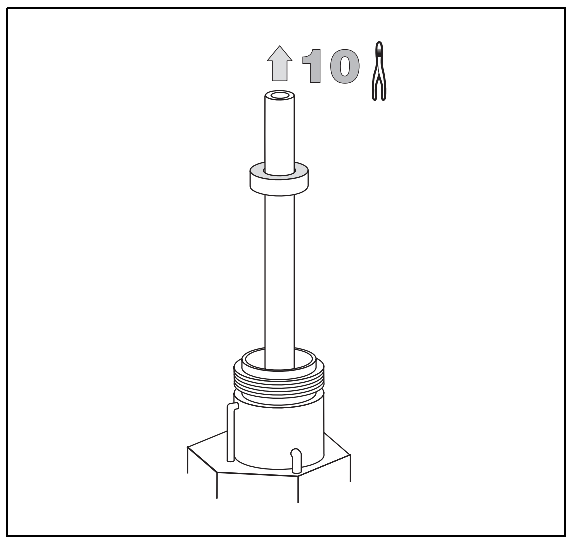
9. Use the liner cap wrench provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the liner with the graphite seal.

CAUTION **Be careful not to break the glass liner inside the injector when removing it. Glass splinters might fall into the lower part of the vaporization chamber.**

If the liner breaks, refer to the ***Replace a Broken Liner*** **Operating Sequence on page 67.**



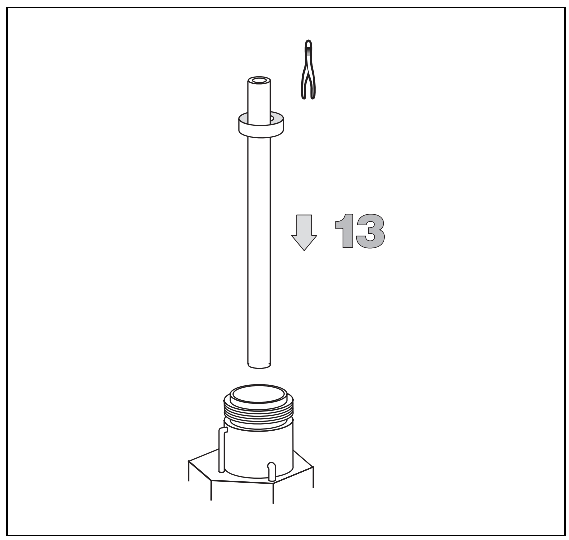
11. If you are going to use a new liner, go to step 14. If you are going to clean the liner, put the dirty liner into an ultrasonic bath filled with a methanol/acetone mixture (1:1) and sonicate it for about half an hour.

12. Using tweezers, remove the liner from the bath and dry it with compressed clean air.

NOTE For trace analysis, you should pre-treat the liner with a suitable silylating reagent prior to re-inserting it into the injector.

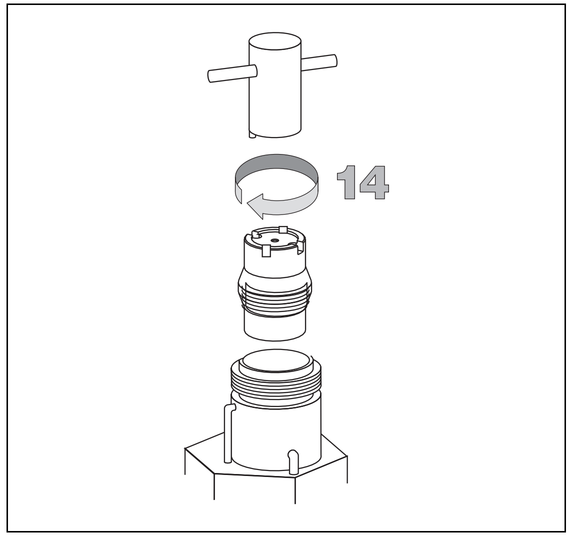
PRECAUTIONS

13. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting.



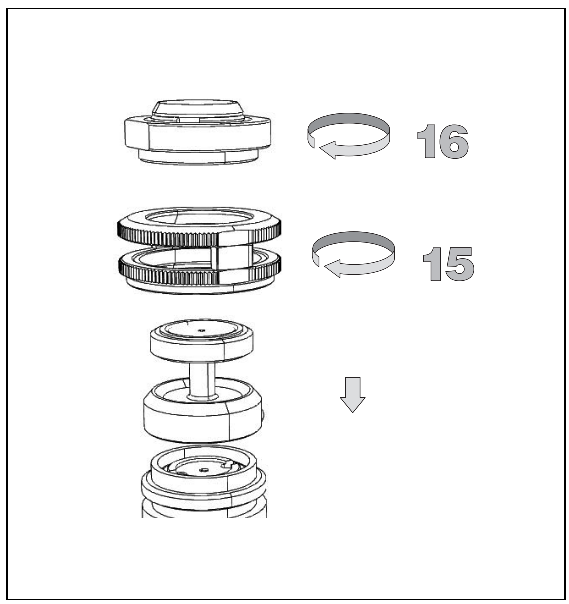
CAUTION **Make sure you insert the bevelled end of the liner towards the bottom of the injector.**

14. Tighten the liner cap using the liner cap wrench provided with the GC.



15. Place the septum holder with the septum onto the inlet port, then screw and tighten the ring nut up to the stop.
16. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



17. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
18. Press **OVEN**, scroll to `Temp` and press **ON**.
19. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
20. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace a Broken Liner

Materials needed

- Liner cap removal tool (P/N 205 070 10)
- Glass liner (see Table 3-2 on page 57)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
               OVEN
Temp                   50      50 <
Initial time                   2
Ramp 1                      10.0
```

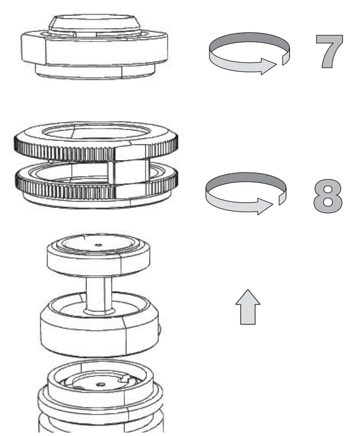
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

```
        RIGHT INLET (S/SL)
Temp                  250     250 <
Pressure              100     100
Mode                        split
```

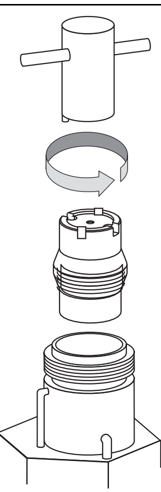
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (S/SL)
Pressure            30.0   30.0
Col. flow           3.00        <
Lin. Veloc.              (60.9)
```

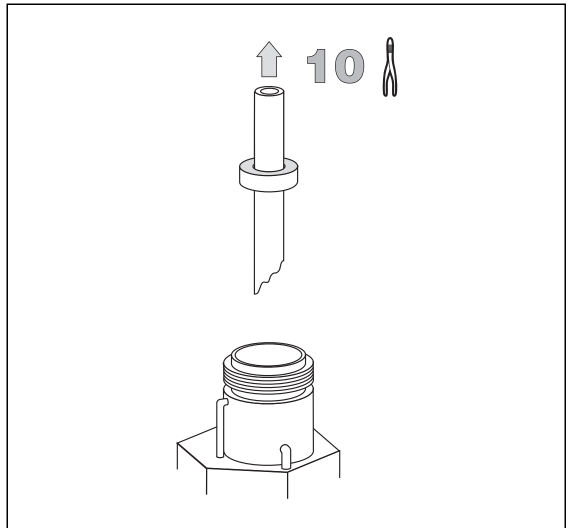
7. Unscrew the injector cap.
8. Unscrew the ring nut and remove the septum holder with the septum from the inlet port.



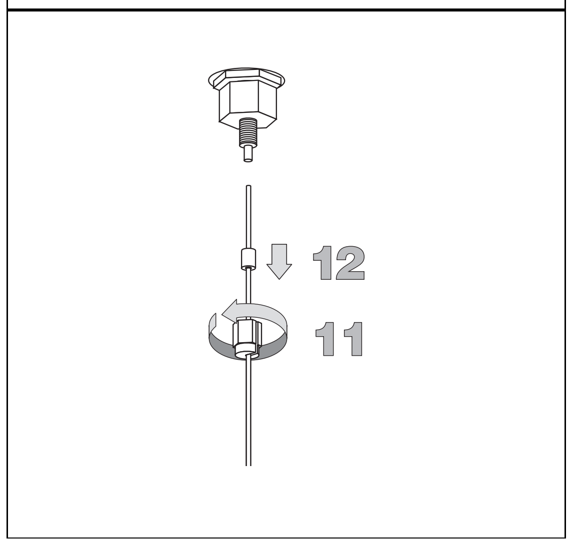
9. Use the liner cap wrench provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the upper part of the broken liner (with the graphite seal) from the injector.



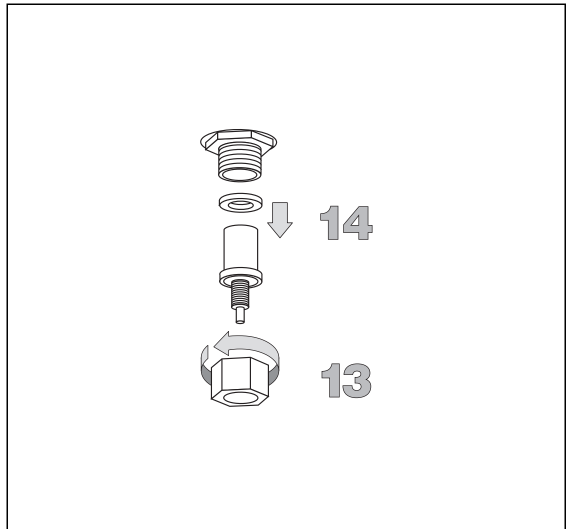
11. Unscrew the nut that retains the analytical column.
12. Remove the analytical column with its ferrule.



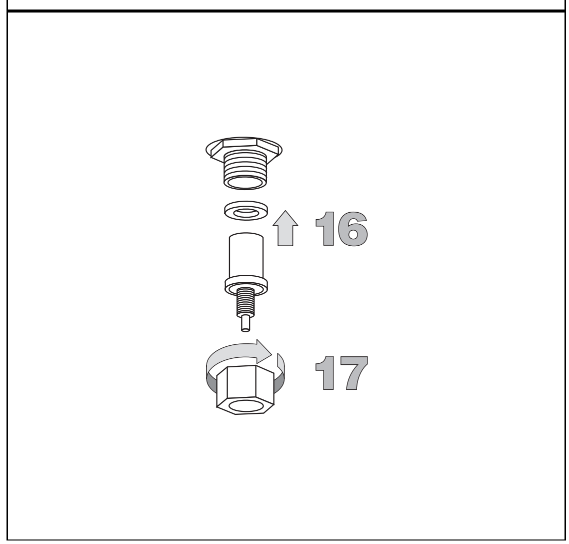
13. Unscrew the retaining nut at the bottom of the injector.
14. Remove the terminal fitting with its silver seal.

CAUTION **Glass splinters from the broken liner will fall from the injector.**

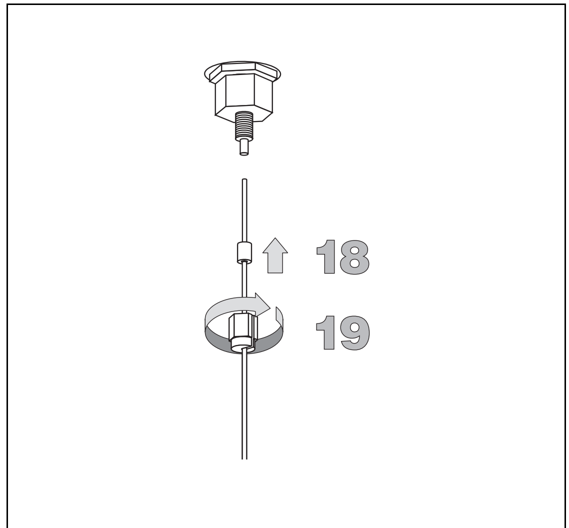
15. With the aid of a pipe cleaner, remove the possible glass fragments from the vaporization chamber.



16. Reinsert the silver seal and the terminal fitting.
17. Tighten the nut that retains the terminal fitting.

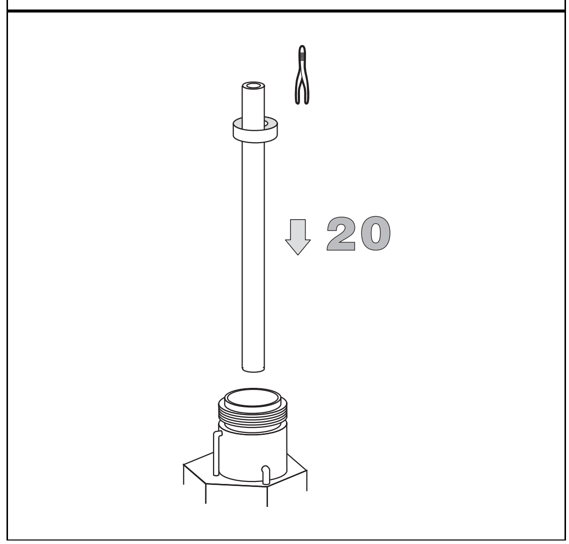


18. Insert the analytical column with its ferrule into the bottom of the injector in its previous position (for instructions, refer to the TRACE™ GC *Operating Manual*).
19. Tighten the M4 retaining nut to hold the column in place.

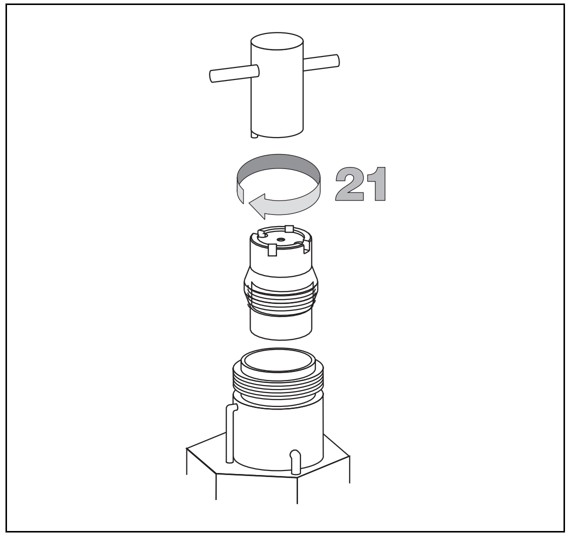


20. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting. Make sure you insert the bevelled end of the liner towards the bottom of the injector.

CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

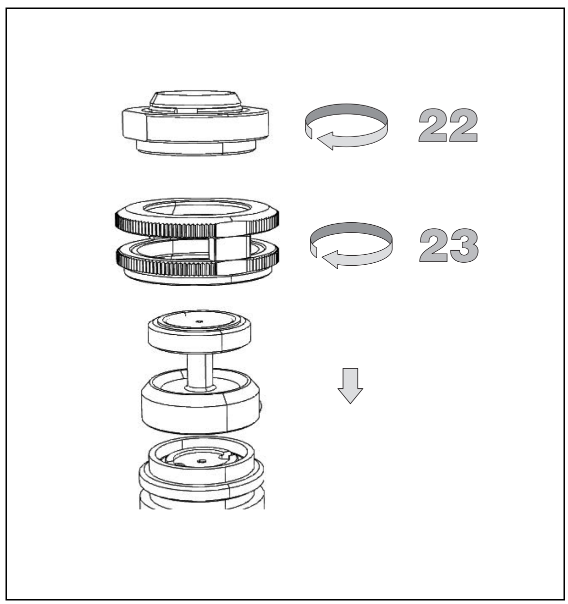


21. Tighten the liner cap using the liner cap wrench provided with the GC.



22. Place the septum holder with the septum onto the inlet port, then screw and tighten the ring nut up to the stop.
23. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



24. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.

25. Press **OVEN**, scroll to `Temp` and press **ON**.
26. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
27. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace the Split Line Tubing

Materials needed

- 7/16” wrenches
- Two 8-10 mm wrences
- Kit Assy Split line 1/16” (PN 190 507 10)
    - Split line tubing 1/16”
    - Brass front/back ferrule for 1/8” tubing
    - 5/16” brass nut
    - 1.6 mm ID brass ferrule
    - M6x0.75 mm (6MB) nut

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
              OVEN
Temp                  50       50 <
Initial time                    2
Ramp 1                       10.0
```

3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a S/SL injector installed on the right channel is considered.

```
        RIGHT INLET (S/SL)
Temp                 250      250 <
Pressure             100      100
Mode                        split
```

4. Scroll to `Temp` and press **OFF**.

5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.

6. Scroll to `Pressure` and press **OFF**.

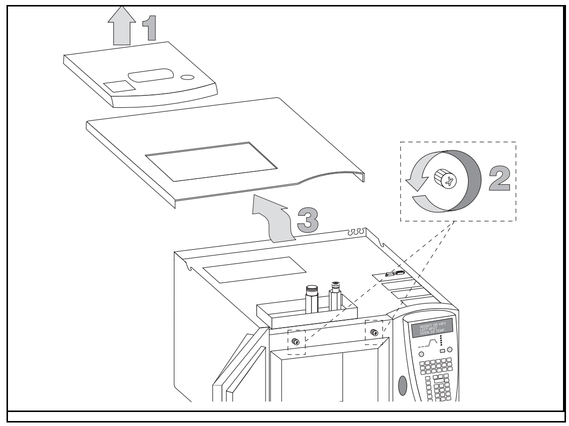
RIGHT CARRIER (S/SL)			
Pressure	30.0	30.0	
Col. flow	3.00		<
Lin. Veloc.		(60.9)	

7. Remove the GC top cover.

   a. (1) Lift the detector cover off the GC top cover.

   b. (2) Open the oven door and unscrew the two top cover fastening screws.

   c. (3) Push the cover back about 1 cm and lift it up and off the GC.

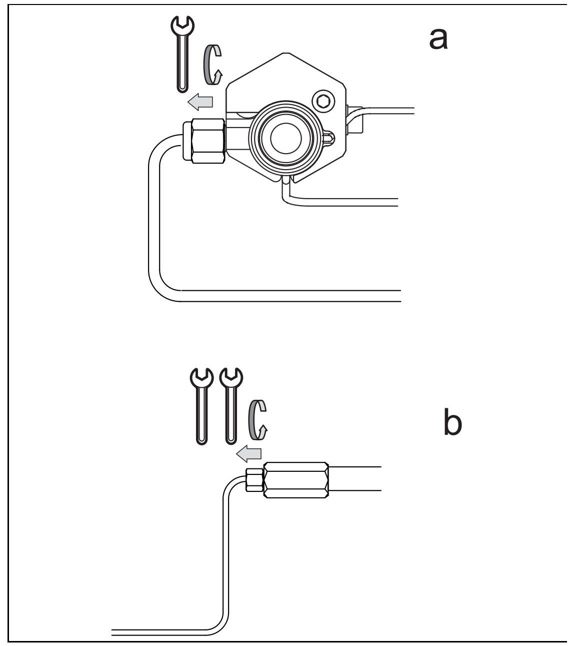


8. Look for the split line tubing connections.

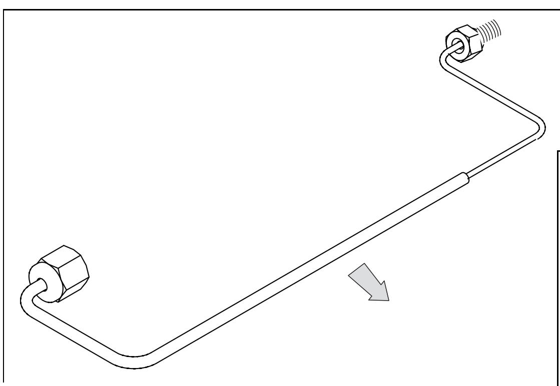
9. Disconnect the split line tubing from the injector body and from the split line filter.

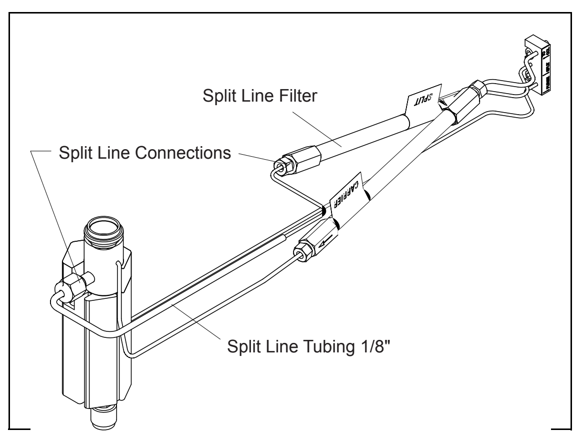
   a. Using the 7/16” wrench, unscrew and disconnect the split line tubing from the injection body.

   b. Using two 8-10 mm wrenches, unscrew and disconnect the split line tubing from the split line filter.



10. Remove the split line tubing from the GC.





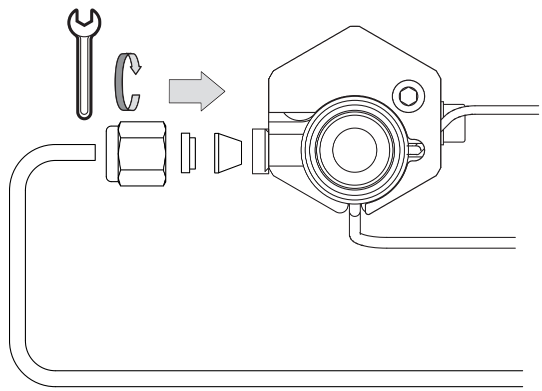
11. Connect the new split line tubing to the injector body.

    a. On the end of the new split line tubing having the greater diameter, slide the 5/16" brass nut and the brass front/back ferrule.

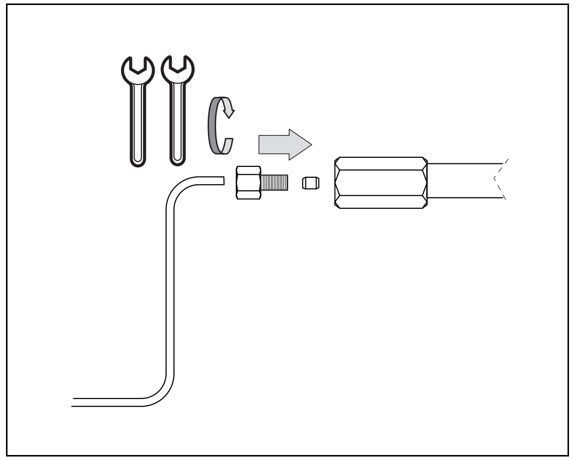
    b. Fully insert the tubing into the fitting on the injector body and rotate the nut finger-tight.

    c. Mark the nut at the 12 o'clock position.

    d. While holding the fitting body steady, tighten the nut tree-quarters turn to the 9 o'clock position.



12. Connect the new split line tubing to the split line dummy filter



   a. On the end of the new split line tubing having the lower diameter, slide the nut 6MB nut and the 1.6 mm ID brass ferrule.

   b. Insert the tubing into the outlet of the split line dummy filter.

   c. Finger-tighten the 6 MB nut until it start to grip the filter.

   d. Use two 8-10 mm wrench to tighten the nut. Use no more pressure than is necessary to obtain a good seal.

13. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
14. Press **OVEN**, scroll to `Temp` and press **ON**.
15. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
16. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.If leaks are detected, check the critical connections with an electronic leak detector to locate the possible leaks.
17. When the injector is free of leaks, reinstall the GC top cover.
18. Set the normal injector and GC working condition.

4

Maintaining a Cold On-Column Injector for Standard Automatic Injections

This inlet works in combination with the TriPlus autosampler allowing standard injections into a 0.53-mm ID capillary column.

Chapter at a Glance…

Operating Sequences

Automated Operation Cold On-Column Injector

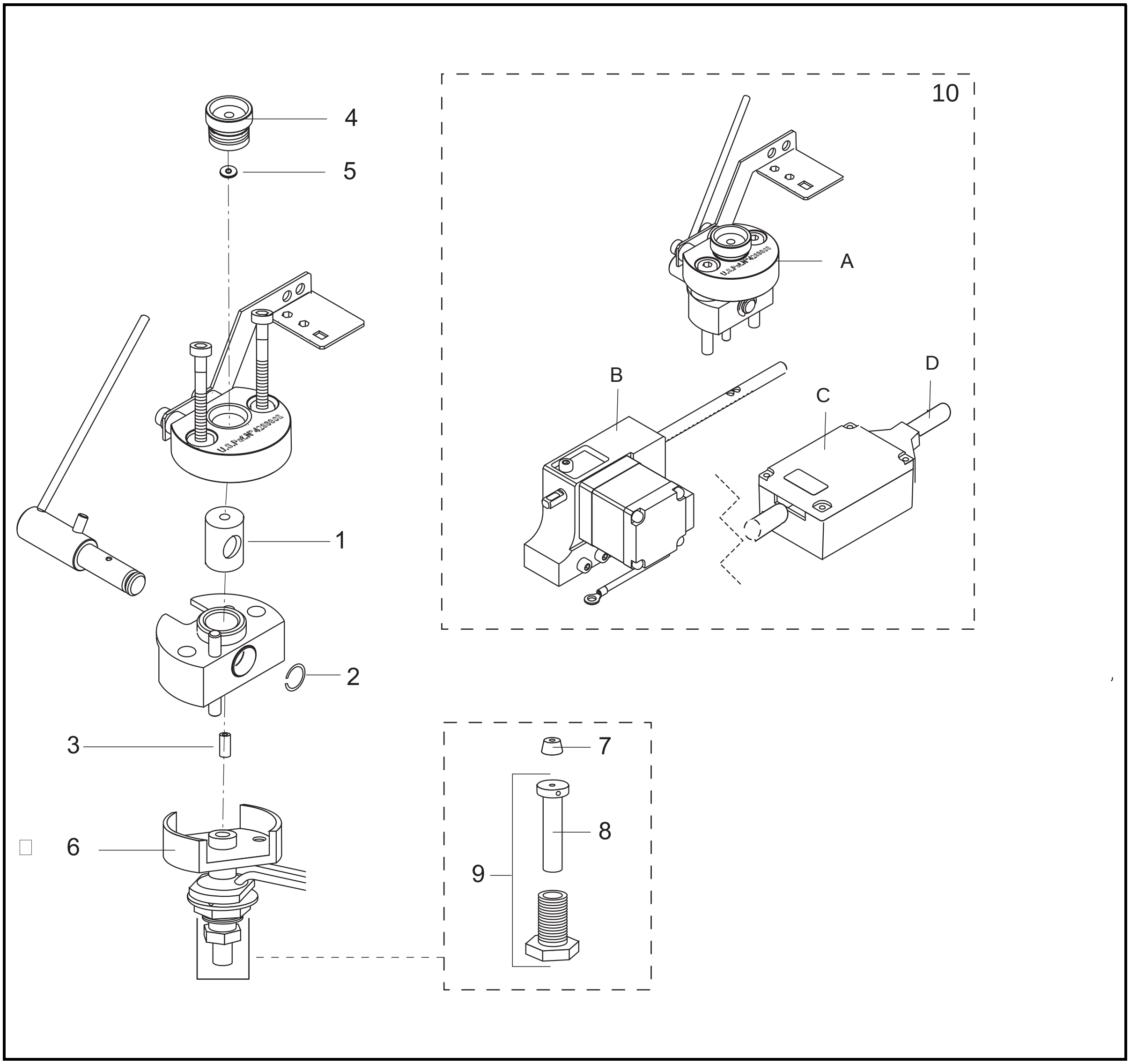


Table 4-1. Automated Operation Cold On-Column Injector Parts Identification Table

	Description	P/N
1	Seal, PTFE	290 070 01
2	Clip	426 003 57
3	Needle guide	453 220 36
4	Seal Holder	350 014 71
5	O-ring	290 013 02
6	OC base	299 022 56
7	Vespel ferrule for 0.25 mm ID column	290 134 61
7	Vespel ferrule for 0.32 mm ID column	290 134 60
7	Vespel ferrule for 0.53 mm ID column	290 134 71
8	Backwasher/cooling sleeve for OC	452 000 01
9	Column retaining nut and backwasher for OC	452 100 01
10	Kit On-Column Actuator for TriPlus (Left) The kit includes: • A = Cold on-column injector head for TriPlus autosampler • B = Automatic actuator Assy • C = Control Interface • D = Control interface to TriPlus sampler connecting cable	190 502 62

Cold On-Column Injector Maintenance

This injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel and do not require periodic maintenance, except for O-ring replacement in the automatic version. The PTFE seal of the rotary valve, if worn, can be replaced by the user.

Replace the O-ring

This sequence is specific for the Automated Operation Cold On-Column Injector with TriPlus sampler. In this version of the Cold On-Column Injector, the o-ring should be replaced at least every 200 injections, or every time a problem related to O-ring damage or wear occurs. Refer to Chapter 19, *Analytical Troubleshooting*, for more information. The correct sequence is explained in the Operating Sequence *Replace the O-Ring* on page 83.

Possible leaks in the rotary valve

Some symptoms, particularly a low reproducibility in results, may indicate that the connection between the rotary valve and the relevant PTFE seal is not in good condition. Refer to Chapter 19, *Analytical Troubleshooting* for more information.

To eliminate possible leaks on the rotary valve, tighten the Allen screws that fix the upper and the lower parts of the injector, then test the injector to see if the symptom has disappeared. If not, replace the seal of the rotary valve, as explained in the *Replace the Rotary Valve Seal* Operating Sequence on page 87.

OPERATING SEQUENCE

Replace the O-Ring

NOTE This sequence is used for the Automated Operation Cold On-Column Injector for TriPlus Sampler.

Materials needed

- Flat screwdriver
- O-Ring (See # 5, Table 4-1 on page 81)
- Tweezers

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

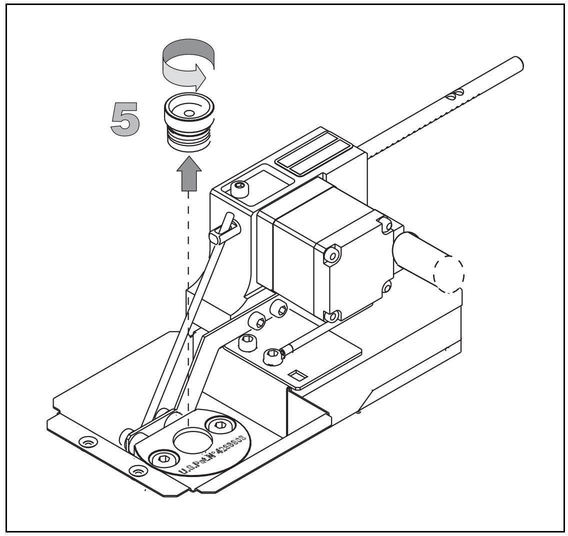
```
                OVEN
Temp                    70      70 <
Initial time                   1.0
Ramp 1                        20.0
```

3. Press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating, scroll to `Pressure` and press **OFF**.

```
         LEFT CARRIER (OCI)
Pressure              30.0    30.0
Col. flow             3.00         <
Lin. Veloc.                 (60.9)
```

4. Remove the injector/detector cover.

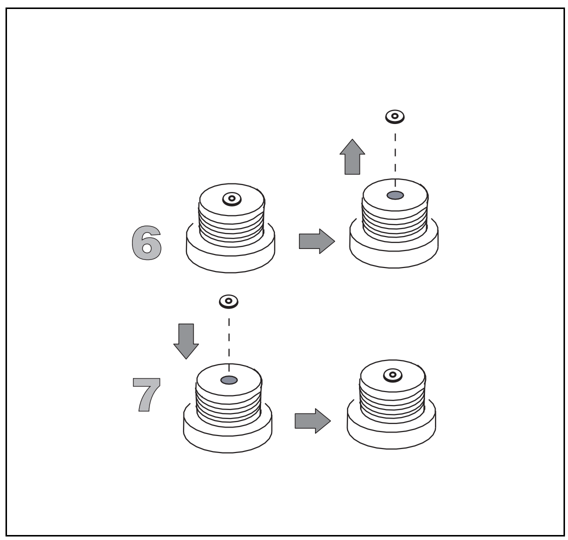
5. Unscrew the seal holder and remove it.



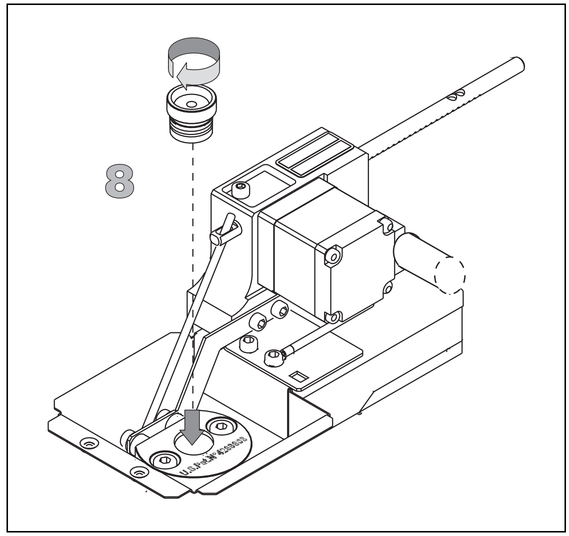
6. Remove the O-ring from the seal holder with the aid of a sharp, non-metallic tool.

7. Insert a new O-ring in the relevant seat.

CAUTION **Use tweezers to avoid touching the O-ring with your fingers.**



8. Screw and tighten the seal holder onto its housing.



CAUTION **Tightening of the seal holder must be performed carefully in order to properly seal the On-Column needle guide. Because of the tightening, the Viton O-Ring expands into its seat and closes the needle path, sealing the On-Column injector.**

With the injection valve on the open position, use an appropriate syringe (needle 80-mm long) to verify that the needle freely slides inside the injector.

WARNING! **Please verify that the syringe needle, due to the friction exercised against the compressed O-Ring seal, enters with a reasonable applied force into the On-Column injector port. The needle must enter with some noticeable effort, but smoothly, and must not be bent by the force applied, in order to avoid needle jamming during automated injections. If the needle tends to bend, loosen and tighten again the seal holder until it is properly tightened.**

9. Reposition the injector/detector cover.

10. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
11. Press **OVEN**, scroll to `Temp` and press **ON**.
12. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace the Rotary Valve Seal

Materials needed

- 5.5 mm hexagonal wrench
- PTFE seal (P/N 290 070 01)
- 3 mm Allen wrench
- 4-mm Allen wrench
- 5.5-mm wrench

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

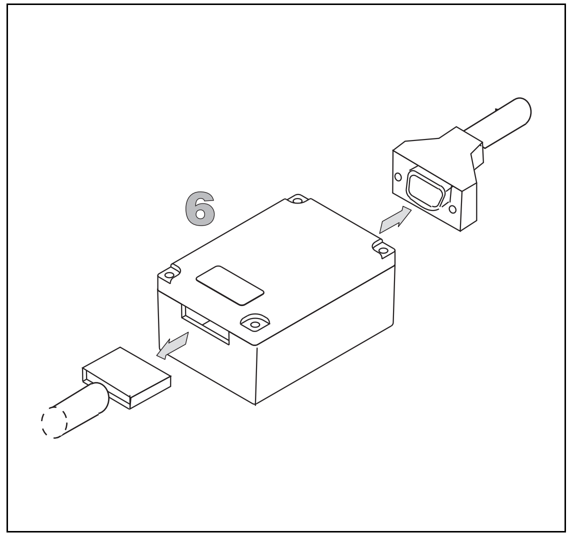
```
                 OVEN
Temp                    70      70 <
Initial time                   1.0
Ramp 1                        20.0
```

3. Press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating, scroll to `Pressure` and press **OFF**.

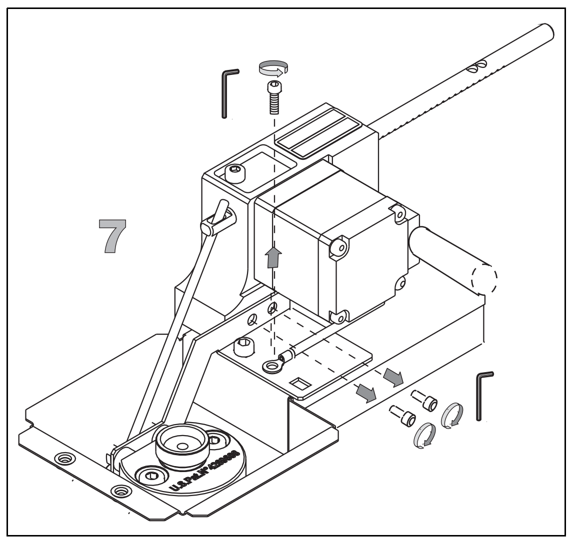
```
        LEFT CARRIER (OCI)
Pressure              30.0    30.0
Col. flow             3.00         <
Lin. Veloc.                 (60.9)
```

4. Remove the injector/detector cover.
5. Switch off the TriPlus sampler

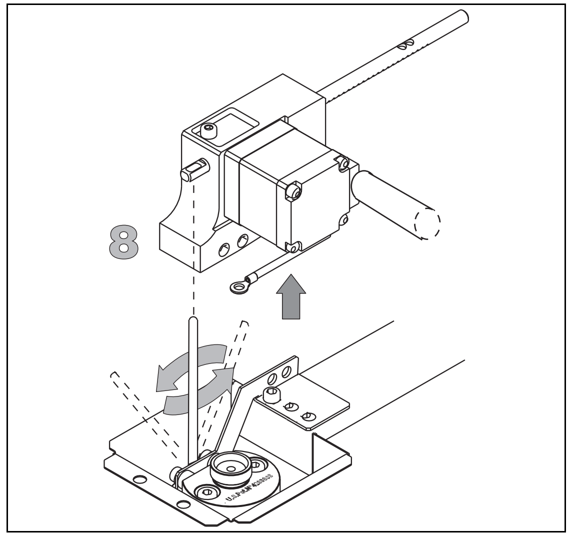
6. Disconnect the cables coming from the actuator and the TriPlus sampler from the control interface.



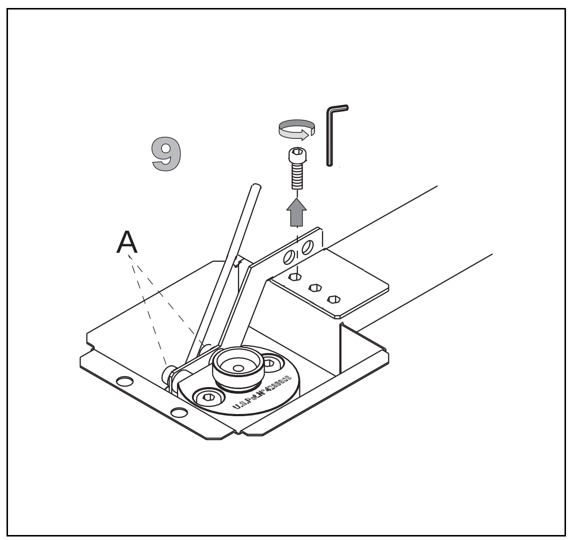
7. Remove the actuator body and the ground wire fixing screw.
   Remove the two screws that fix the actuator body to the fixing arm of the OC head.



8. Move vertically the valve lever to allows its extraction through the slot on the actuator shaft. Carefully lift up the automatic actuator to remove it from the OC housing.

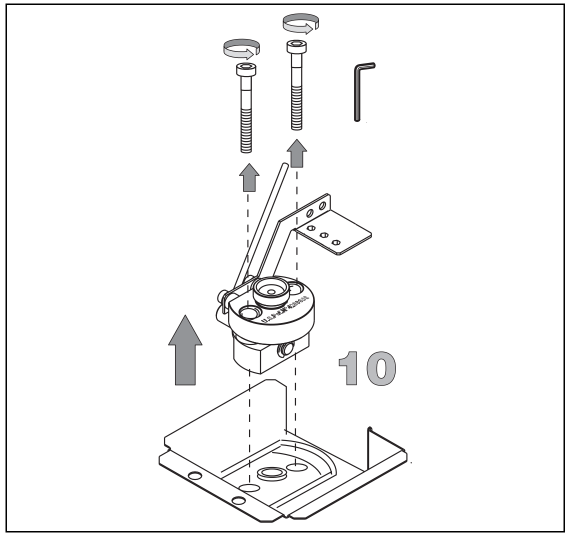


9. Loosen the two screws A on the side of the OC head. Unscrew the remaining fixing screws of the head arm by using a 3-mm Allen wrench.

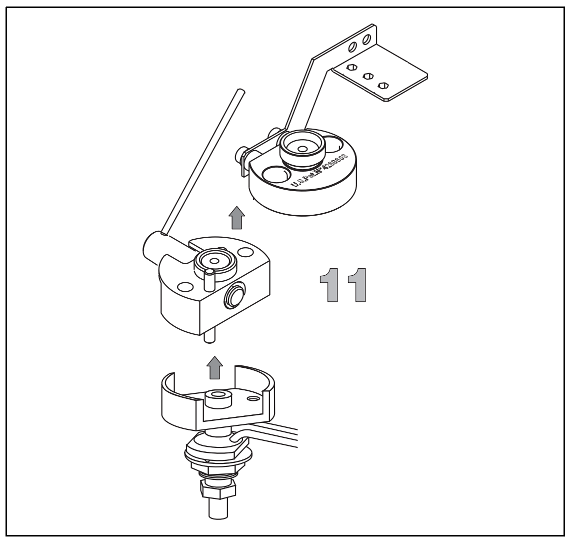


10. Unscrew the two Allen bolts that fix the injector body to the injector base. Remove the injector body from its base.

CAUTION **Pay attention not to lose the small needle guide when removing the injector body. The guide normally can be found inserted in the needle channel of the injector base or of the valve seal.**

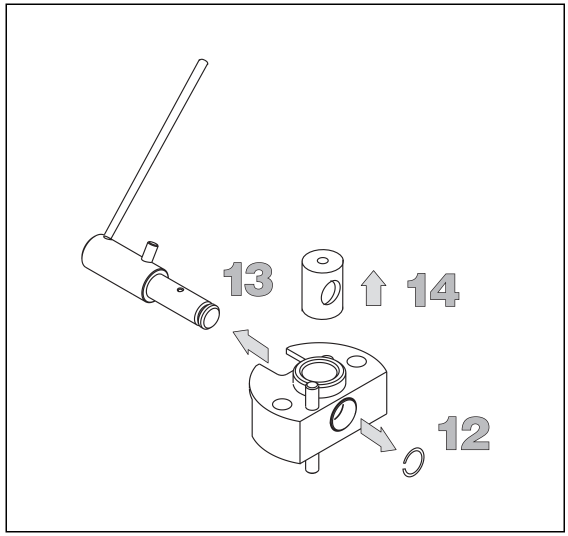


11. Disassemble the injector body, separating the head from the lower part.

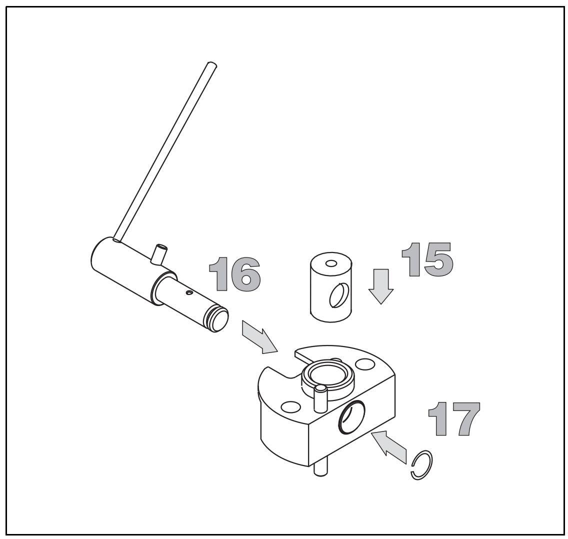


12. Remove the clip that retains the rotary valve.
13. Push the rotary valve out from the injector head.
14. Push the PTFE seal upwards to eject it.

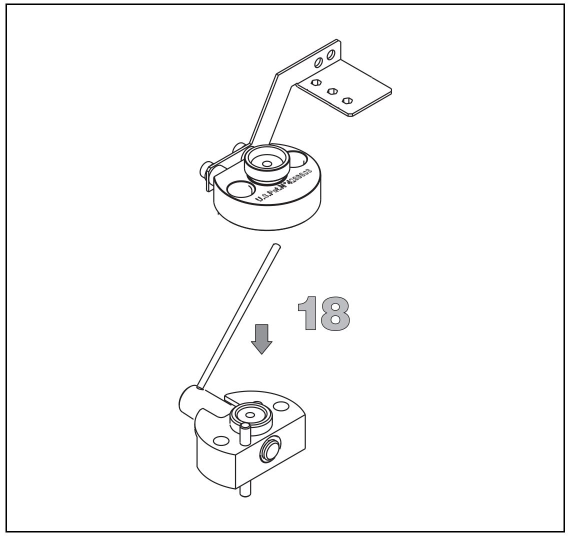
CAUTION **In order to avoid leaks of sample from the valve assembly, take care not to damage the inside of the head. Eject the seal with the aid of a non-metallic tool.**



15. Insert a new PTFE seal into its housing, paying attention to the alignment of the holes.
16. Insert the rotary valve into its housing.
17. Fix the rotary valve with the relevant clip.

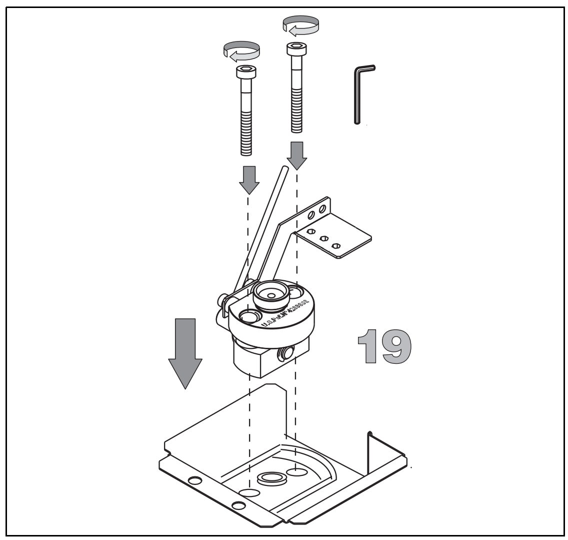


18. Reassemble the two parts of the injector body.

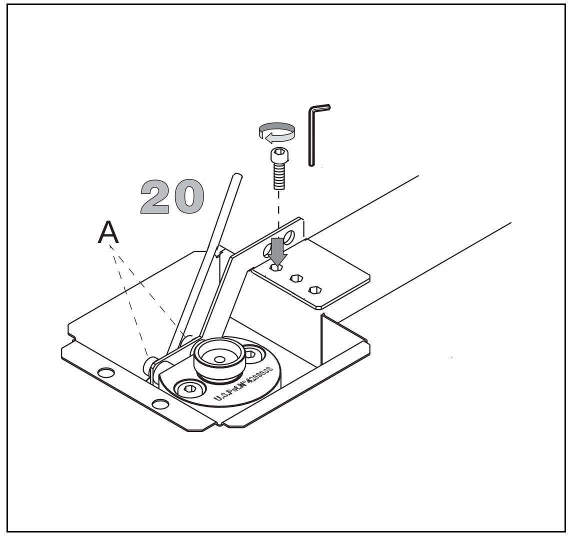


19. Remount the injector body on its base, tightening the two Allen screws.

CAUTION **Be careful to reinstall the needle guide with the small cut downwards, otherwise no flow of carrier gas to the column will be allowed.**

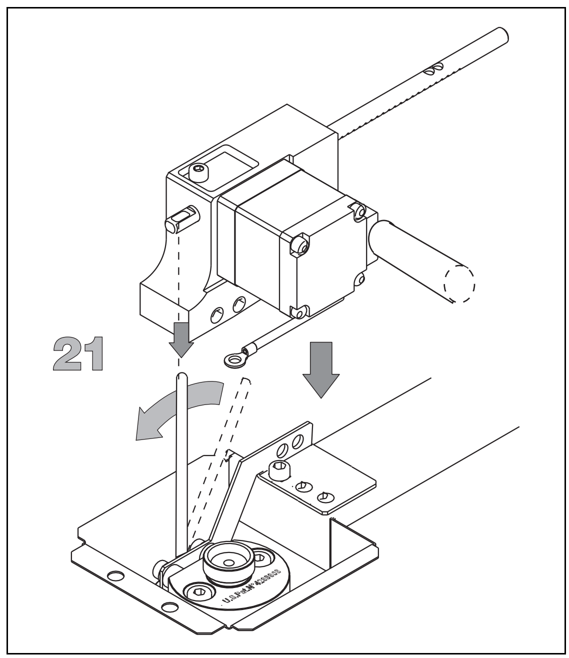


20. .Fix the OC head on the ceiling of the GC and tighten the two screws A on the side of the OC head previously loosened.

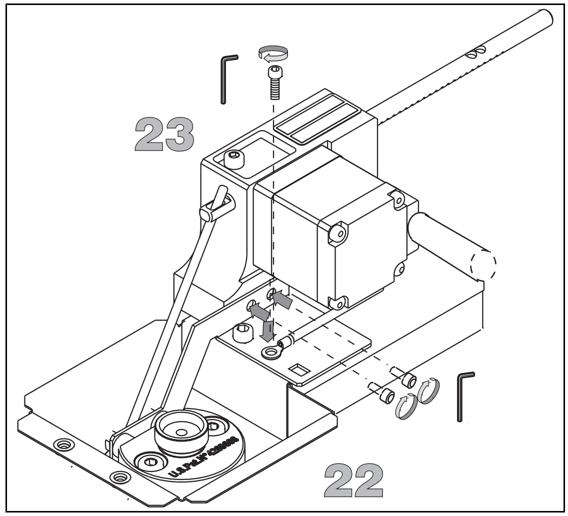


21. .Reinstall the automatic actuator on the OC housing paying attention to insert the valve lever through the slot provided on the automatic actuator shaft.

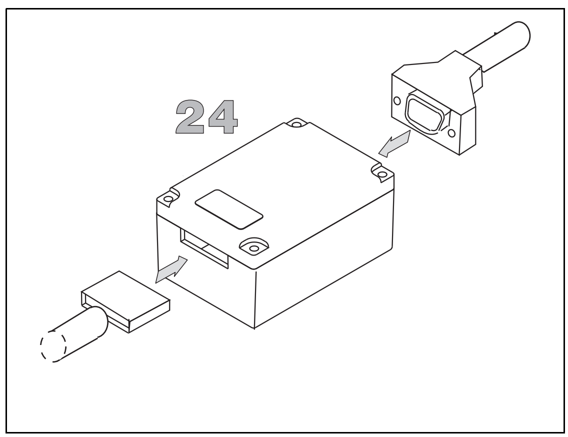
    Place the automatic actuator paying attention to not crush the cables under it.



22. .Adjust the position of the automatic actuator up to align the fixing holes provided on the actuator body with the corresponding fixing holes located on the fixing arm of the OC head. Fix the parts by using the two fixing screws.
23. Reconnect the ground wire coming from the actuator motor

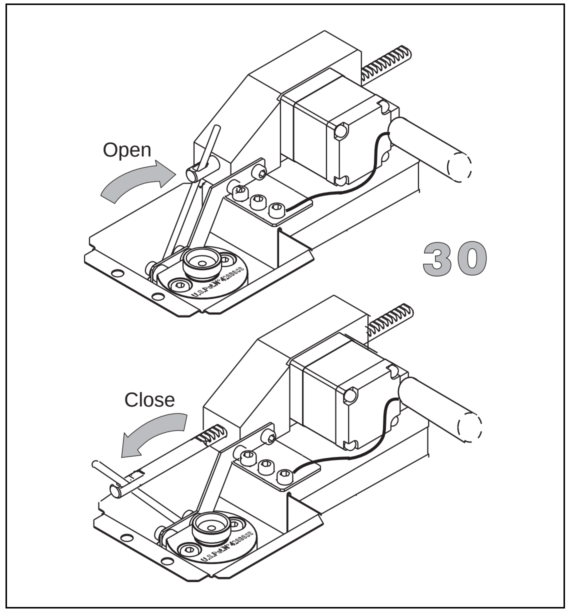


24. .Reconnect the cables coming from the actuator and the TriPlus sampler to the control interface.



25. Reposition the injector/detector cover.
26. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
27. Switch on the TriPlus sampler.

28. Press **OVEN**, scroll to `Temp` and press **ON**.
29. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.
30. From the data system in use, enable the automatic actuator to open and close the rotary valve of the OC injector by using the direct command **`OC Actuator`**. The actuator move the OC valve lever as shown in the figure on the right.



5

Maintaining a Cold On-Column Injector Direct Narrow-Bore

This inlet works in combination with the TriPlus autosampler allowing standard injections into a 0.25/0.32-mm. ID capillary column.

Chapter at a Glance…

Operating Sequences

Automated Operation Cold On-Column Injector

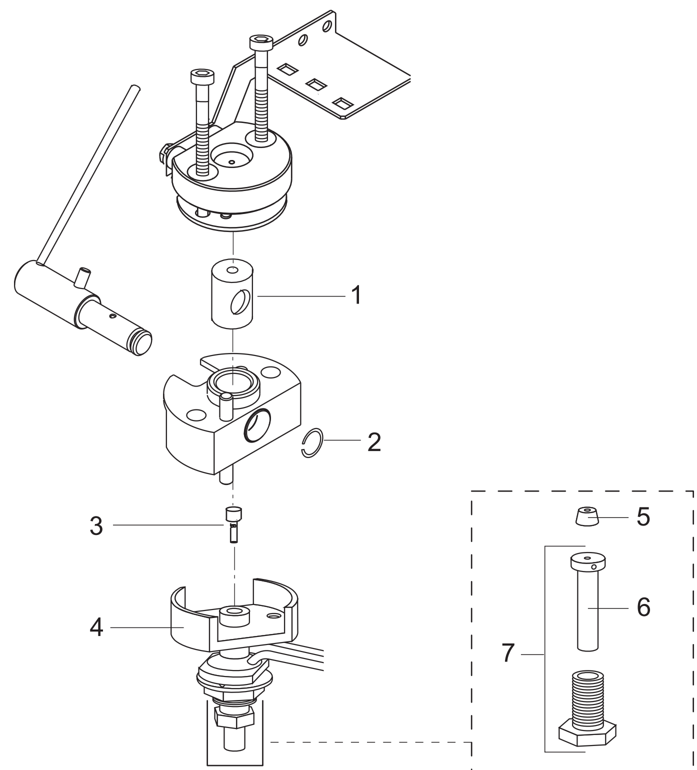


Table 5-1. Automated Operation Cold On-Column Injector parts identification table

	Description	P/N
1	Seal, PTFE	290 071 06
2	Clip	426 003 57
3	Needle guide (set of 2)	453 220 63
4	OC base	299 022 56
5	Vespel ferrule for 0.25 mm ID column	290 134 61
5	Vespel ferrule for 0.32 mm ID column	290 134 60
5	Vespel ferrule for 0.53 mm ID column	290 134 71
6	Backwasher/cooling sleeve for OC	452 000 01
7	Column retaining nut and backwasher for OC	452 100 01
	On-Column injector head assy	299 022 48
	Kit upgrade OC Direct narrow-bore for TriPlus	190 502 63

Cold On-Column Injector Maintenance

This injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel and do not require periodic maintenance. The PTFE seal of the rotary valve, if worn, can be replaced by the user.

Possible leaks in the rotary valve

Some symptoms, particularly a low reproducibility in results, may indicate that the connection between the rotary valve and the relevant PTFE seal is not in good condition. Refer to Chapter 19, *Analytical Troubleshooting* for more information.

To eliminate possible leaks on the rotary valve, tighten the Allen screws that fix the upper and the lower parts of the injector, then test the injector to see if the symptom has disappeared. If not, replace the seal of the rotary valve, as explained in the *Replace the Rotary Valve Seal* Operating Sequence on page 101.

OPERATING SEQUENCE

Replace the Rotary Valve Seal

Materials needed

- 5.5 mm hexagonal wrench
- PTFE seal (P/N 29007106)
- 3 mm Allen wrench
- 4-mm Allen wrench
- 5.5-mm wrench

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

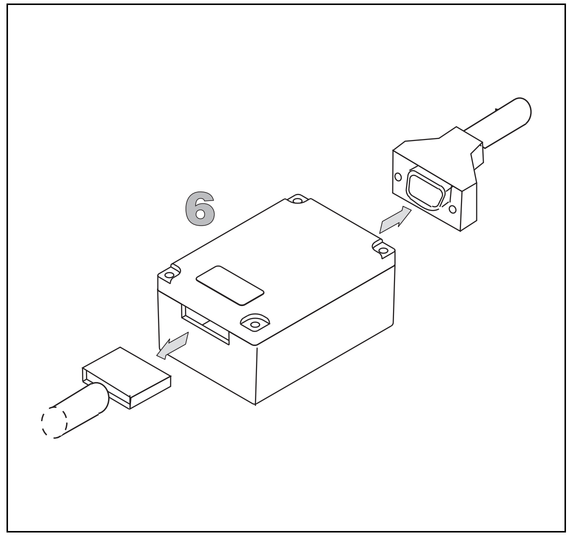
```
               OVEN
Temp                    70      70 <
Initial time                   1.0
Ramp 1                        20.0
```

3. Press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating, scroll to `Pressure` and press **OFF**.

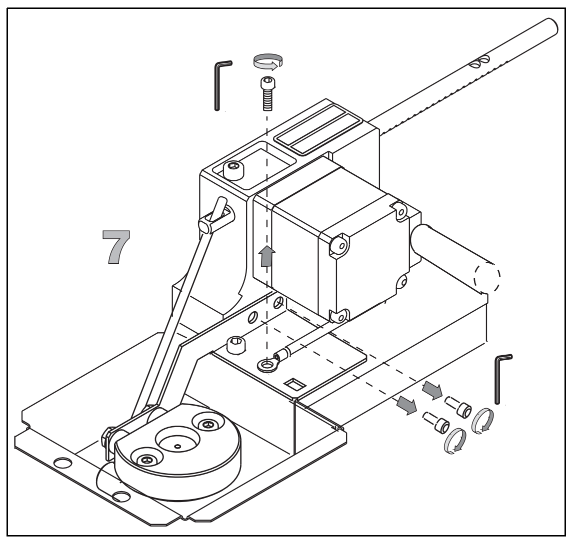
```
        LEFT CARRIER (OCI)
Pressure              30.0    30.0
Col. flow             3.00           <
Lin. Veloc.                  (60.9)
```

4. Remove the injector/detector cover.
5. Switch off the TriPlus sampler

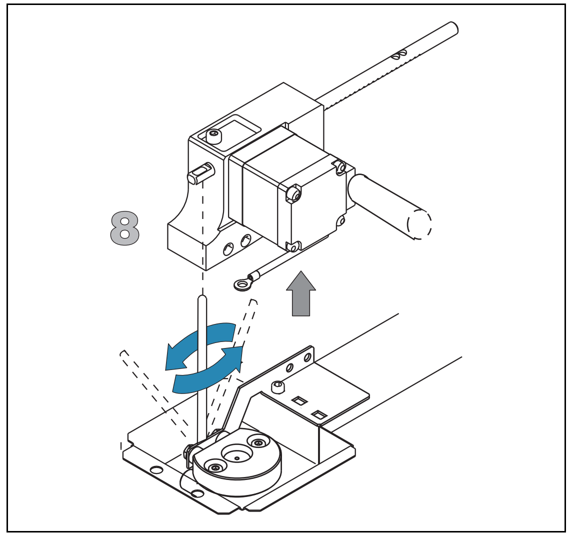
6. Disconnect the cables coming from the actuator and the TriPlus sampler from the control interface.



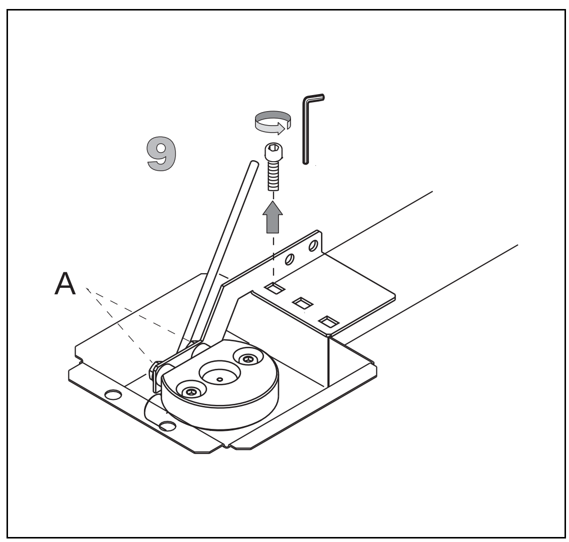
7. Remove the actuator body and the ground wire fixing screw.
   Remove the two screws that fix the actuator body to the fixing arm of the OC head.



8. Move vertically the valve lever to allows its extraction through the slot on the actuator shaft. Carefully lift up the automatic actuator to remove it from the OC housing.

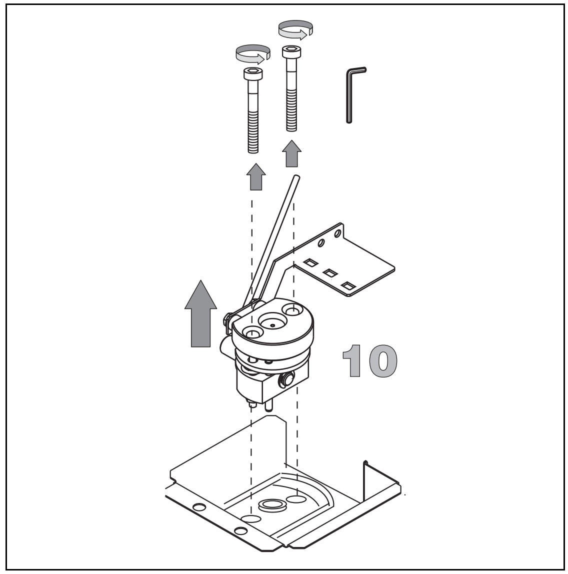


9. Loosen the two screws A on the side of the OC head. Unscrew the remaining fixing screws of the head arm by using a 3-mm Allen wrench.

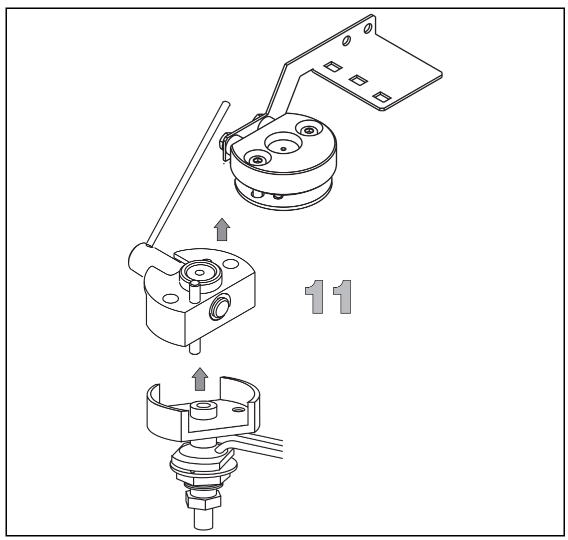


10. Unscrew the two Allen bolts that fix the injector body to the injector base. Remove the injector body from its base.

CAUTION **Pay attention not to lose the small needle guide when removing the injector body. The guide normally can be found inserted in the needle channel of the injector base or of the valve seal.**

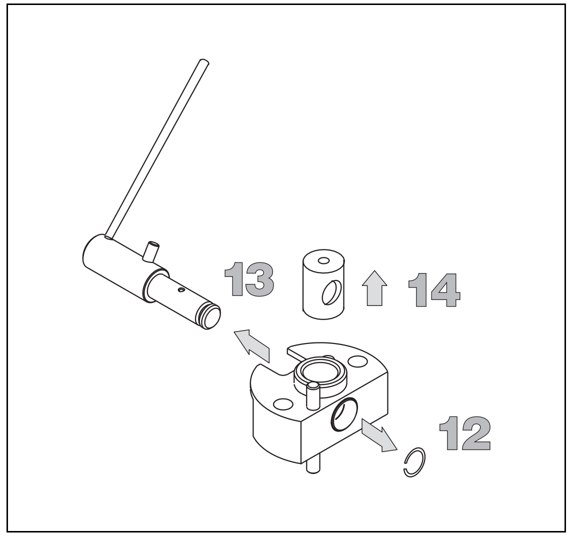


11. Disassemble the injector body, separating the head from the lower part.

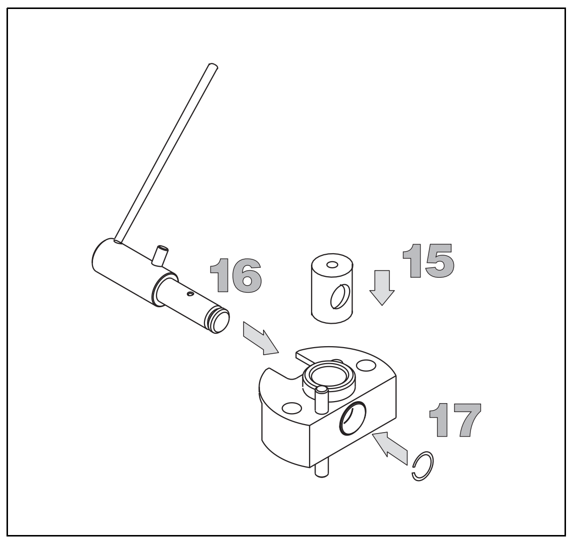


12. Remove the clip that retains the rotary valve.
13. Push the rotary valve out from the injector head.
14. Push the PTFE seal upwards to eject it.

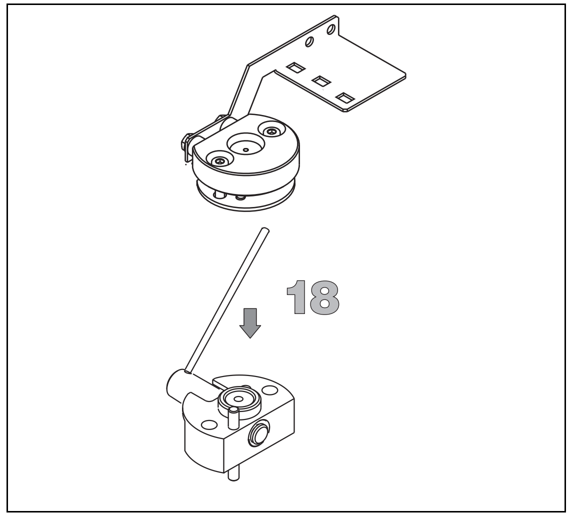
CAUTION **In order to avoid leaks of sample from the valve assembly, take care not to damage the inside of the head. Eject the seal with the aid of a non-metallic tool.**



15. Insert a new PTFE seal into its housing, paying attention to the alignment of the holes.
16. Insert the rotary valve into its housing.
17. Fix the rotary valve with the relevant clip.

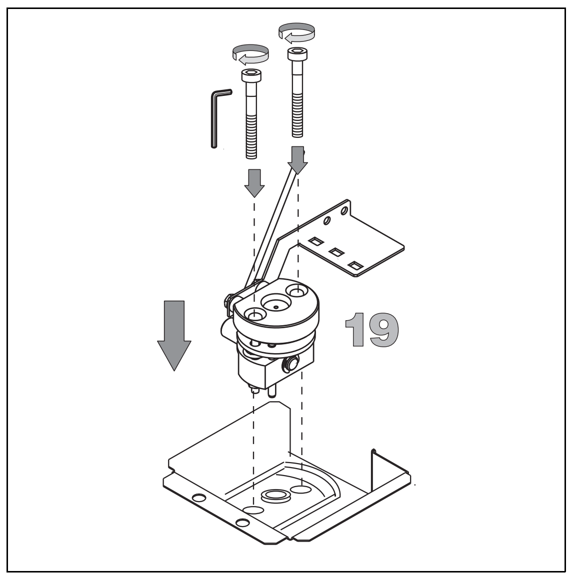


18. Reassemble the two parts of the injector body.

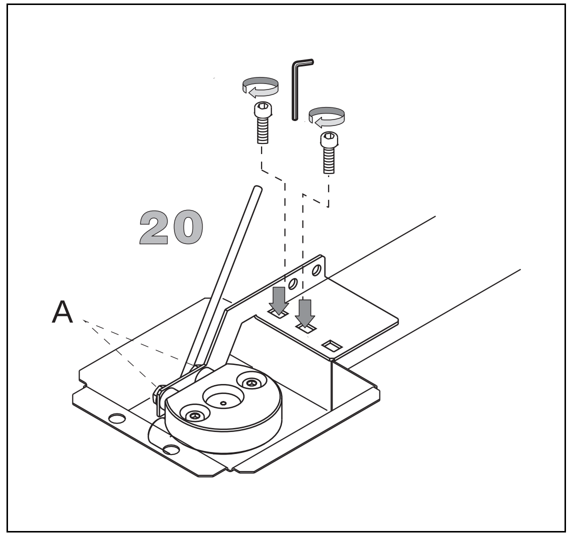


19. Remount the injector body on its base, tightening the two Allen screws.

CAUTION **Be careful to reinstall the needle guide with the small cut downwards, otherwise no flow of carrier gas to the column will be allowed.**

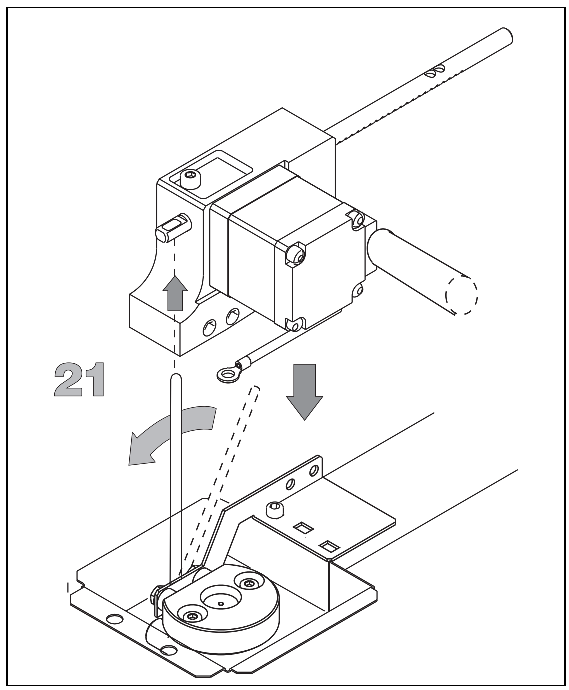


20. .Fix the OC head on the ceiling of the GC and tighten the two screws A on the side of the OC head previously loosened.

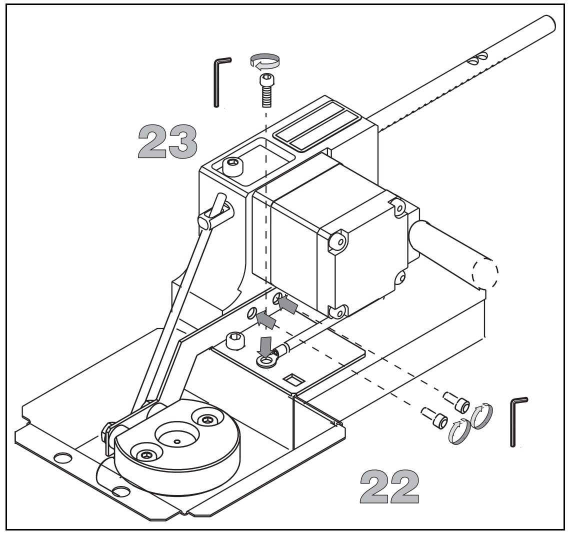


21. .Reinstall the automatic actuator on the OC housing paying attention to insert the valve lever through the slot provided on the automatic actuator shaft.

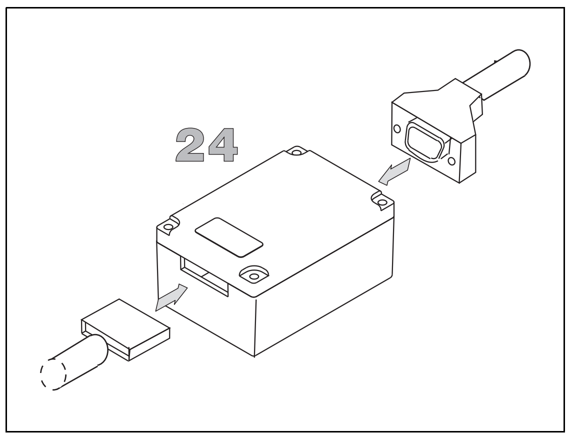
    Place the automatic actuator paying attention to not crush the cables under it.



22. .Adjust the position of the automatic actuator up to align the fixing holes provided on the actuator body with the corresponding fixing holes located on the fixing arm of the OC head. Fix the parts by using the two fixing screws.
23. Reconnect the ground wire coming from the actuator motor

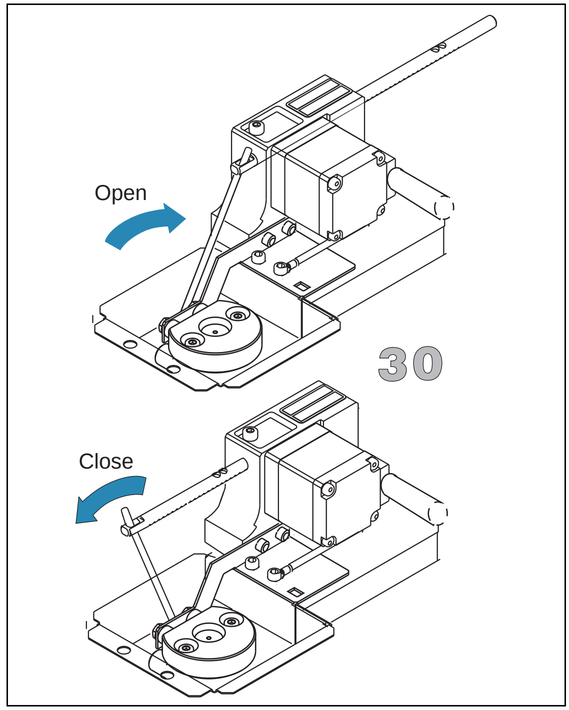


24. .Reconnect the cables coming from the actuator and the TriPlus sampler to the control interface.



25. Reposition the injector/detector cover.
26. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
27. Switch on the TriPlus sampler.

28. Press **OVEN**, scroll to `Temp` and press **ON**.
29. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.
30. From the data system in use, enable the automatic actuator to open and close the rotary valve of the OC injector by using the direct command **`OC Actuator`**. The actuator move the OC valve lever as shown in the figure on the right.



6

Maintaining a Cold On-Column Injector

Chapter at a Glance…

Operating Sequences

Manual Cold On-Column Injector

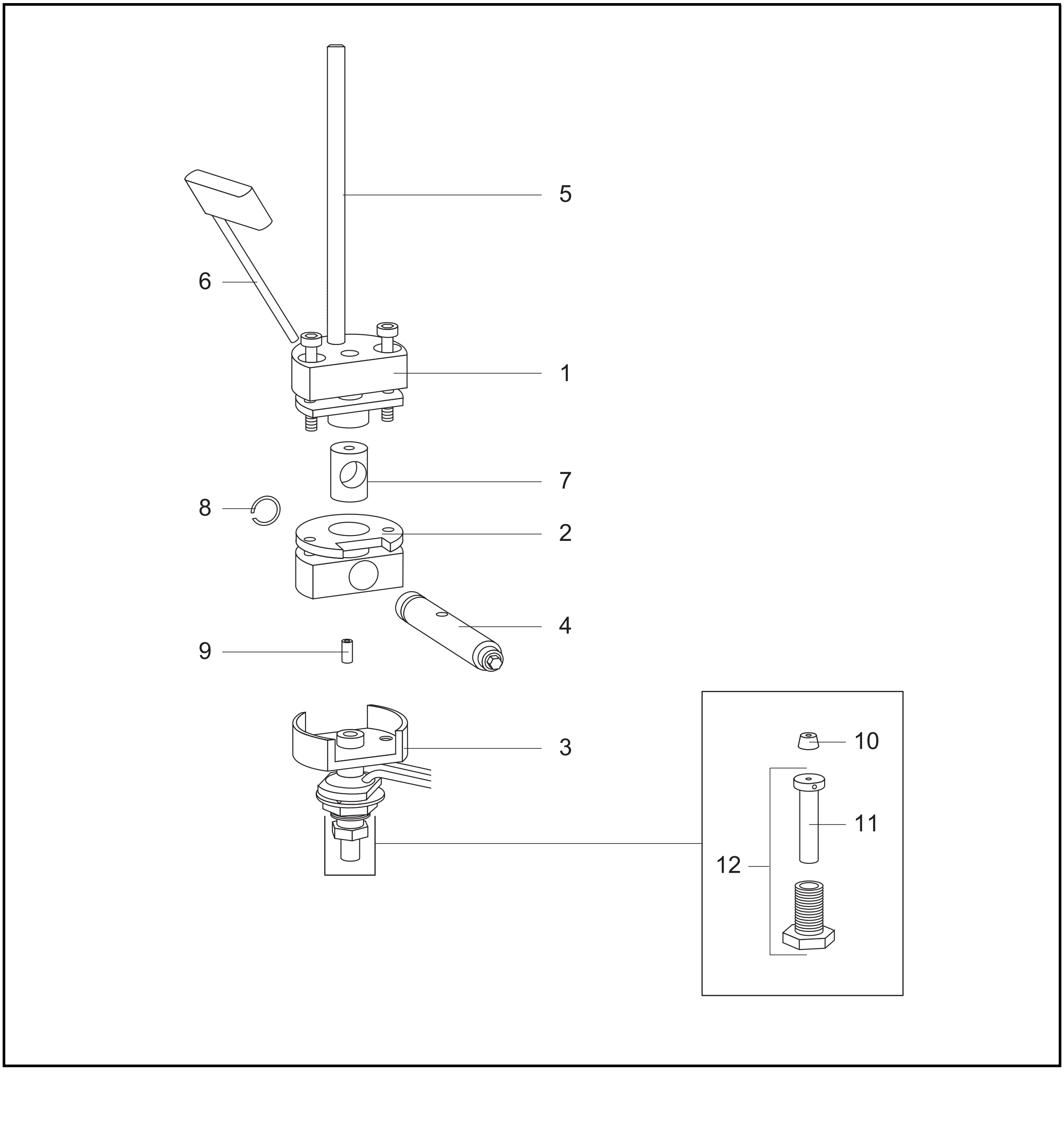


Table 6-1. Manual Cold On-Column Injector parts identification table

No.	Description	P/N
1	Injector body (upper part)	
2	Injector body (lower part)	
3	Injector base	
4	Rotary valve	
5	Syringe support	
6	Valve actuation lever	
7	PTFE seal of the rotary valve	290 070 01
8	Rotary valve retaining clip	426 003 57
9	Needle guide	453 220 36
10	Vespel ferrule for 0.25 mm ID column	290 134 61
	Vespel ferrule for 0.32 mm ID column	290 134 60
	Vespel ferrule for 0.53 mm ID column	290 134 71
11	Backwasher/cooling sleeve	452 000 01
12	Column retaining nut and backwasher	452 100 01

Cold On-Column Injector Maintenance

This injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel and do not require periodic maintenance, except for septum replacement in the automatic version. The PTFE seal of the rotary valve, if worn, can be replaced by the user.

Possible leaks in the rotary valve

Some symptoms, particularly a low reproducibility in results, may indicate that the connection between the rotary valve and the relevant PTFE seal is not in good condition. Refer to Chapter 19, *Analytical Troubleshooting* for more information.

To eliminate possible leaks on the rotary valve, tighten the Allen screws that fix the upper and the lower parts of the injector, then test the injector to see if the symptom has disappeared. If not, replace the seal of the rotary valve, as explained in the *Replace the Rotary Valve Seal* Operating Sequence on page 115.

OPERATING SEQUENCE

Replace the Rotary Valve Seal

NOTE Drawings refer to the Manual Cold On-Column Injector.

Materials needed

- 5.5 mm hexagonal wrench
- PTFE seal (P/N 290 070 01)
- 3 mm Allen wrench

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

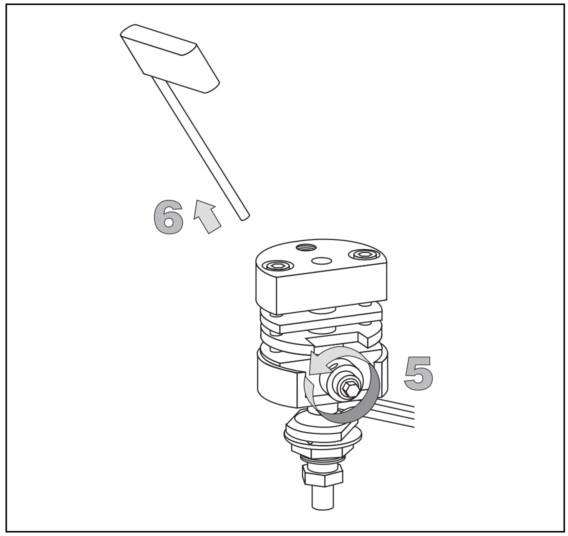
```
                OVEN
Temp                     70      70 <
Initial time                    1.0
Ramp 1                         20.0
```

3. Press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating, scroll to `Pressure` and press **OFF**.

```
         LEFT CARRIER (OCI)
Pressure               30.0    30.0
Col. flow              3.00        <
Lin. Veloc.                  (60.9)
```

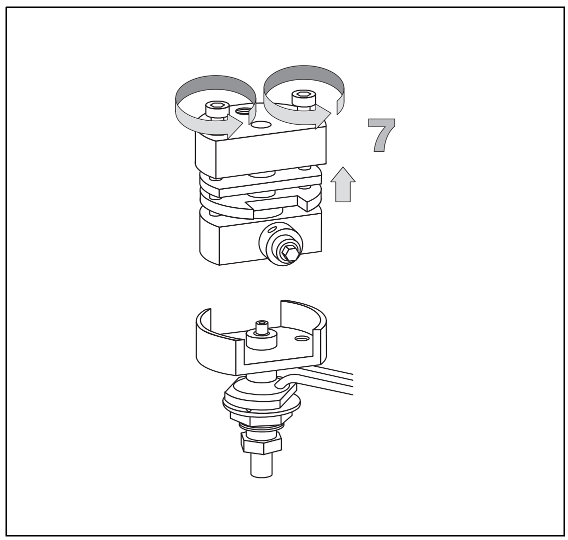
4. Remove the injector/detector cover.

5. Loosen the screw that retains the actuation lever of the injector.
6. Remove the lever.

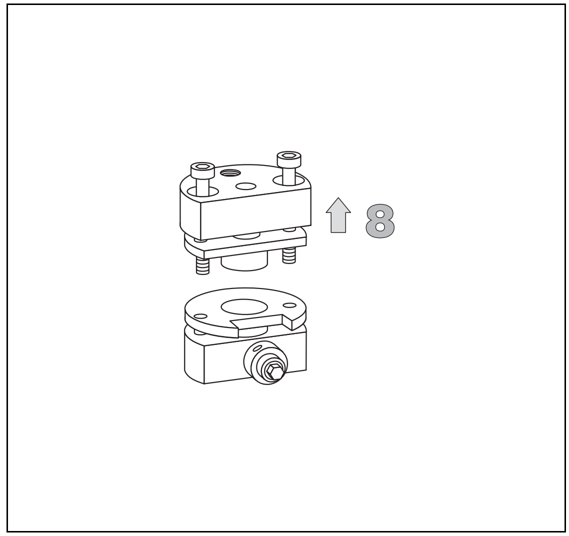


7. Unscrew the two Allen bolts that fix the injector body to the injector base. Remove the injector body from its base.

CAUTION **Pay attention not to lose the small needle guide when removing the injector body. The guide normally can be found inserted in the needle channel of the injector base or of the valve seal.**

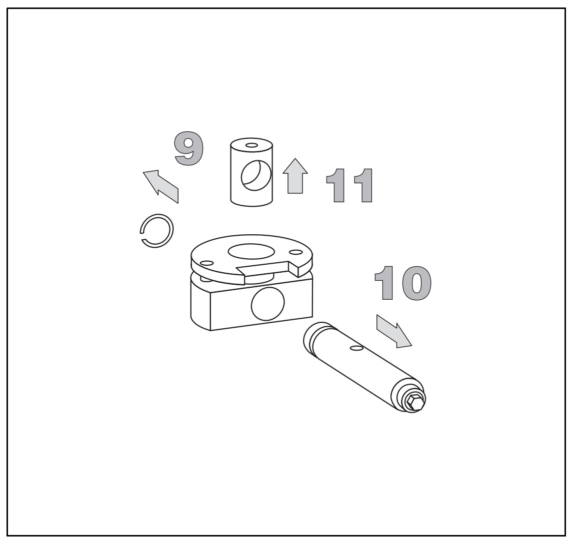


8. Disassemble the injector body, separating the head from the lower part.

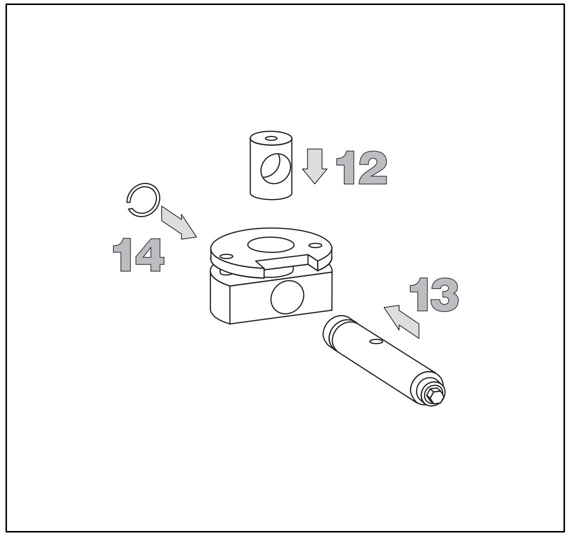


9. Remove the clip that retains the rotary valve.
10. Push the rotary valve out from the injector head.
11. Push the PTFE seal upwards to eject it.

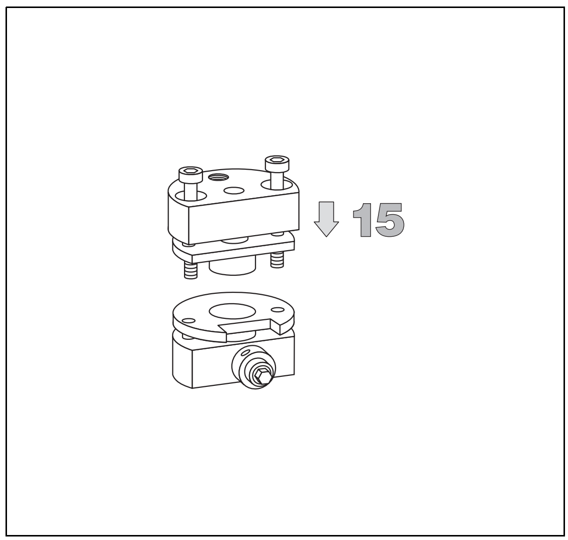
CAUTION **In order to avoid leaks of sample from the valve assembly, take care not to damage the inside of the head. Eject the seal with the aid of a non-metallic tool.**



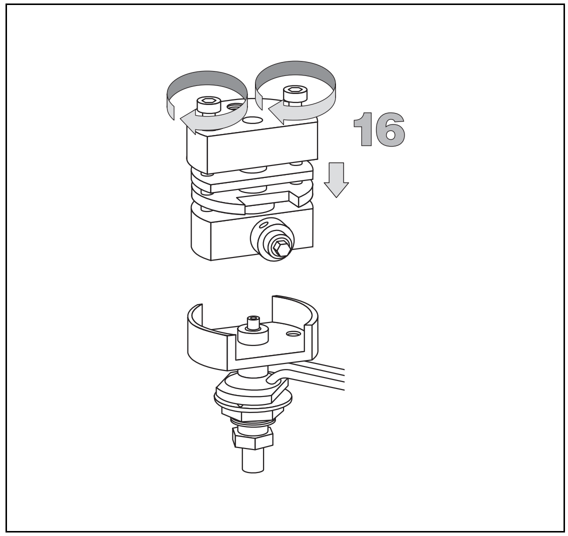
12. Insert a new PTFE seal into its housing, paying attention to the alignment of the holes.
13. Insert the rotary valve into its housing.
14. Fix the rotary valve with the relevant clip.



15. Reassemble the two parts of the injector body.

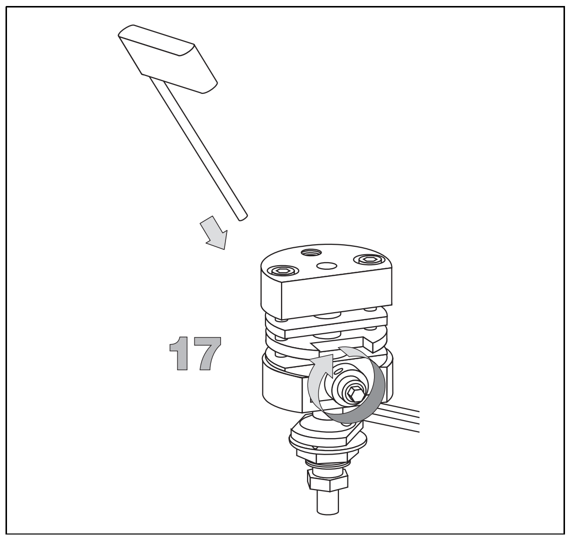


16. Remount the injector body on its base, tightening the two Allen screws.



17. Insert the valve lever into its housing, tightening the retaining screw.

CAUTION **Be careful to reinstall the needle guide with the small cut downwards, otherwise no flow of carrier gas to the column will be allowed.**



18. Reposition the injector/detector cover.
19. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.

20. Press **OVEN**, scroll to `Temp` and press **ON**.
21. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

7

Maintaining a Packed Column Injector

Chapter at a Glance…

Operating Sequences

Packed Column Injector

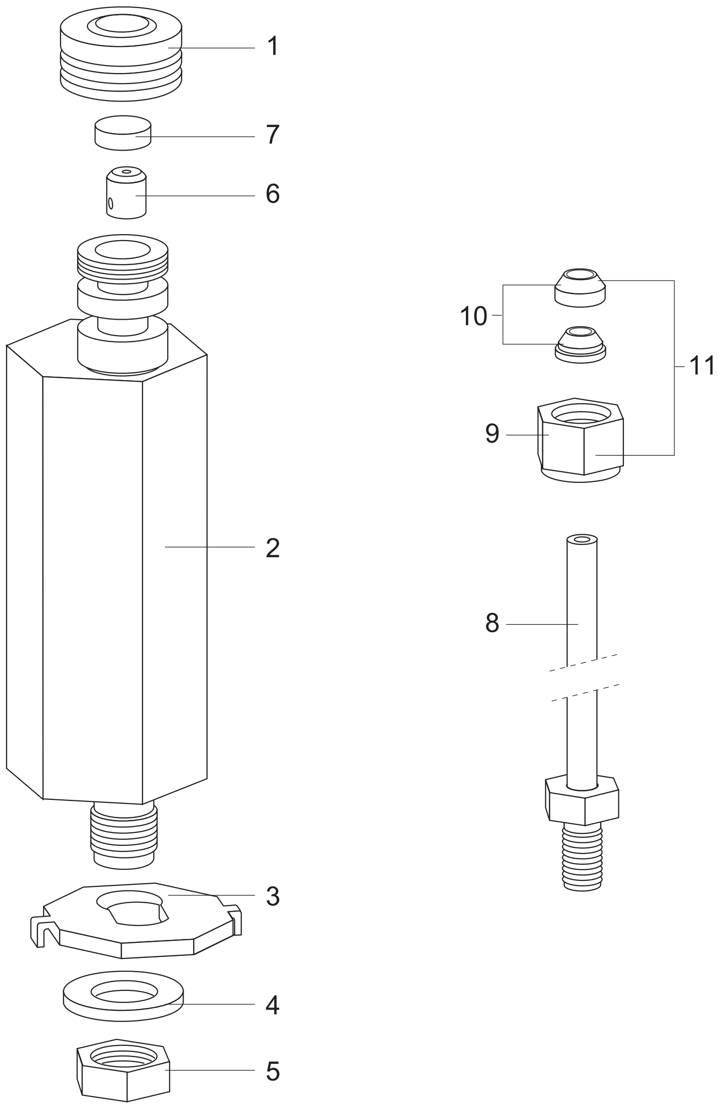


Table 7-1. Packed Column Injector parts identification table

No.	Description	P/N
1	Septum cap	
2	Injector body	
3	Anti-rotation plate	
4	Washer	
5	Retaining nut	
6	Spacer	453 020 48
7	Septum	313 032 00
	High Temperature septum	313 032 09
8	Adapter	
9	Retaining nut for adapter	350 401 18
10	Brass ferrule for adapter	290 341 37
11	Retaining nut and ferrule for adapter	350 201 17

Packed Column Injector Maintenance

The Packed Column injector and its interface module will normally be serviced by Thermo Fisher Scientific authorized technical personnel. In order to keep the injector operating at peak performance, you should periodically replace the septum.

When replacing the septum

The septum must be replaced at least every 200 injections, or every time a problem occurs related to a septum damage or wear. Refer to Chapter 19, *Analytical Troubleshooting*, for more information. Instructions are reported in the *Replacing the Septum* Operating Sequence on page 125.

OPERATING SEQUENCE

Replacing the Septum

Materials needed

- Non metallic sharp tool
- Septum (see # 7, Table 7-1 on page 123)
- Tweezers

CAUTION **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.

2. Scroll to `Temp` and press **OFF**.

OVEN		
Temp	80	80 <
Initial time		20
Ramp 1		Off

3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a PKD injector installed on the right channel is considered.

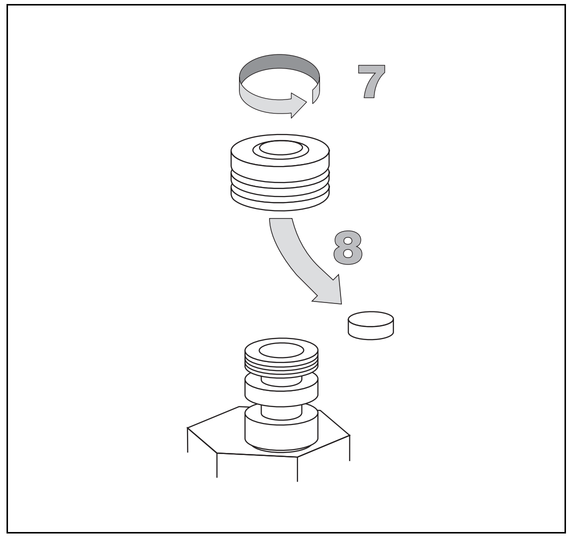
4. Scroll to `Temp` and press **OFF**.

RIGHT INLET (PKD)		
Temp	180	180 <
Pressure	100	100

5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (PKD)
Pressure           30.0   30.0
Col. flow          3.00        <
Lin. Veloc.            (60.9)
```

7. Unscrew the injector cap.
8. Remove the septum using a non-metallic sharp tool.

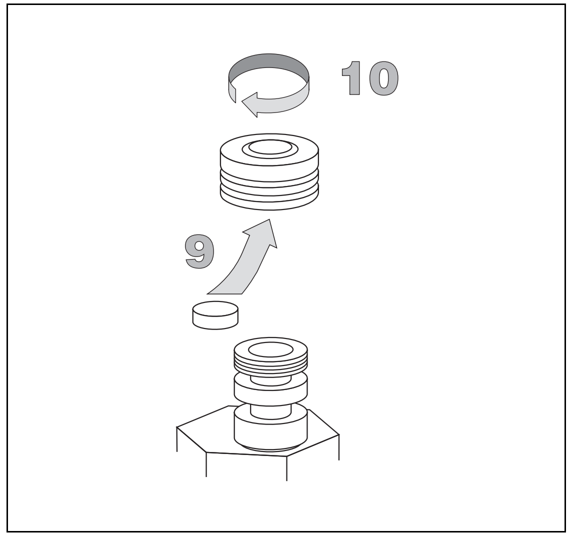


9. Insert a new septum in the septum cap.

> **CAUTION** **Use tweezers to avoid touching the septum with your fingers.**

10. Tighten the septum cap.

> **CAUTION** **Do not overtighten the injector cap. You could damage the septum and affect performance.**



11. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
12. Press **OVEN**, scroll to `Temp` and press **ON**.
13. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
14. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

8

Maintaining a Purged Packed Column Injector

Chapter at a Glance...

Operating Sequences

Purged Packed Column Injector

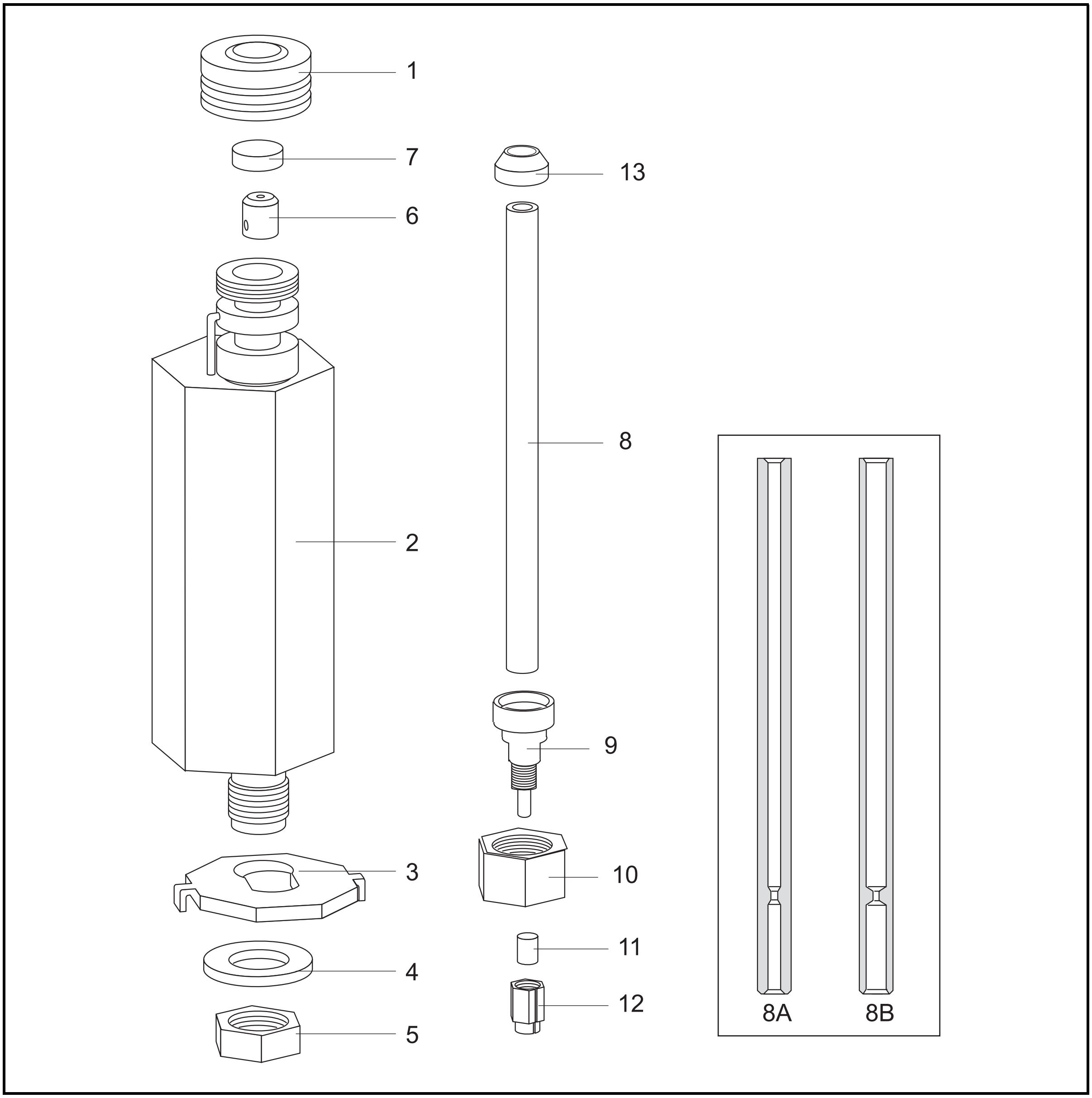


Table 8-1. Purged Packed Column Injector parts identification table

No.	Description	P/N
1	Septum cap	
2	Injector body	
3	Anti-rotation plate	
4	Washer	
5	Retaining nut	
6	Spacer	453 020 48
7	Septum (set of 10)	313 032 31
	High Temperature septum	313 032 09
8A	Liner, wide bore 2 mm	453 220 49
8B	Liner, wide bore 4 mm	453 220 50
9	Adapter for wide bore columns	347 003 03
10	Retaining nut	350 024 04
11	Graphite ferrule for 0.53 mm ID column	290 134 85
12	Fixing nut for column	350 324 23
13	Liner seal	290 334 05

Purged Packed Column Injector Maintenance

The Purged Packed Column injector and its interface module will normally be serviced by Thermo Fisher Scientific authorized technical personnel.

In order to operate at peak performances, the injector requires periodic maintenance from the user. This maintenance includes:

- the replacement of the septum
- the cleaning or replacement of the liner.

When replacing the septum

The septum must be replaced at least every 200 injections, or every time a problem occurs related to a septum damage or wear. Refer to Chapter 19, *Analytical Troubleshooting*, for more information. Instructions are reported in the *Replace the Septum* Operating Sequence on page 133.

It is good practice to replace the septum every time the liner is replaced.

When replacing the liner

The liner must be replaced periodically, depending on the number of injections performed and on the characteristics of the samples injected. The most common symptom indicating that the liner should be replaced is the appearance of tailing peaks in the chromatogram, particularly for polar compounds.

You can replace the liner with a new one or clean the liner and reinstall it.

Every time the liner is replaced, it is a good practice to replace the septum too.

OPERATING SEQUENCE

Replace the Septum

Materials needed

- Non metallic sharp tool
- Septum (see # 7, Table 8-1 on page 131)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
              OVEN
Temp                  80      80 <
Initial time                 200
Ramp 1                       Off
```

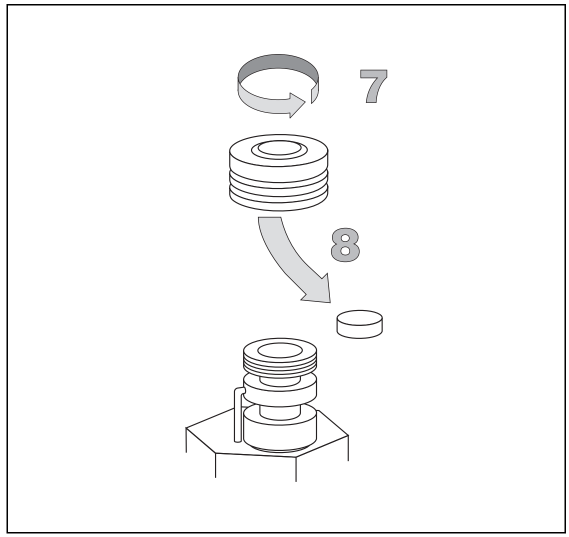
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a PPKD injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

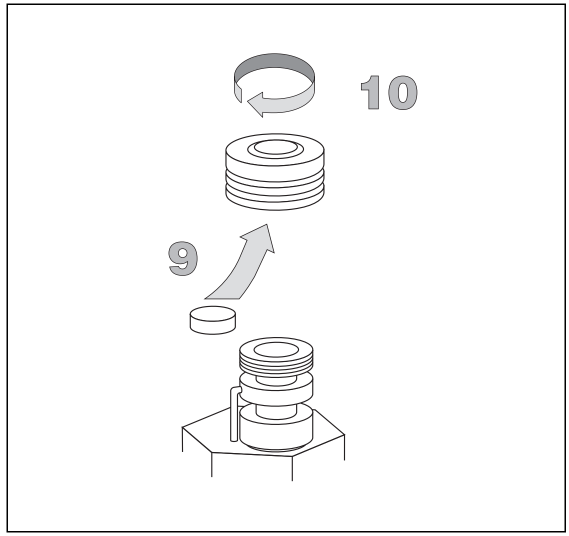
```
        RIGHT INLET (PPKD)
Temp                 200     200 <
Pressure              30      30
Mode:                 Wide Bore
```

5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (PPKD)
Pressure            30.0   30.0
Col. flow           3.00        <
Lin. Veloc.              (60.9)
```

7. Unscrew the injector cap.
8. Remove the septum using a non-metallic sharp tool.





9. Insert a new septum in the septum cap.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**

10. Tighten the septum cap.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**

11. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.
12. Press **OVEN**, scroll to `Temp` and press **ON**.
13. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
14. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Clean or Replace the Liner

Materials needed

- Glass liner (see item 8, Table 5-1)
- Ultrasonic bath
- Methanol/acetone mixture (1:1)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                OVEN
Temp                       80      80 <
Initial time                      200
Ramp 1                            Off
```

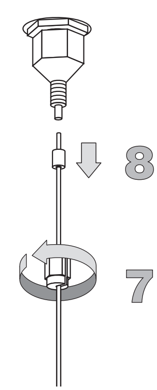
3. Press **LEFT INLET** or **RIGHT INLET** depending on which injector is operating. In the following example, a PPKD injector installed on the right channel is considered.
4. Scroll to `Temp` and press **OFF**.

```
      RIGHT INLET (PPKD)
Temp                200    200 <
Pressure             30     30
Mode:                Wide Bore
```

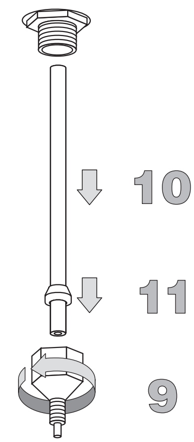
5. When the inlet reaches the room temperature, press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
     RIGHT CARRIER (PPKD)
Col. flow           3.0        <
Pressure             30     30
Flow mode             con pres
```

7. Unscrew the nut that retains the analytical column.
8. Remove the column with the inserted ferrule.



9. Unscrew the retaining nut at the bottom of the injector.
10. Remove the liner and the ferrule.
11. Remove the ferrule from the liner.

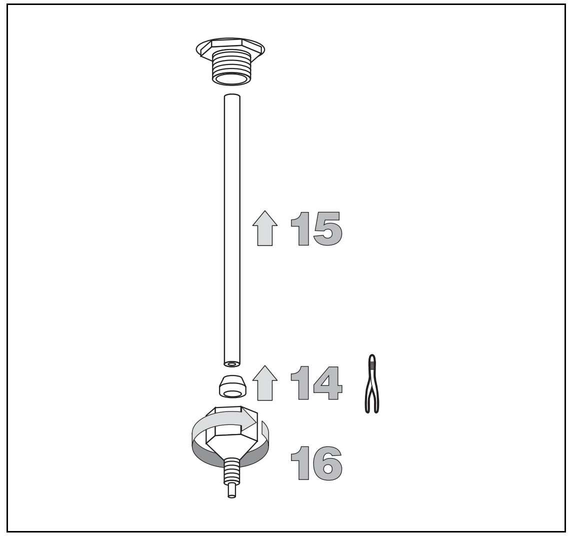


12. If you are using a new liner, go to step 14. If you are cleaning the liner, put the dirty liner into an ultrasonic bath filled with a methanol/ acetone mixture (1:1) and clean it for about half an hour.
13. Using tweezers, remove the liner from the bath and dry it with compressed clean air.

PRECAUTIONS

14. Insert a ferrule on the new (or cleaned) liner, using tweezers to hold it.

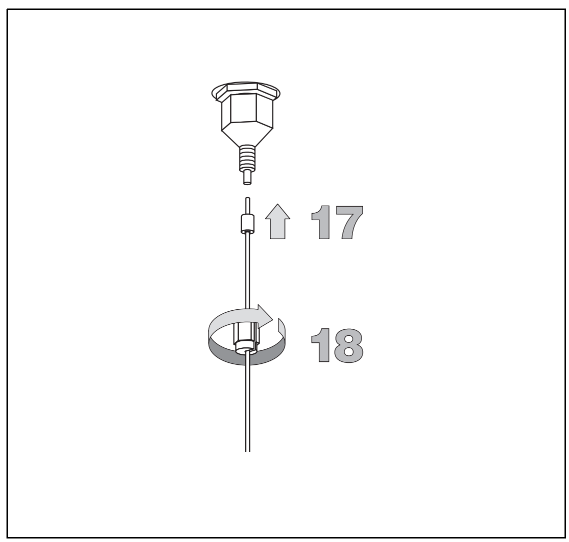
CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**



15. Insert the liner into the injector and push it gently upwards.
16. Tighten the retaining nut to close the bottom of the injector.

NOTE For trace analysis, you should pre-treat the liner with a suitable silylating reagent prior to inserting it into the injector.

17. Insert the analytical column with its ferrule into the bottom of the injector in its previous position. For detailed instructions, refer to the TRACE™ GC Ultra *Operating manual*.
18. Tighten the M4 nut that retains the column.



19. Press **LEFT CARRIER** or **RIGHT CARRIER,** depending on which injector is operating, scroll to `Pressure` and press **ON**.

20. Press **OVEN**, scroll to `Temp` and press **ON**.
21. Press **LEFT INLET** or **RIGHT INLET,** depending on which injector is operating, scroll to `Temp` and press **ON**.
22. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

9

Maintaining a PTV Injector

Chapter at a Glance...

Operating Sequences

PTV Injector

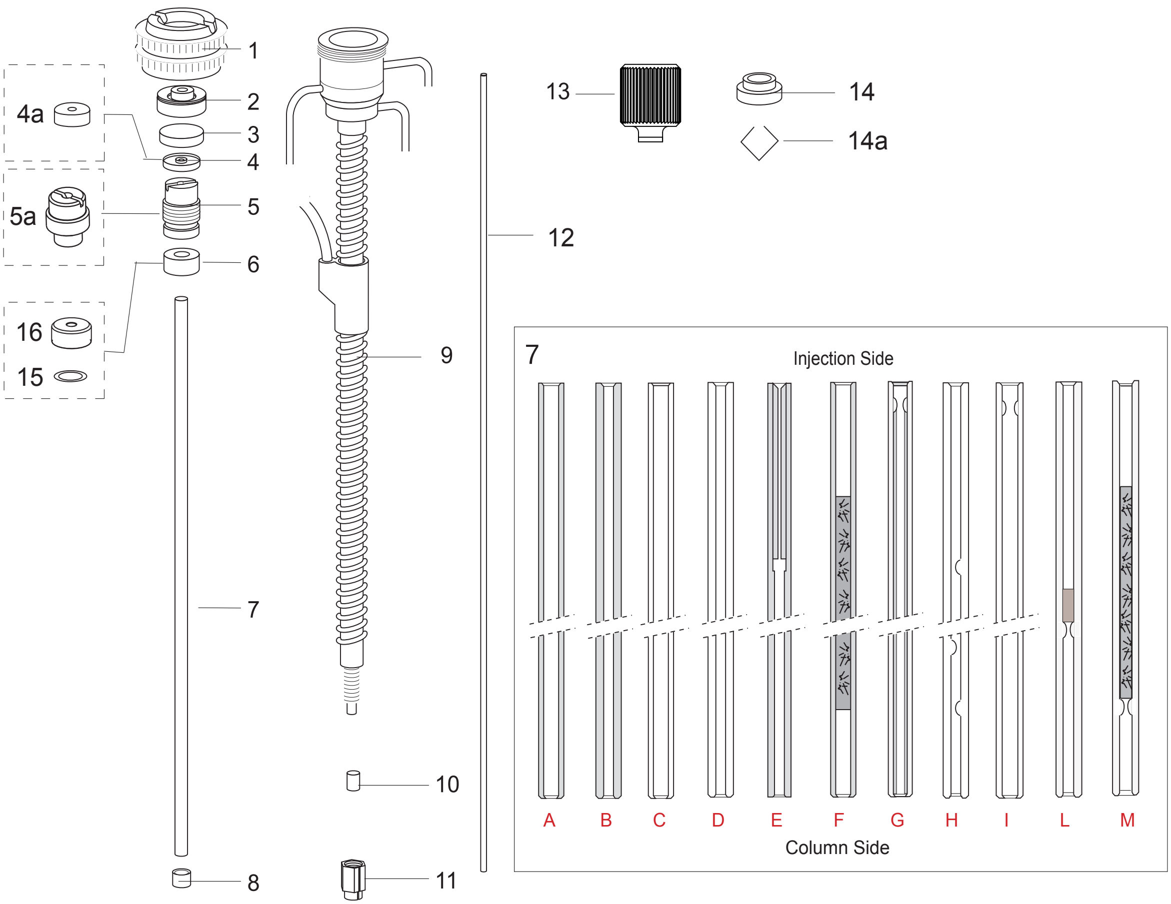


Table 9-1. PTV Injector parts identification table

No	Description	P/N
1	Septum cap	350 010 47
2	Septum holder	233 030 18
3	Septum	313 132 25
4	Septum support	350 054 35
4a	Septum support for PTV 816 RP-2004	350 054 36
5	Liner cap	290 042 77
5a	Liner cap for PTV 816 RP-2004	290 042 79
6	Liner seal	290 134 17
6a	Viton® O-ring for sinterized liner	290 313 05
	Kalrez® O-ring for sinterized liner	290 137 02
6b	Sealing cup for PTV liner. To be used with PN 290 313 05 or PN 290 137 02	290 034 20
7A	Silcosteel® liner, 2 mm ID	453 220 44
7B	Silcosteel® liner, 1 mm ID	453 220 46
7C	Glass liner, 2 mm ID	453 220 45
7D	Deactivated glass liner, 1 mm ID	453 220 54
7E	Silcosteel® liner for simile On-column injections	453 220 52
7F	Silcosteel® liner for Large Volume Injections	453 220 56
7G	Glass sintered deactivated liner for Large Volume Injections	453 220 60
7H	Deactivated glass liner with baffles	453 220 62
7I	Deactivated glass liner for split and splitless injections	453 220 57
7L	Glass liner, 2 mm ID; with silica wool	453 200 70
7M	2 mm ID Deactivated Glass Liner with 6 mm of silica wool	453 520 99
8	Spacer	290 142 78
9	PTV main body, including heater, thermocouple and filter	299 022 61
9	PTV 816 (RP 2004) main body, including heater, thermocouple without filter	299 022 63

Table 9-1. PTV Injector parts identification table (Continued)

10	Graphite ferrule for 0.2 mm ID column	290 134 89
	Graphite ferrule for 0.25 mm ID column	290 134 88
	Graphite ferrule for 0.32 mm ID column	290 134 87
	Graphite ferrule for 0.53 mm ID column	290 134 86
11	Fixing nut for column	350 324 23
12	Liner removing tool	398 014 04
13	Screwdriver	205 026 03
14	Ceramic washer	344 011 00
14a	Clip for ceramic washer	
	Viton® O-ring for sintered liner	290 013 05
	Kalrez® O-ring for sintered liner	290 037 02
	Sealing cup for PTV liner (to be used with 290 013 05 or 290 037 02)	290 034 20

PTV Injector Maintenance

The PTV injector will normally be serviced by Thermo Fisher Scientific authorized technical personnel. In order to operate at peak performances, the injector requires periodic maintenance from the user. This maintenance includes:

- the replacement of the septum
- the cleaning or replacement of the liner.

In addition, you will find information on how to adjust the quartz wool packing inside the liner used in the Large Volume version of the PTV injector (PTVLV).

When replacing the septum

If you use the suggested syringe (P/N 365 020 01), the septum of your PTV injector should be replaced after about 200 injections. The septum should be also replaced every time a problem that is related to septum damage or wear occurs (refer to Chapter 19, *Analytical Troubleshooting* for more information).

It is a good practice to change the septum with a new one every time the liner is replaced or cleaned. The operating sequence suggested for the replacement of the septum is reported on page 147.

When cleaning or replacing the liner

The liner must be replaced periodically, depending on the number of injections performed and the characteristics of the samples injected. Typical symptoms will indicate that the liner must be replaced (refer to Chapter 19, *Analytical Troubleshooting*). A dirty liner will generate tailing of sample peaks, particularly for polar compounds, or disappearing of some peaks.

You can replace the liner with a new one or clean the liner and reinstall it. Table 9-2 shows the available types of liners for the PTV injector.

Table 9-2. Available liners for PTV injector

Application	P/N
Silcosteel® liner, 2 mm ID (provided with the GC)	453 220 44
Silcosteel® liner, 1 mm ID (provided with the GC)	453 220 46
Glass liner, 2 mm ID	453 220 45
Glass liner, 1 mm ID	453 220 54
Silcosteel® liner for simile On-column injections	453 220 52
Silcosteel® liner packed with quartz wool for LV injections	453 220 56
Glass sintered deactivated liner for Large Volume Injections	453 220 60
Deactivated glass liner with buffles	453 220 62
Deactivated glass liner for split and splitless injections	453 220 57
Glass liner, 2 mm ID with glass wool	453 220 70

It is good practice to replace the septum every time the liner is cleaned or replaced. The operating sequence reported on page 151 describes how to:

- Remove the liner and the septum
- Clean and reinstall the liner

If a liner breaks inside the injector

When replacing or removing a glass liner, it might break inside the injector. In this case the broken parts of the liner must be removed from the injector, including the glass splinters that might fall into the lower part of the vaporization chamber. The suggested operating sequence is reported on page 157.

Adjusting the quartz wool packing

PTV injector arranged for Large Volume injections can be fitted with a liner (see *Available liners for PTV injector*, page 145, #6) packed with quartz wool. A casual increase of pressure inside the liner or removing the column without closing the carrier gas pressure could cause the packing glide towards the bottom of the liner, altering the conditions of sample reconcentration and transfer. In such a case, the user should replace the liner with a new one or, alternatively, adjust the quartz wool packing inside the liner in use. The suggested operating sequence for adjusting the packing is reported on page 165.

OPERATING SEQUENCE

Replace the Septum

Materials needed

- Non metallic sharp tool
- Septum (P/N 313 132 25)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                 OVEN
Temp                     50       50 <
Initial time                       1
Ramp 1                          10.0
```

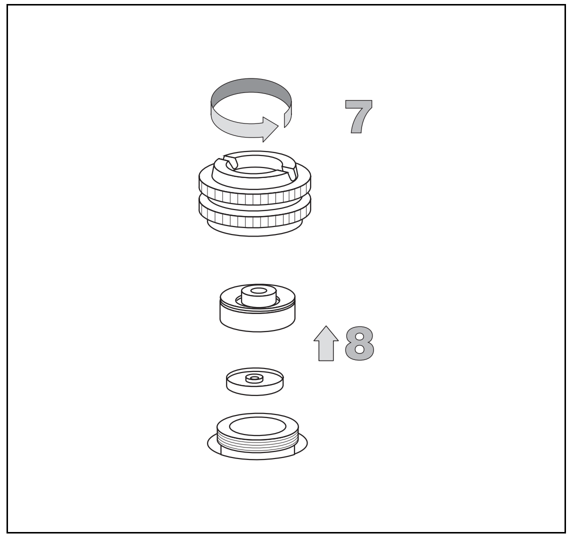
3. Press **RIGHT INLET**.
4. Scroll to `Temp` and press **OFF**.

```
            RIGHT INLET (PTV)
Temp                     50       50 <
Pressure                100      100
Mode                           split
```

5. When the inlet reaches the room temperature, press **RIGHT CARRIER**.
6. Scroll to `Pressure` and press **OFF**.

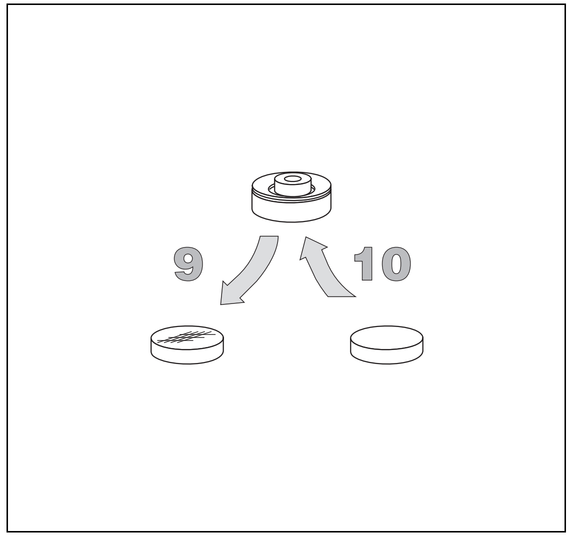
```
        RIGHT CARRIER (PTV)
Pressure              30.0   30.0
Col. flow             3.00        <
Lin. Veloc.                (60.9)
```

7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



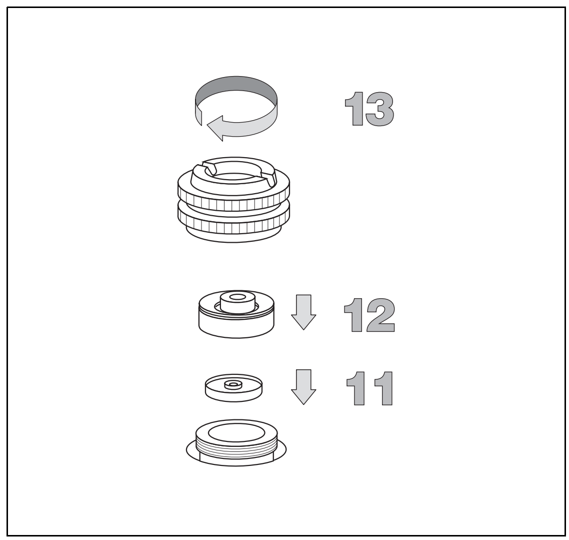
9. Remove the septum from the septum holder (use non-metallic tools)
10. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



11. Clean the septum support from possible fragments left by the septum and reinsert it into the injector.
12. Place the septum holder on the top of the septum support.
13. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



14. Press **RIGHT CARRIER,** scroll to `Pressure` and press **ON**.
15. Press **OVEN**, scroll to `Temp` and press **ON**.

16. Press **RIGHT INLET,** scroll to `Temp` and press **ON**.
17. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Clean or Replace the Liner

Materials needed

- Screwdriver (P/N 205 026 03)
- Liner (see Table 9-2, on page 145)
- Graphite seal (P/N 290 134 17)
- Ultrasonic bath
- Methanol/acetone mixture (1:1)
- Non metallic sharp tool
- Septum (P/N 313 132 25)
- Tweezers

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

PRECAUTIONS

WARNING! When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                OVEN
Temp                     50       50 <
Initial time                       5
Ramp 1                          10.0
```

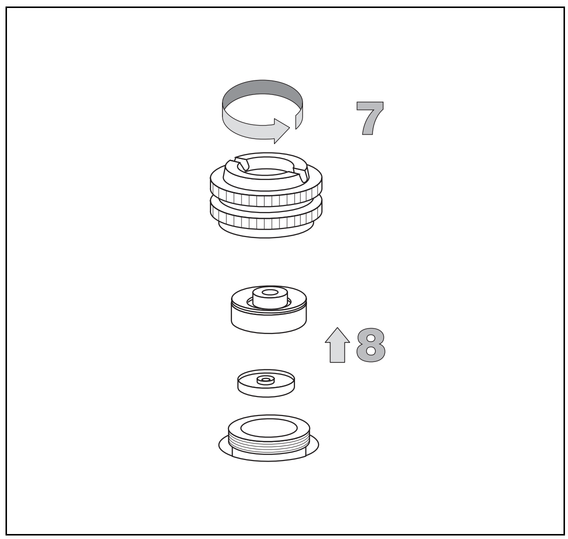
3. Press **RIGHT INLET**.
4. Scroll to `Temp` and press **OFF**.

```
        RIGHT INLET (PTV)
Temp                   50     50 <
Pressure              100    100
Mode                       split
```

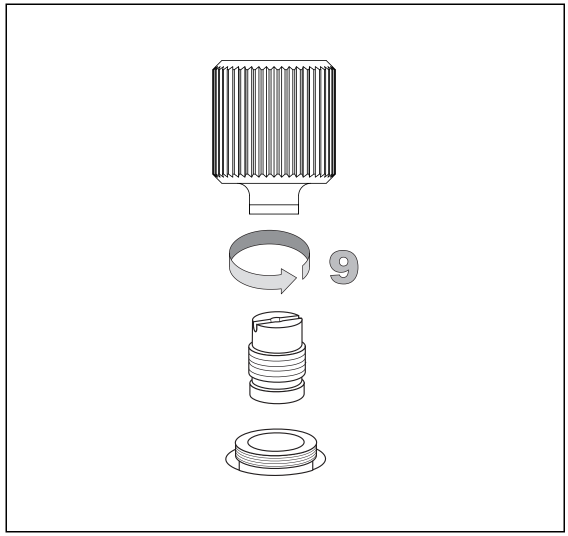
5. When the inlet reaches the room temperature, press **RIGHT CARRIER** depending on which injector is operating.
6. Scroll to `Pressure` and press **OFF**.

```
       RIGHT CARRIER (PTV)
Col. flow             2.5        <
Pressure              100    100
Flow mode               con pres
```

7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.

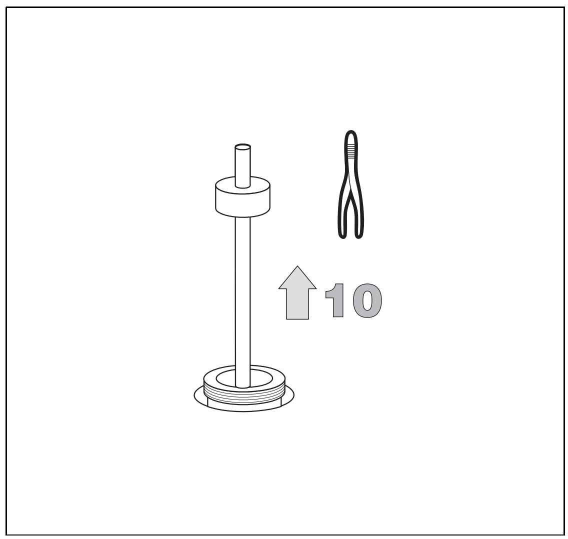


9. Use the screwdriver provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the liner with the graphite seal.

CAUTION **If you are using a glass liner, be careful not to break it inside the injector when removing it. Glass splinters might fall into the lower part of the vaporization chamber. If the liner breaks, refer to the *Replace a Broken Liner* Operating Sequence on page 157.**



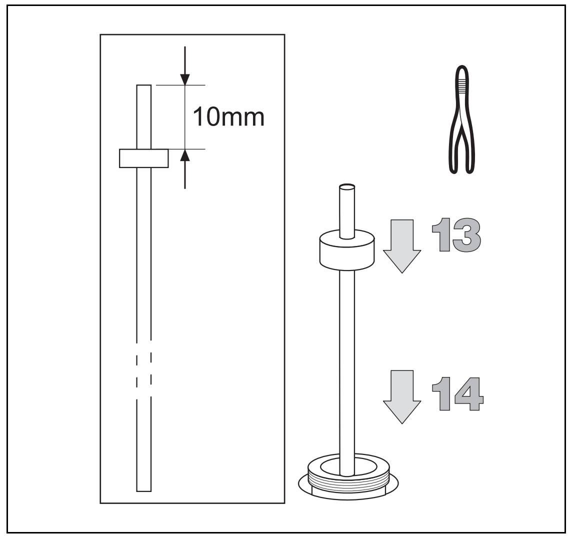
11. If you are going to replace the liner with a new one, go to step 13. If you are going to clean the liner, put the dirty liner into an ultrasonic bath filled with a methanol/acetone mixture (1:1) and sonicate it for about half an hour.

12. Using tweezers, remove the liner from the bath and dry it with compressed clean air.

PRECAUTIONS

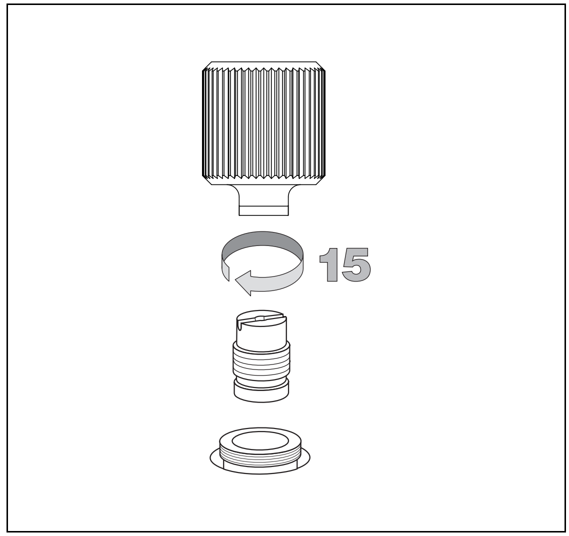
13. Holding the new (or cleaned) liner with tweezers, place a graphite seal over the liner, making sure to leave a distance of about 10 mm between the seal and the liner end.

14. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting.



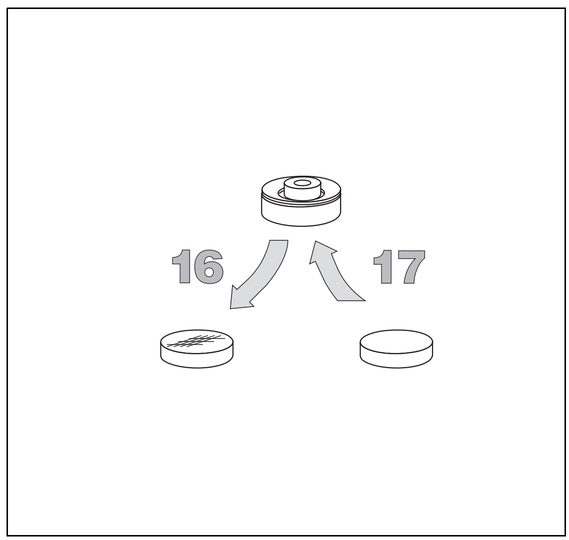
CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

15. Tighten the liner cap using the screwdriver provided with the GC.



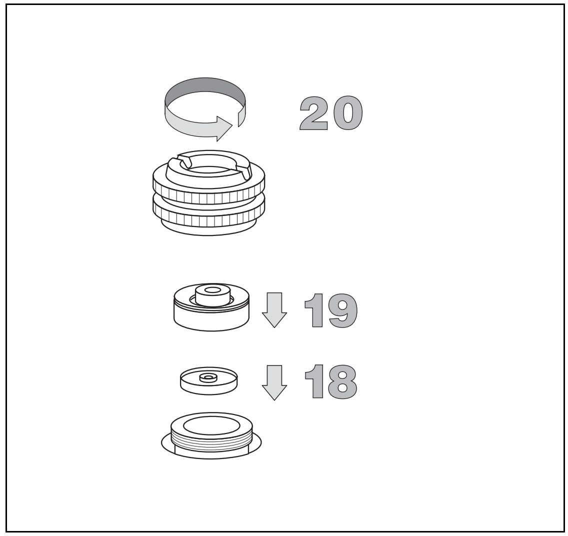
16. Remove the septum from the septum holder using a non-metallic sharp tool.
17. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



18. Place the septum support into its housing inside the injector.
19. Reinsert the septum holder with the septum.
20. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**



21. Press **RIGHT CARRIER,** scroll to `Pressure` and press **ON**.
22. Press **OVEN**, scroll to `Temp` and press **ON**.
23. Press **RIGHT INLET,** scroll to `Temp` and press **ON**.
24. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Replace a Broken Liner

Materials needed

- Screwdriver (P/N 205 026 03)
- Liner removing tool (P/N 398 014 04)
- Glass liner (see Table 9-2, on page 145)
- Tweezers

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                 OVEN
Temp                      50      50 <
Initial time                       1
Ramp 1                          10.0
```

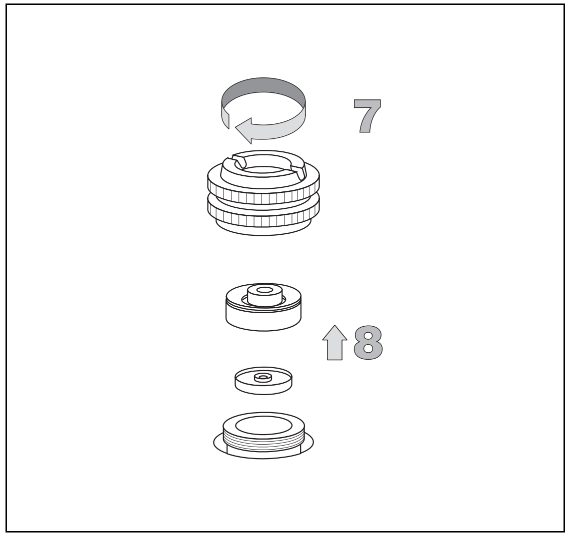
3. Press **RIGHT INLET**.
4. Scroll to `Temp` and press **OFF**.

```
           RIGHT INLET (PTV)
Temp                      50      50 <
Pressure                 100     100
Mode                           split
```

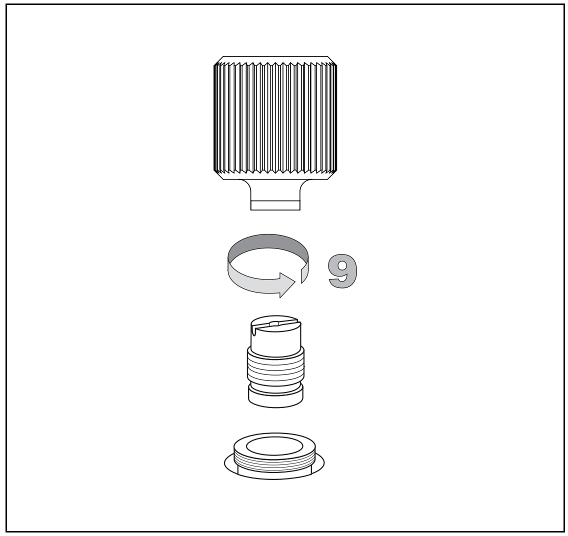
5. When the inlet reaches the room temperature, press **RIGHT CARRIER**.
6. Scroll to `Pressure` and press **OFF**.

```
        RIGHT CARRIER (PTV)
Col. flow              2.5        <
Pressure               100    100
Flow mode                con pres
```

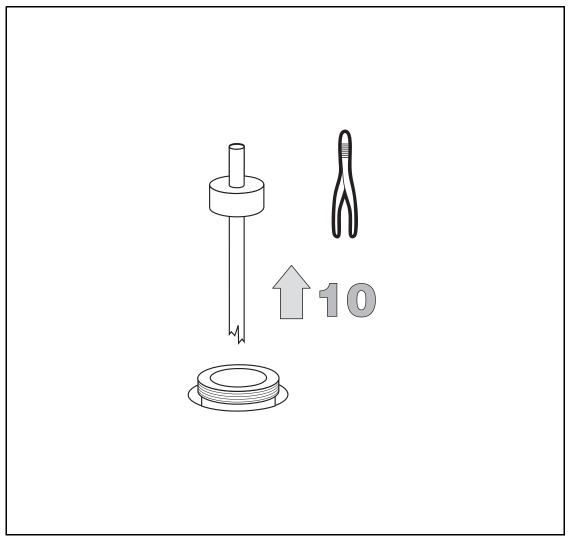
7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



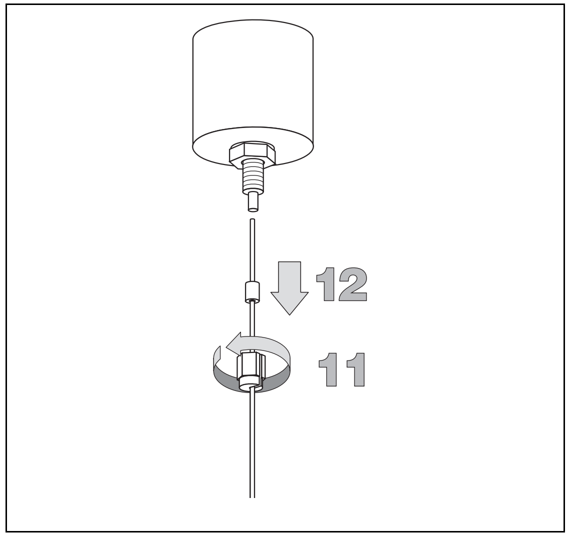
9. Use the screwdriver provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the upper part of the broken liner (with the graphite seal) from the injector.

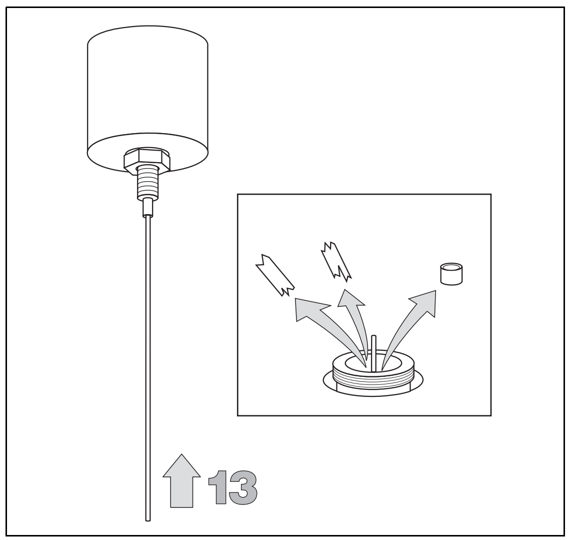


11. Unscrew the nut that retains the analytical column.
12. Remove the analytical column with its ferrule.

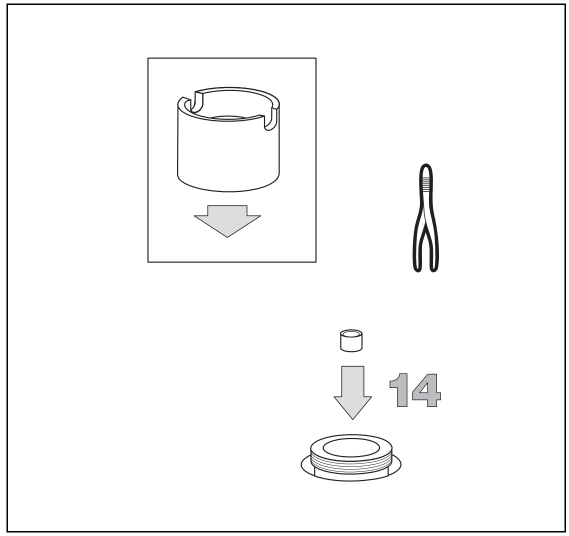


13. Inserting the steel thread provided specifically for this purpose, remove the possible glass fragments from the vaporization chamber pushing them out through the upper opening of the injector.

CAUTION **The spacer placed below the liner will also be ejected from the injector: pay attention not to lose it.**

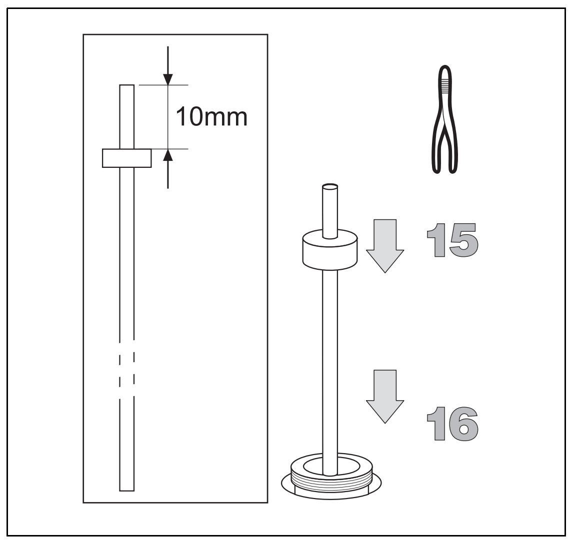


14. Use tweezers to reinsert the spacer inside the injector, complying with the correct orientation.



15. Holding a liner with tweezers, place a graphite seal over it, making sure to leave a distance of about 10 mm between the seal and the liner end.

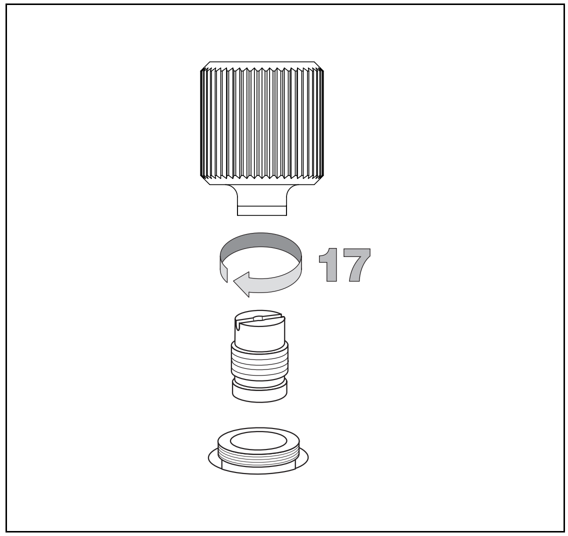
16. Using tweezers, insert the liner into the injector and push it gently towards the bottom.



CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

PRECAUTIONS

17. Tighten the liner cap using the screwdriver provided with the GC.

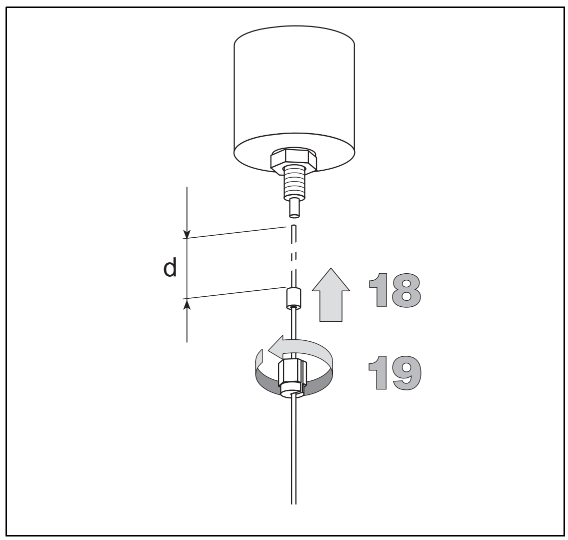


18. Insert the analytical column with its ferrule into the bottom of the injector in its previous position. Refer to the TRACE™ GC Operating Manual for instructions.

NOTE In split and splitless injection modes, the distance **d** between the top of the capillary column and the ferrule must be 3 cm.

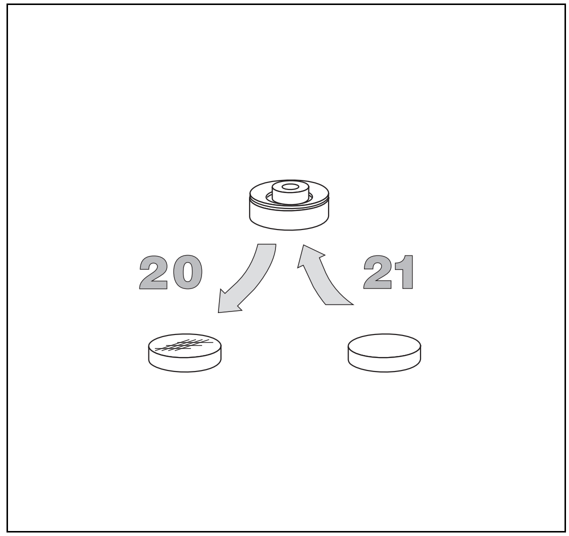
If you are using a **simile on-column** liner, in order to operate in on-column injection mode, insert the column as far as it goes.

19. Tighten the M4 retaining nut to hold the column in place.

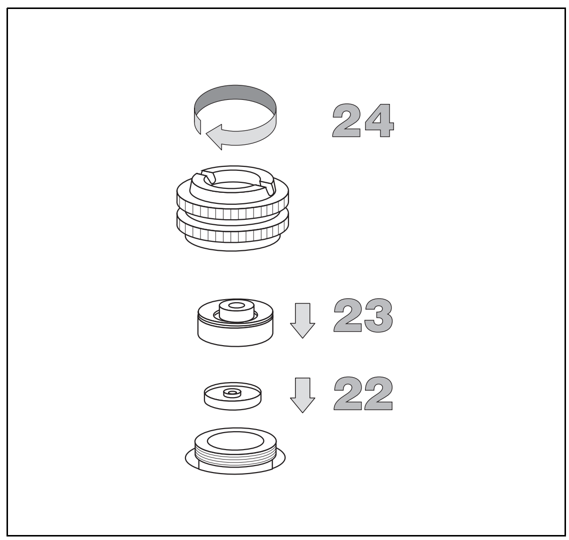


20. Remove the septum from the septum holder using a non-metallic sharp tool.
21. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



22. Place the septum support into its housing inside the injector.
23. Reinsert the septum holder with the septum.



24. Tighten the injector cap to finger tight.

CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**

25. Press **RIGHT CARRIER,** scroll to `Pressure` and press **ON**.
26. Press **OVEN**, scroll to `Temp` and press **ON**.
27. Press **RIGHT INLET,** scroll to `Temp` and press **ON**.
28. Perform an automatic leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

OPERATING SEQUENCE

Adjust a Quartz Wool Packing

Materials needed

- Screwdriver (P/N 205 026 03)
- Graphite seal (P/N 290 134 17)
- Non metallic sharp tool
- Thin (<2 mm diameter) non metallic bar (e.g. 1.6 mm PEEK tube)
- Septum (P/N 313 132 25)
- Tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the injector must be cooled to room temperature.**

PRECAUTIONS

1. While in stand-by condition, press **OVEN** to access the oven control table.
2. Scroll to `Temp` and press **OFF**.

```
                 OVEN
Temp                      50      50 <
Initial time                       5
Ramp 1                          10.0
```

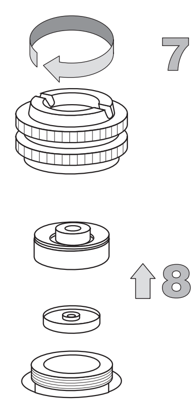
3. Press **RIGHT INLET**.
4. Scroll to `Temp` and press **OFF**.

```
          RIGHT INLET (PTV)
Temp                      50      50 <
Pressure                 100     100
Mode                           split
```

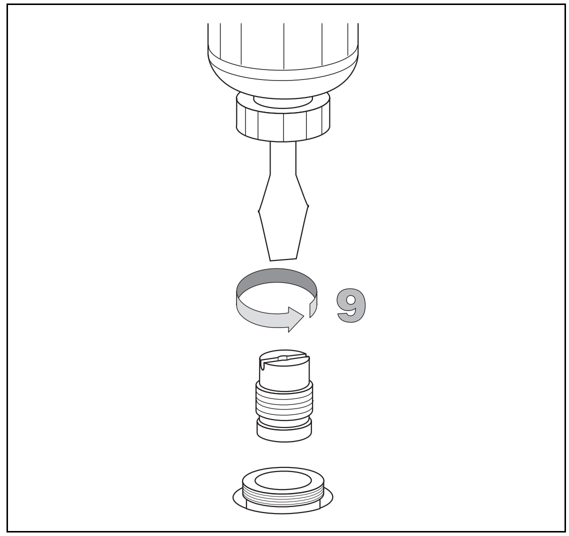
5. When the inlet reaches the room temperature, press **RIGHT CARRIER**.
6. Scroll to `Pressure` and press **OFF**.

```
      RIGHT CARRIER (PTV)
Col. flow            2.5       <
Pressure             100    100
Flow mode              con pres
```

7. Unscrew the injector cap.
8. Remove the septum holder with septum, then the septum support.



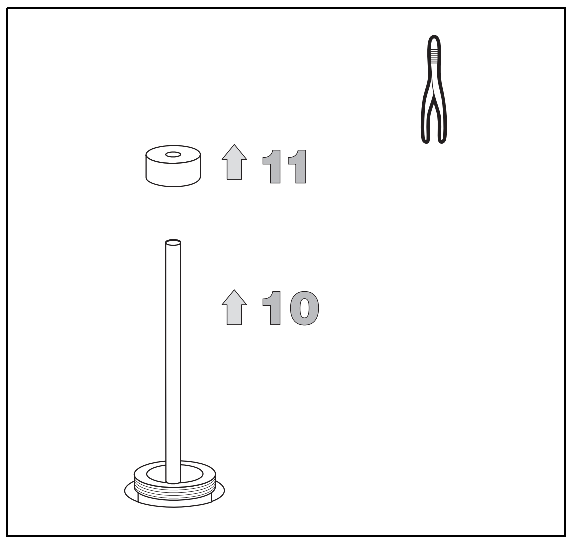
9. Use the screwdriver provided with the GC to remove the injector liner cap.



10. Use tweezers to remove the liner with the graphite seal.
11. Remove the graphite seal from the liner. Use tweezers to handle the liner.

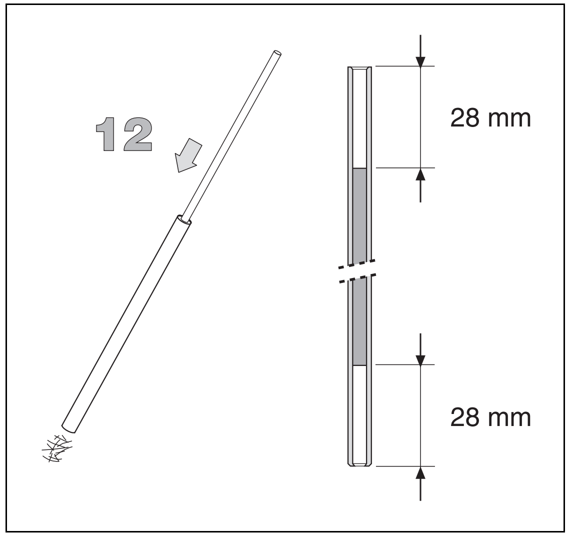
CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

PRECAUTIONS



12. Insert a thin, non metallic bar into the ends of the liner and push the quartz wool so to have a distance of about 28 mm between the packing and the ends of the liner.

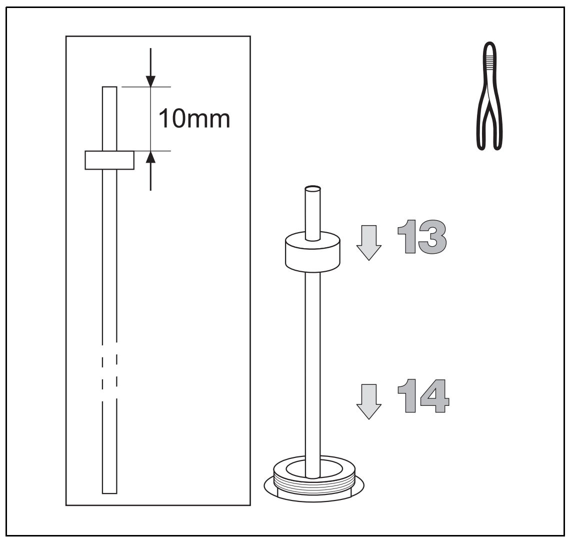
CAUTION **Do not compress the wool, to avoid carrier gas cannot flow through the liner.**



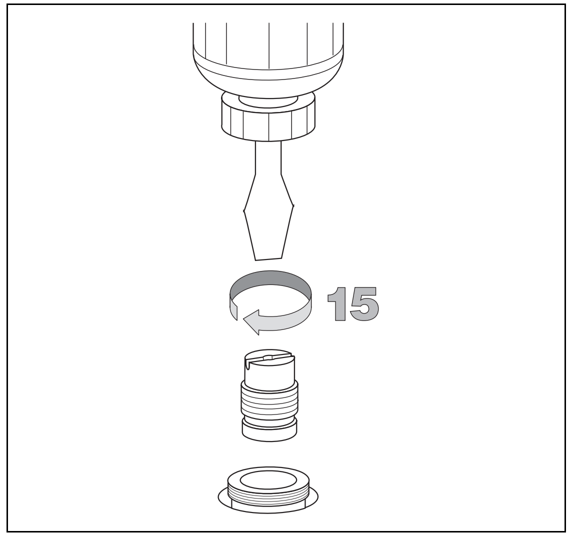
13. Place a new graphite seal over the liner, making sure to leave a distance of about 10 mm between the seal and the liner end.
14. Using tweezers, insert the liner into the injector and push it gently towards the bottom fitting.

CAUTION **Be careful not to damage the graphite seal or allow graphite to entering the liner. Should this occur, clean the liner with an inert gas.**

PRECAUTIONS

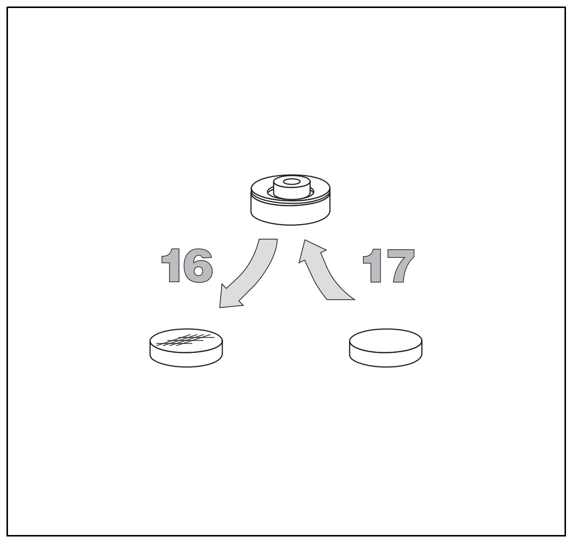


15. Tighten the liner cap using the screwdriver provided with the GC.

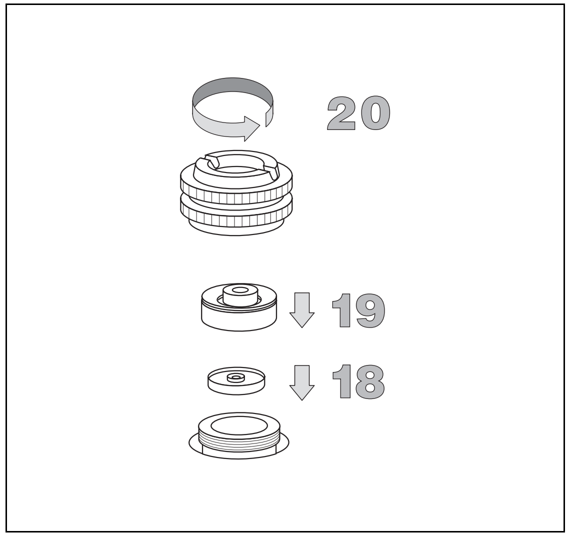


16. Remove the septum from the septum holder using a non-metallic sharp tool.
17. Insert a new septum into the septum holder.

CAUTION **Use tweezers to avoid touching the septum with your fingers.**



18. Place the septum support into its housing inside the injector.
19. Reinsert the septum holder with the septum.
20. Tighten the injector cap to finger tight.



CAUTION **Do not overtighten the injector cap. You could damage the septum and affect performance.**

21. Press **RIGHT CARRIER**, depending on which injector is operating, scroll to `Pressure` and press **ON**.
22. Press **OVEN**, scroll to `Temp` and press **ON**.
23. Press **RIGHT INLET**, depending on which injector is operating, scroll to `Temp` and press **ON**.
24. Perform a leak check according to *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181.

PTV Troubleshooting

Sensitivity

Low sensitivity is commonly caused by incorrect operating conditions. It might also be due to a dirty liner or to a liner wrong sized with regards to the volume of the sample injected.

Lack of sensitivity for selected compounds can also be related to discrimination phenomena induced by inadequate injection conditions.

Discrimination

Possible discrimination of the heavy or volatile fraction of the sample may be caused by operating conditions set incorrectly with regard to the chosen injection mode.
Discrimination could also be caused by:

- degradation of the thermally labile compounds due to the cathalytic effect of bad deactivated quartz wool inside the liner
- by-products originated in previous injections that have been not completely eliminated from the liner during the cleaning phase or the cleaning operating sequence.

The *PTV Troubleshooting Table* on page 172 reports a list of possible symptoms, their probable causes and the suggested remedies when using a PTV injector.

Large Volume PTV

The *PTVLV Troubleshooting Table* on page 176 will help you in troubleshooting a PTV injector adapted for Large Volume injections.

Troubleshooting the PTV Injector

Table 9-3. PTV Troubleshooting Table

Symptom	Diagnosis	Remedy
Discrimination of heavy compounds in splitless mode	Transfer temperature too low	Set `Transfer temp` to a higher value.
	Transfer time too low	Set `transfer time` or `cleaning time` to a value close to the whole GC run time.
	Splitless time too short	Set `Splitless time` to a higher value.
	Initial temperature too high with regard to the solvent boiling point	Set `Inject temp` to a value closer to the solvent boiling point.
	Flooding of the liner due to low injection temperature	Set `Inject temp` to a higher value. If the symptom does not disappear, insert quartz wool in the liner. Refer to *Adjust a Quartz Wool Packing* Operating Sequence on page 165.
	Flooding of the liner due to injected volume too large	Reduce the amount of sample to be injected. Replace the liner with one of adequate diameter. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Flooding of the liner due to insufficient size	Replace the liner with one of larger diameter. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Dirty liner	Clean or replace the liner. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.

Table 9-3. PTV Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Discrimination of heavy compounds in splitless mode	Liner not suitable for the actual sample	Replace the liner with an adequate one. A lower diameter liner would improve the sample transfer. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Quartz wool causing excessive retention of high molecular compounds	Replace the packed liner with a new one filled will quartz wool. Replace the packed liner with an empty one. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
Discrimination of volatile compounds in splitless mode	Initial temperature too high	Set `Inject temp` to a lower value.
	Liner diameter too small	Replace the liner with one of larger diameter. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Volatiles are eliminated through the purge line	Set `Const purge flow?` to `N`, then set `Stop purge for` to a value corresponding to `Splitless time`
Discrimination of volatile compounds in solvent split mode	Initial temperature too high	Set `Inject temp` to a lower value.
	Injection and evaporation time are set as to exceed the time required for the solvent elimination	Set `Inject time` and `Evaporation time` to adequate values.
	Split flow set too high	Set `Split flow` to a lower value.
	Lack of an adequate quartz wool packing inside the liner	Insert quartz wool in the liner.

Table 9-3. PTV Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Discrimination in split mode	Transfer temperature too low	Set `Transfer temp` to a higher value.
	Transfer time too low	Set `transfer time` or `cleaning time` to a value close to the whole GC run time.
	Initial temperature too low	Set `Inject temperature` to an higher value.
	Injected volume too large	Reduce the amount of sample to be injected. Replace the liner with the 2 mm ID version. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Lack of an adequate quartz wool packing inside the liner	Replace the packed liner with a new one filled will quartz wool. Fix the quartz wool packing inside the liner. Refer to *Adjust a Quartz Wool Packing* Operating Sequence on page 165.
Excessive broadening of the solvent peak. Volatile compounds obscured. Lack of sensitivity	Injection pressure set too high	Set `Inject pressure` to a lower value.
Sample degradation	Dirty liner	Clean or replace the liner. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Transfer temperature set too high	Set `Transfer temp` to a value adequate to the nature of the sample compounds.

Table 9-3. PTV Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Sample degradation	Liner diameter too large	Replace the liner with one of smaller diameter to improve transfer efficiency. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Cathalytic and thermal degradation of sensitive compounds caused by the quartz wool inside the liner	Use a new packed liner. If the symptom does not disappear, use an empty liner. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.

Table 9-4. PTVLV Troubleshooting Table

Symptom	Diagnosis	Remedy
Poor reproducibility of results	Transfer temperature too low	Set `Transfer temp` to a higher value.
	Transfer time too low	Set `transfer time` or `cleaning time` to a value close to the whole GC run time.
	Injection speed set too high	Reduce injection speed.
	Injected volume too large	Reduce the amount of sample to be injected.
	Initial temperature set too low	Set `Inject temperature` to an higher value.
	Injection pressure set too high	Set `Inject pressure` to a lower value.
	Lack of an adequate quartz wool packing inside the liner	Fix the quartz wool packing inside the liner. Refer to *Adjust a Quartz Wool Packing* Operating Sequence on page 165. Replace the liner with another filled with quartz wool. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
Discrimination of volatile compounds	Injection speed set too low	Set `Inject speed` to a higher value.
	Initial temperature set too high	Set `Inject temperature` to a lower value.
	Split flow set too high	Lower the split flow.
	Lack of an adequate quartz wool packing inside the liner	Fix the quartz wool packing inside the liner. Refer to *Adjust a Quartz Wool Packing* Operating Sequence on page 165. Replace the liner with another filled with quartz wool. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.

Table 9-4. PTVLV Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Sample degradation	Cathalytic degradation of sensitive compounds caused by the quartz wool	Use an empty deactivated liner reducing the volume injected. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.
	Cathalytic degradation of sensitive compounds caused by-products accumulated on the quartz wool	Replace the packed liner with a new one. Refer to *Clean or Replace the Liner* Operating Sequence on page 151.

Table 9-5. PTV General Troubleshooting

Symptom	Diagnosis	Remedy
Broadening or loss of peaks at the end of the chromatogram	Injector temperature too low	Increase the injector temperature
	Premature cooling of the injector before the end of the analysis	Keep the transfer phase up to the end of the analysis, unless the cleaning phase is set.

10

Ensuring Tightness

Chapter at a Glance...

Operating Sequences

Keeping Leaks under Control

In this chapter you will find instructions about the operations needed to keep under control the tightness of your chromatographic system. The sequences of operations to be used to find and correct possible leaks in the system are organized according to the possible instrument configurations (i.e. the type of column installed and the type of gas control system).

If the Leak Test (or any significant symptom) has notified a possible leak in the system you should:

- Locate the leaks from the carrier gas inlet to the detector base body.
- Check the accessible, critical connections (column to injector, column to detector, split and purge valves, septum caps).

A possible leak source may be the gas bottles/gas chromatograph connections. Check these lines before the others, if specific symptoms indicate that the leak is outside the gas chromatograph.

DCC-equipped systems

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. In particular an assisted **Leak Check** procedure is available to easily verify the tightness of the system.

Leak Check

Perform the *Leak check* at the desired pressure following the instructions reported in *Performing a Leak Check (Capillary Column)* operating sequence.

OPERATING SEQUENCE

Performing a Leak Check (Capillary Column)

This procedure allows to check for leaks on the injector side only and it is suitable only for capillary column.
Before starting this sequence, install the column into the injector port only, leaving free the column end.
This Leak Check procedure can be easily performed by means of the provided column-flow meter connector shown in Figure 10-1.

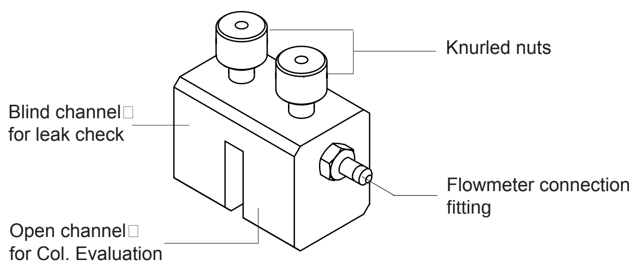


Figure 10-1. Column-flow Meter Connector

Materials needed:

- Leak detector (Thermo Scientific GLD Pro, or equivalent)
- 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 (not used in this procedure).

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 10-2.

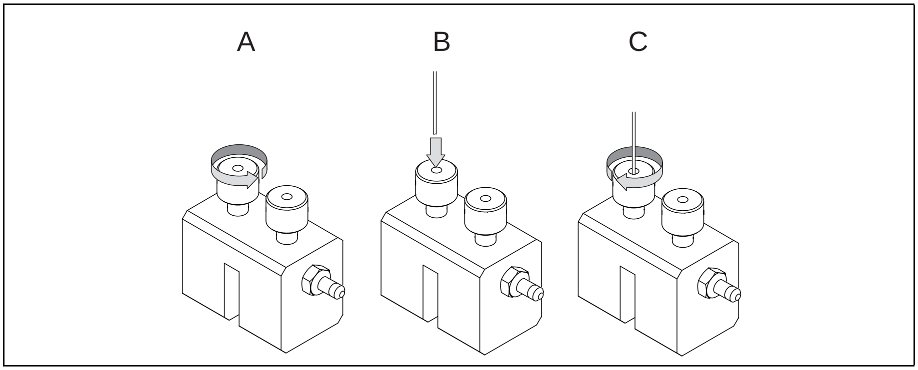


Figure 10-2. Leak Check

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 column¹
Left column¹                <
```

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 at press          200
Sensitivity             5.0
```

6. In **Check at 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
```

NOTE 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** value set, 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.

11. If leaks are detected, check the accessible, critical connections with an electronic leak detector to locate the possible leaks.
12. Eliminate the leaks and repeat the leack check procedure.
13. If the leaks cannot be located, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

OPERATING SEQUENCE

Performing a Leaks Check (Capillary/Packed/UFM Columns)

This procedure allows to check for leaks on both injector and detector sides and it is suitable for capillary, packed and UFM columns.

Before starting this sequence, install the column into the injector port and the detector base body.

Materials needed:

- Detector base body blind cap (for FID, NPD, FPD, PID, PDD)
- 6MB blind nut (for ECD)
- Leak detector (Thermo Scientific GLD Pro, or equivalent)

1. In case the column is already installed, cool down the injector and the detector base body to room temperature.
2. *Only for FID, NPD, FPD:* remove the detector. Do not remove ECD detector.
3. *Only for FPD:* remove the jet. In case of capillary columns, use typewriter correction fluid to mark the position of the column entering the detector base body. Lower the column end down to the top of the detector base body, then tighten the M4 fixing nut to restore system tightness.
4. Finger tighten the relevant blind cap (fitted with a silicon seal) on the detector base body, to seal the column flow path.
5. *Only for ECD:* remove the chimney and seal the line with the relevant blind 6MB nut.
6. Turn off any H_2, air and make-up gas flows. Do not close the detectors gases supplies.
7. Press **LEAK CHECK** to enter **Leak Check** menu.

```
      LEAK CHECK COLUMN
Right column[1]
Left column[1]              <
```

1. This item appears if the relevant channel is present and configured.

8. Scroll to the channel of interest and press **ENTER** to open the following menu:

.

```
     L. COL. LEAK CHECK

Start Leak Check
Leak Check Settings
```

9. Scroll to **Leak Check Settings** and press **ENTER** to open the relevant menu.

```
        L. COL. SETT.

Check press           200
Sensitivity           5.0
```

10. 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).

11. 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).

12. Press **CLEAR** to exit **Leak Check Settings** menu and return to the previous menu.

```
     L. COL. LEAK CHECK

Start leak check
Leak Check settings
```

13. 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
```

NOTE To abort Leak Check, press **STOP**. A relevant message will be displayed.

14. 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.

15. If leaks are detected, check the accessible, critical connections with an electronic leak detector to locate the possible leaks.
16. Eliminate the leaks and repeat the leak check procedure.
17. If the leaks cannot be located, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information..

OPERATING SEQUENCE

Leak Test the Lines from the Gas Source to the GC

1. Switch off the gas chromatograph power supply.
2. Turn on all gas supply lines to the GC, setting the input pressure to 420 kPa (60 psi). Allow the gas lines to pressurize for several seconds.
3. Turn off the input gas source. The pressure value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present. In this case:
4. Check the connections with the Thermo Scientific GLD Pro leak detector, or equivalent handheld electronic leak detector, to find possible leaks.

Multiple systems

If more than a system is attached to the same gas source, you will need to isolate each GC system.

1. Install a cut-off valve and pressure gauge with an 1/8 inch input tee in the gas line leading to the GC that you are leak testing, as shown in Figure 10-3. Be sure the pressure gauge is connected in line between the on/off valve and the GC.
2. Turn on the gas supply and allow the system to pressurize.
3. Turn off the input gas source. The pressure value should not change more than 5% in 10 minutes. If the value drops down immediately, one or more leaks are present in the plumbing from the cut-off valve to the GC. In this case:
4. Check the connections of the line you are testing with the leak detector to find the leaks.

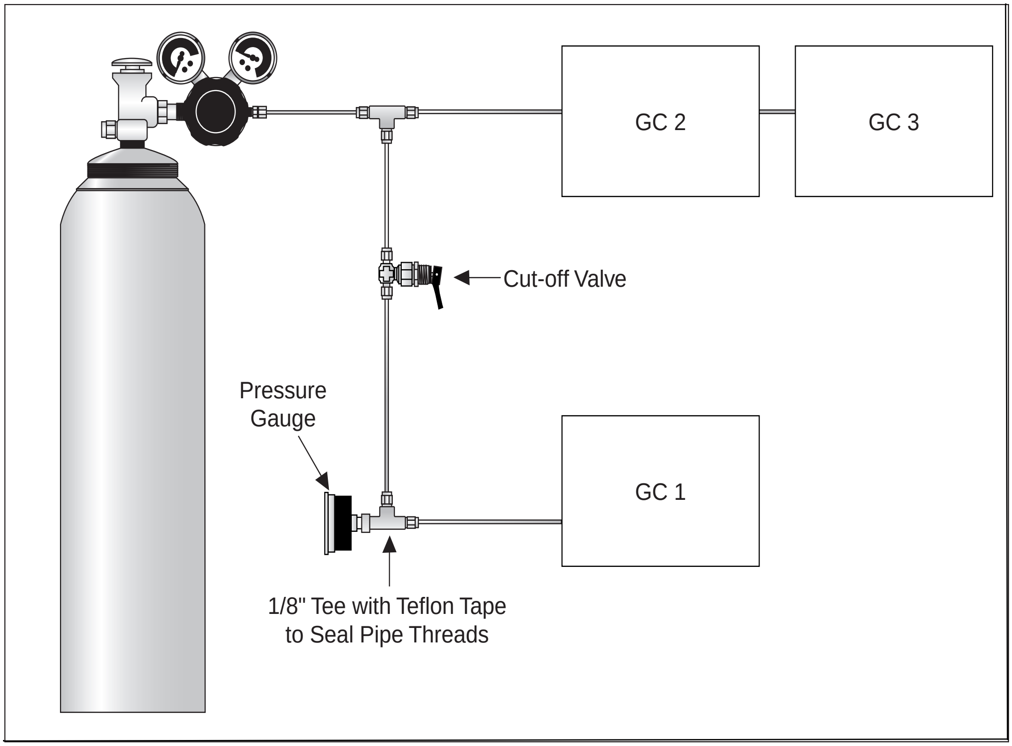


Figure 10-3. Testing Multiple Systems

11

Maintaining a FID

Chapter at a Glance…

Operating Sequences

Flame Ionization Detector (FID)

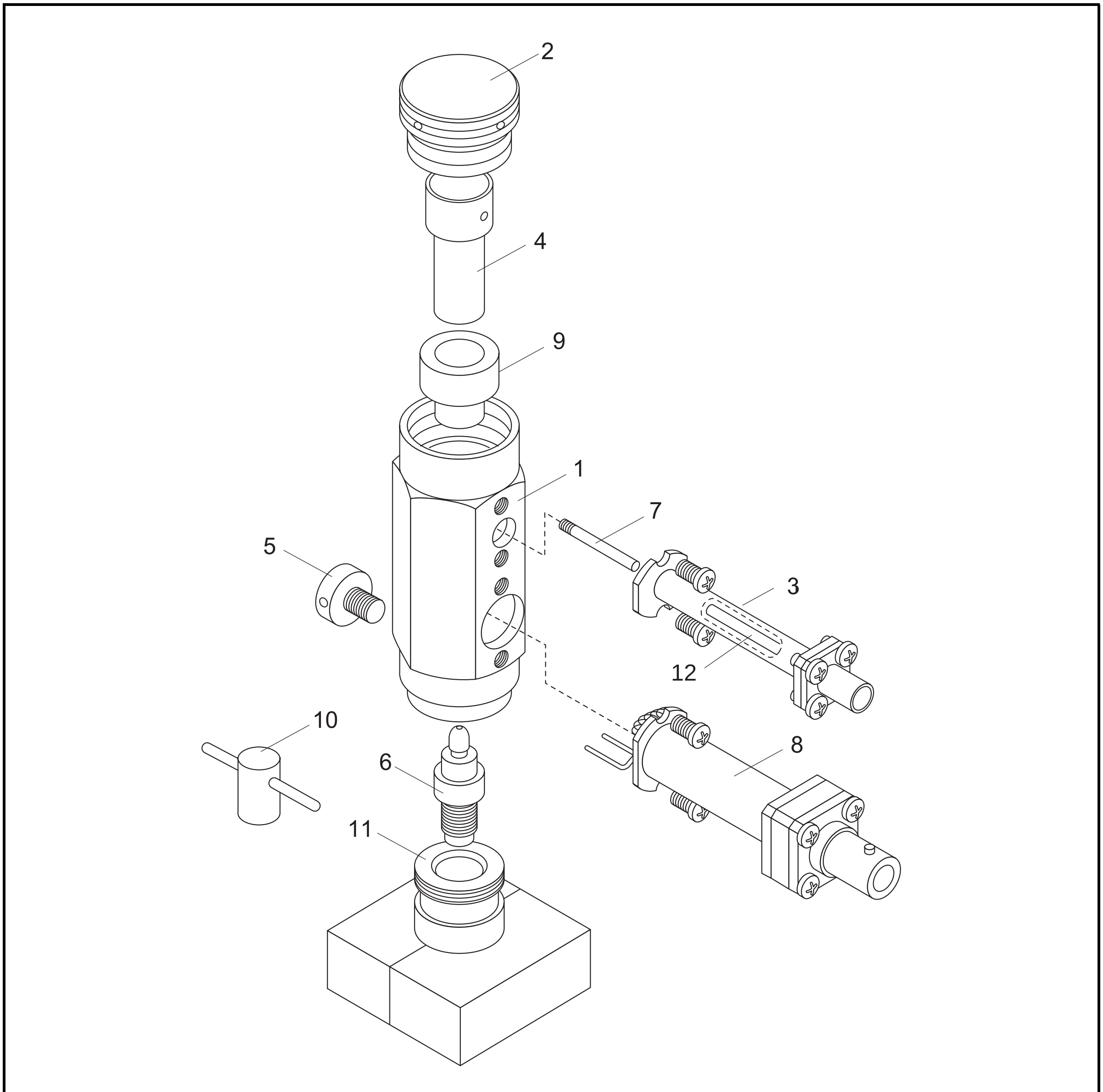


Table 11-1. Flame Ionization Detector (FID) parts identification table

No.	Description	P/N
1	Detector body	
2	Chimney	
3	Electrical connection assembly	
4	Collecting electrode	259 011 15
5	Retaining screw	311 060 08
6	Jet	404 043 01
7	Collecting electrode pin	206 016 02
8	Flame ignition coil/polarization assembly	206 016 03
9	Ceramic insulator	302 023 10
10	Jet removing tool	205 019 00
11	Detector base body	
12	Spring contact	206 031 00

FID Maintenance

To ensure optimum performance of the FID, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector needs cleaning.

To properly maintain the FID, you should perform the following cleaning or replacement sequences:

- cleaning the jet
- replacing the jet
- cleaning the collecting electrode
- replacing the collecting electrode
- replacing the ignition assembly

OPERATING SEQUENCE

Clean the Jet

Materials needed:

- Screwdriver
- Tool for extracting the jet (P/N 205 019 00)
- Forceps or tweezers
- Distiller water
- Clean compressed air
- GC-grade methanol
- Clean paper towel

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

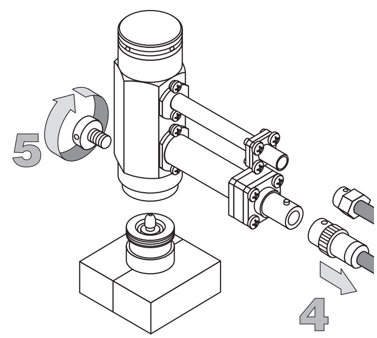
PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FID detector, to access the FID control table. In the following example, a FID installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

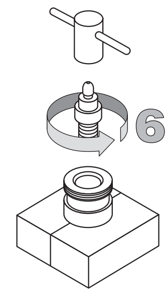
RIGHT DETECTOR (FID)		
Flame		Off <
Base temp	300	300
Signal pA		(5.4)
Ign. tresh		2.0
Flameout retry		Off
H2	35	Off
Air	350	Off
Mkup N2	30	30

4. Disconnect the signal and ignition polarization cables from the detector.
5. Loosen the fixing screw on the front of the detector cell and remove it.

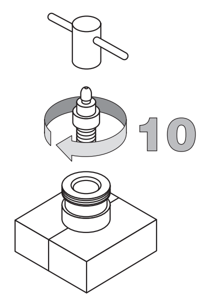


6. Using the tool provided with the GC, unscrew the jet.
7. Mechanically remove any contaminating material and sweep with clean compressed air if necessary.
8. Handling the jet with forceps or tweezers, rinse the jet with distilled water, then with methanol.
9. Place the jet on a paper towel and let it air dry.

PRECAUTIONS



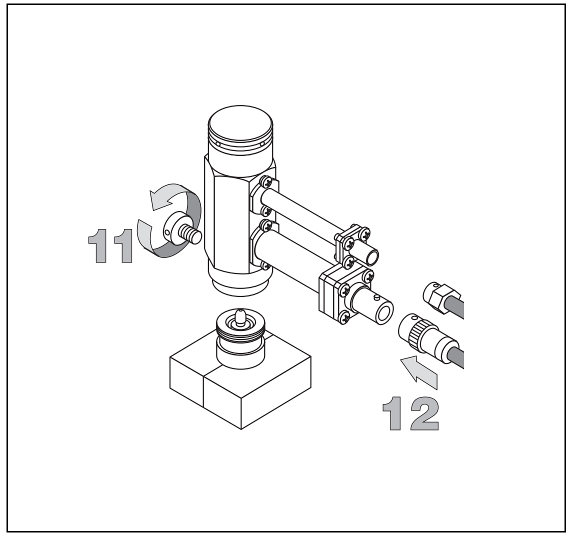
10. When the jet is dry, place the jet into the housing and tighten it with the proper tool. Ensure the jet is perfectly vertically aligned to avoid damaging its ceramic part.



11. Place the detector cell on the detector base body and tighten the fixing screw.

12. Reconnect the signal and ignition cables to the detector.

13. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Jet

Materials needed

- Screwdriver
- Tool for removing the jet (P/N 205 019 00)
- Forceps or tweezers
- Jet for FID (P/N 404 043 01)

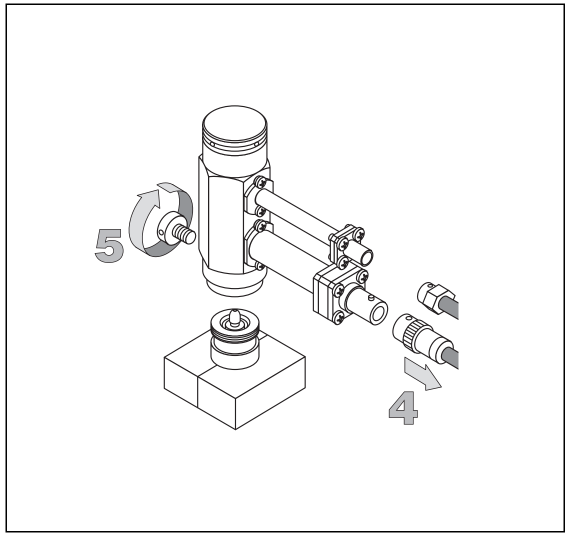
WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

PRECAUTIONS

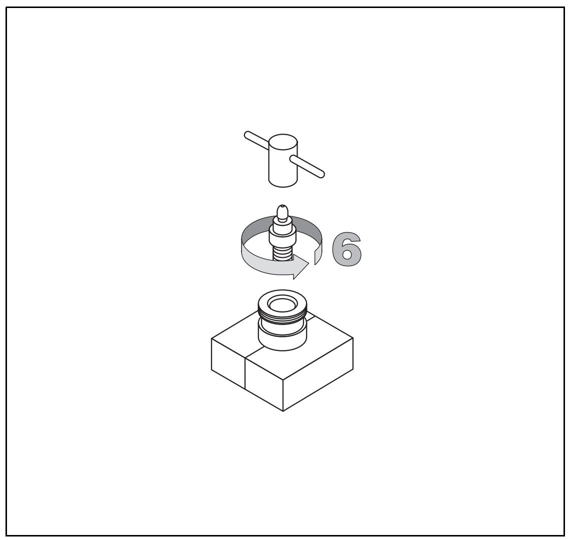
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FID detector, to access the FID control table. In the following example, a FID installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

```
      RIGHT DETECTOR (FID)
Flame                       Off <
Base temp           300     300
Signal pA                 (5.4)
Ign. tresh                  2.0
Flameout retry              Off
H2                   35     Off
Air                 350     Off
Mkup N2              30      30
```

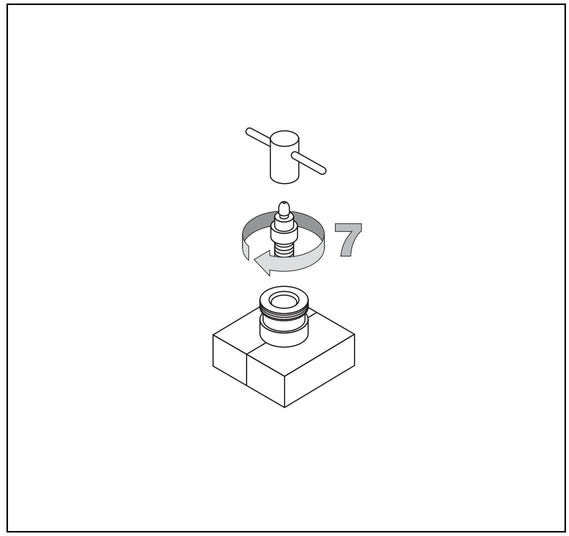
4. Disconnect the signal and ignition cables from the detector.
5. Loosen the fixing nut on the front of the detector cell and remove it.



6. Using the tool provided with the GC, unscrew the worn jet from the detector base body.



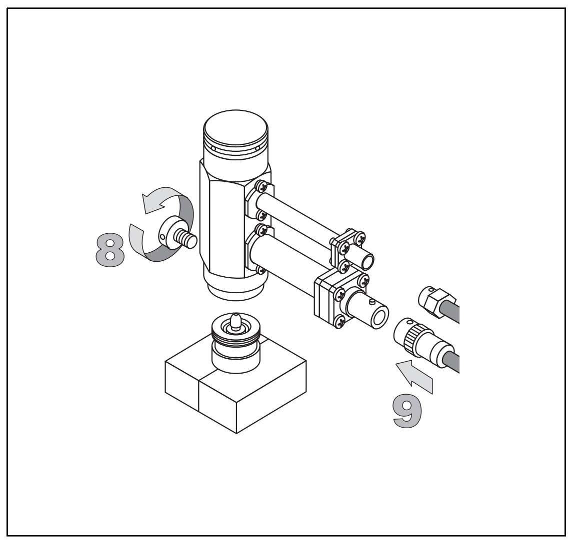
7. Place a new jet into the housing and tighten it with the proper tool. Ensure the jet is perfectly vertically aligned to avoid damaging its ceramic part.



8. Place the detector cell on the detector base body and tighten the fixing screw on the front of the detector cell.

9. Reconnect the signal and ignition cables to the detector.

10. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Clean the Collecting Electrode

Materials needed

- Screwdriver
- Pliers
- Forceps or tweezers
- Ultrasonic cleaning bath
- Liquid detergent
- Clean compressed air
- GC-grade methanol
- Clean paper towel

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

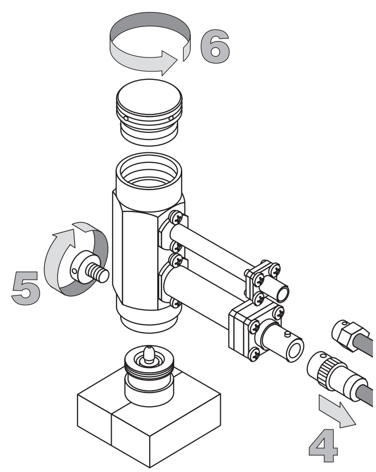
PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

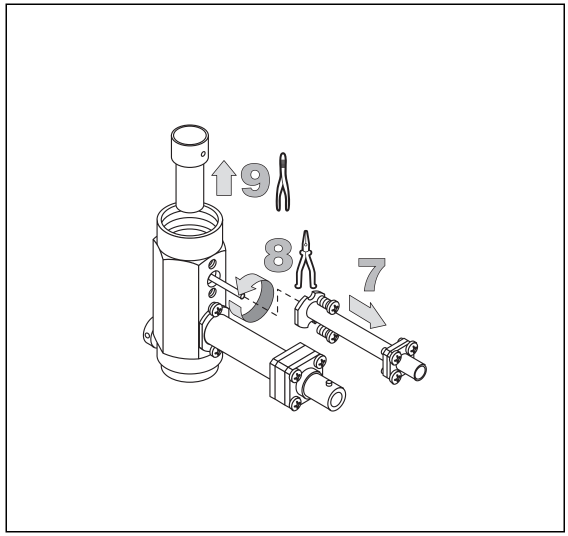
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FID detector, to access the FID control table. In the following example, a FID installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

RIGHT DETECTOR (FID)		
Flame		Off <
Base temp	300	300
Signal pA		(5.4)
Ign. tresh		2.0
Flameout retry		Off
H2	35	Off
Air	350	Off
Mkup N2	30	30

4. Disconnect the signal and ignition cables from the detector.
5. Loosen the fixing nut on the front of the detector cell and remove it.
6. Unscrew and remove the cap of the detector cell.



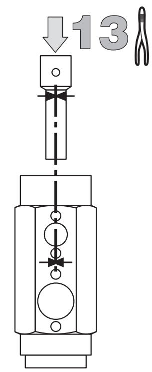
7. Remove the holder assembly of the signal cable by unscrewing the two screws that fix it on the detector cell.
8. Using forceps, unscrew and remove the gold contact pin.
9. Using tweezers, extract the collecting electrode through the top of the detector cell.



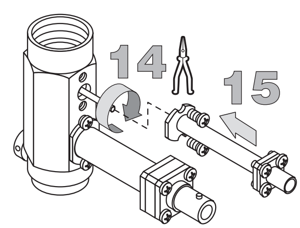
10. Place the collecting electrode in the ultrasonic bath filled with liquid detergent and clean it for about five minutes.
11. Handle the collecting electrode with forceps or tweezers, rinse it using distilled water, then methanol.
12. Place the collecting electrode on a paper towel and let it air dry.

PRECAUTIONS

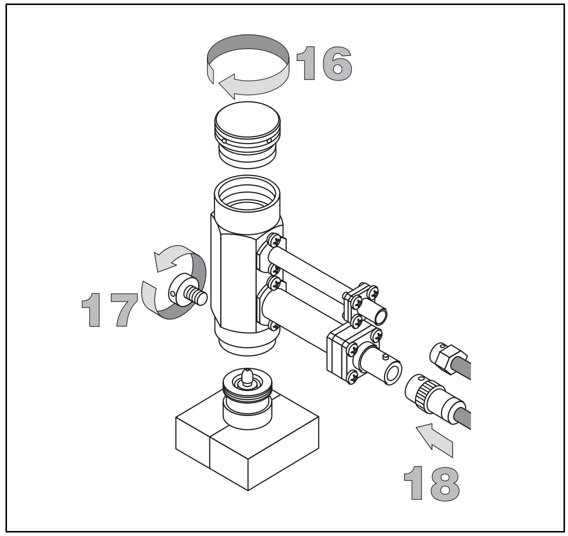
13. When the electrode is dry, place it in its housing. Ensure the insert hole is perfectly vertically aligned with the hole on the cell.



14. Using forceps, reconnect the gold contact pin.
15. Remount the connector holder assembly, verifying the contact efficiency.



16. Screw the detector cap into its housing.
17. Place the detector cell on the base body and tighten the fixing screw.
18. Reconnect the signal and ignition cables to the detector.
19. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Collecting Electrode

Materials needed

- Screwdriver
- Pliers
- Forceps or tweezers
- Collecting electrode (P/N 259 011 15)

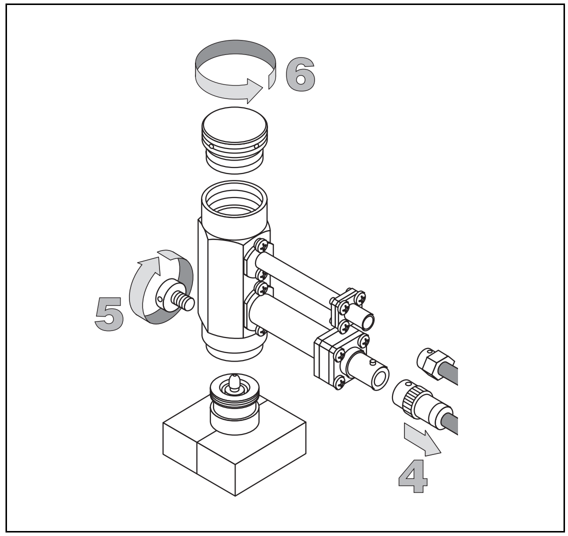
WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

PRECAUTIONS

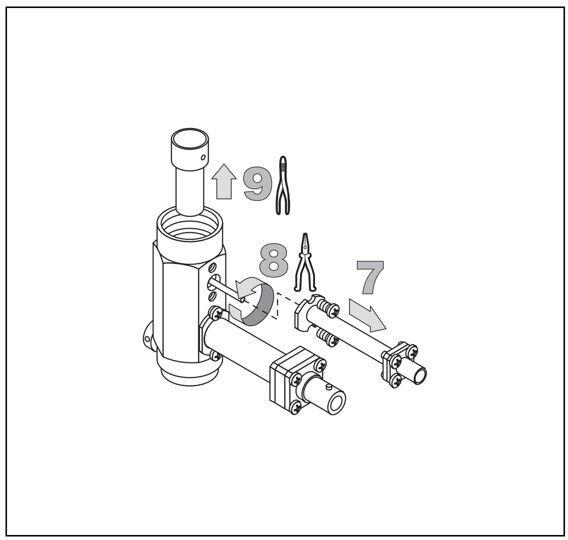
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FID detector, to access the FID control table. In the following example, a FID installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

```
      RIGHT DETECTOR (FID)
Flame                         Off <
Base temp             300     300
Signal pA                    (5.4)
Ign. tresh                    2.0
Flameout retry                Off
H2                     35     Off
Air                   350     Off
Mkup N2                30      30
```

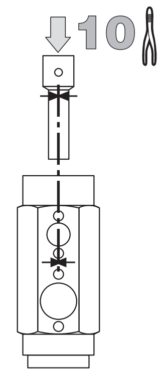
4. Disconnect the signal and ignition cables from the detector.
5. Loosen the fixing nut on the front of the detector cell and remove it.
6. Unscrew and remove the cap of the detector cell.



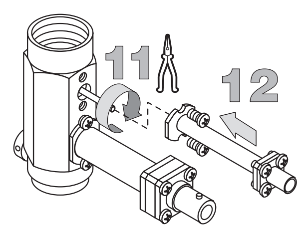
7. Remove the holder assembly of the signal cable by unscrewing the two fixing screws.
8. Using forceps, unscrew and remove the gold contact pin.
9. Using tweezers, extract the collecting electrode through the top of the detector cell.

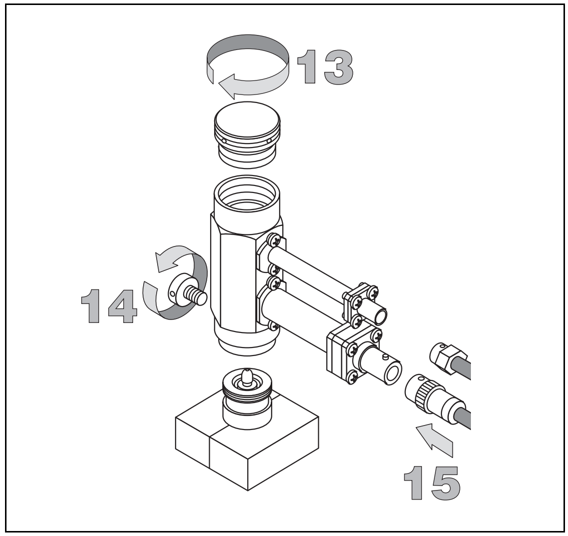


10. Place the new collecting electrode into its housing. Ensure the insert hole is perfectly vertically aligned with the hole on the cell.



11. Using forceps, reconnect the gold contact pin.
12. Remount the connector holder assembly, verifying the contact efficiency.





13. Screw the detector cap into its housing.
14. Place the detector cell on its base body and tighten the fixing screw.
15. Reconnect the signal and ignition cables to the detector.
16. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.

OPERATING SEQUENCE

Replace the Ignition Assembly

Material needed:

- Screwdriver
- Connector Holder Assembly (P/N 206 016 03)

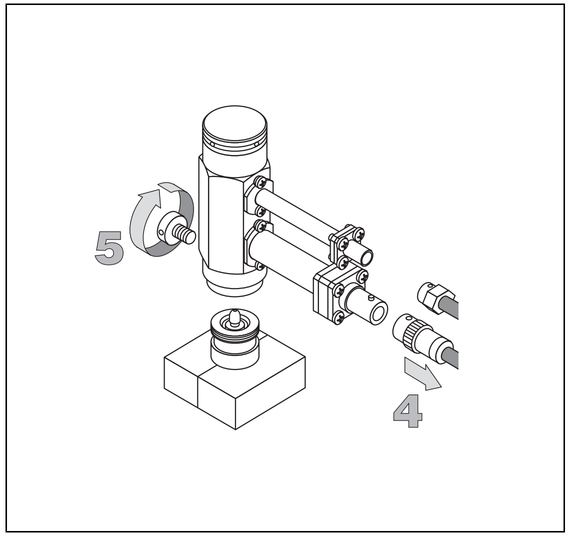
WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

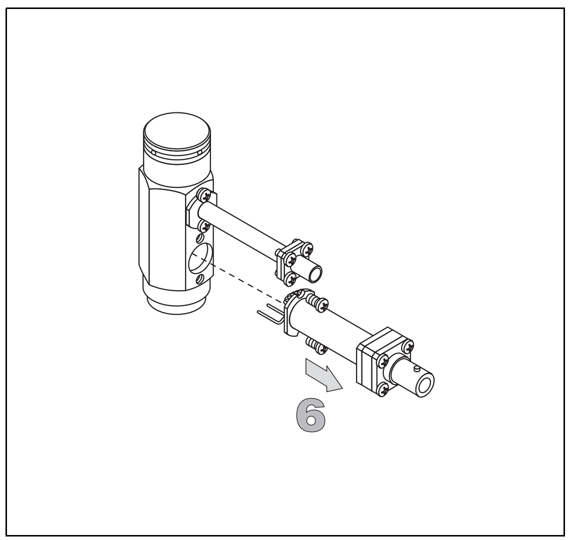
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FID detector, to access the FID control table. In the following example, a FID installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

RIGHT DETECTOR (FID)		
Flame		Off <
Base temp	300	300
Signal pA		(5.4)
Ign. tresh		2.0
Flameout retry		Off
H2	35	Off
Air	350	Off
Mkup N2	30	30

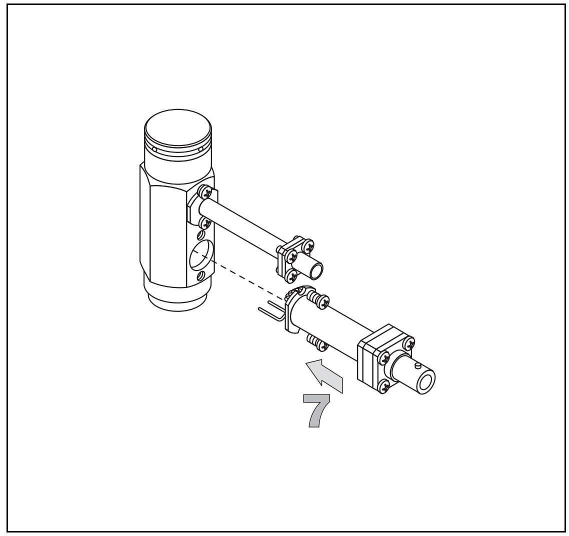
4. Disconnect the signal and ignition cables form the detector.
5. Loosen the fixing nut on the front of the detector cell and remove it.



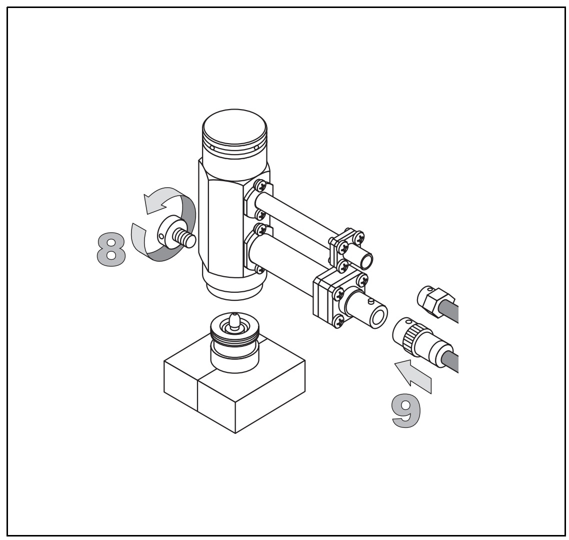
6. Unscrew the two fixing screws and remove the lower connector holder assembly from the detector cell.



7. Remount the new connector holder assembly and fix it to the detector cell using the two screws. Make sure the connecting fork is placed downwards to achieve the correct contact with the jet.



8. Place the detector cell on the detector base body and tighten the retaining screw.
9. Reconnect the signal and ignition cables to the detector.
10. Reconnect the signal and ignition cables to the detector.
11. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



FID Troubleshooting

Change in Sensitivity

The sensitivity of the Flame Ionization Detector depends on the carrier and detector gases flow rates and to the detector temperature.

The gas flow rates must be set correctly for proper FID operation.

Gases

The gases normally used with FID are reported in Table 11-2.

Table 11-2. Gases used for FID

Carrier gas		Detector Gas	Make-up gas Gas
Capillary Columns	Packed Columns		
Helium Nitrogen Hydrogen	Helium Nitrogen Argon	Hydrogen + Air	Nitrogen (recommended) Helium

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.

NOTE Make-up gas is not required when a packed column is used.

The recommended range of detector gas flow rates tolerated by the FID are reported in Table 11-3.

Table 11-3. Recommended flow rates for FID

Gas	Flow rate
Hydrogen	30 - 50 mL/min
Air	300 - 600 mL/min
Make-up gas	10 - 60 mL/min

To gain optimum analytical performance from the FID the hydrogen flow rate should be experimentally optimized, keeping the carrier and air flows constant, to obtain the maximum signal intensity for the components of interest.

The FID sensitivity will be reduced as hydrogen flow rate is above or below the optimal value. The flow rate of the air is less critical than the hydrogen one. An excessive amount of air will destabilize the flame, causing noise and eventual flameout.

NOTE Generally the air flow rate must be set to about 10 times the hydrogen flow rate to keep the flame lit.

A low flow rate of air would reduce the detector sensitivity.

The guidelines in Table 11-4 show typical FID operating conditions.

Table 11-4. Typical FID Operating Conditions

Gas	Capillary Columns	Packed Columns
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

For high sensitivity applications, it is essential that you exclude all traces of organic contamination from the chromatographic system and/or from the gas lines of the detector. Such contamination would cause ghost peaks in the chromatogram or, more commonly, an unstable baseline.

WARNING! **It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.**

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to *Using Hydrogen* on page xvii.

Flame Ignition

You can ignite the flame as soon as the detector temperature has reached 150°C.

After the flame appears to have ignited, check for water vapor condensed on a mirror or on the polished end of a wrench directly over the FID exit. You should observe steam condensing on the cold surface. If not, the flame is not ignited.

WARNING! **Do not lean over the FID to see the flame, it is invisible.**

Table 11-5 reports the possible causes of flame ignition problems and the relevant remedies.

Table 11-5. Difficulty in igniting the flame

Cause	Remedy
Incorrect fuel gas flows	Make sure that all flows are correct.
Defective igniter	Check the igniter element. Refer to the *Replace the Ignition Assembly* Operating Sequence on page 211 for instructions.
Broken or cracked flame jet	Replace jet. Refer to the *Replace the Jet* Operating Sequence on page 199 for instructions.
Blocked jet tip	Check for a blocked jet by measuring the hydrogen flow with the Thermo Scientific GFM Pro Flowmeter (or equivalent). If required, remove and clean the jet following the *Clean the Jet* Operating Sequence on page 195.
Faulty electronics	Contact your customer support organization. Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices and affiliates worldwide.

Contamination

All organic compounds that pass through the FID are burnt in the flame. Some compounds can produce detector contamination, resulting in a high noise baseline. If this symptom is found, clean the collecting electrode. Refer to the *Clean the Collecting Electrode* Operating Sequence on page 202 for instructions.

12

Maintaining an Electron Capture Detector

Chapter at a Glance...

Operating Sequences

Electron Capture Detector (ECD)

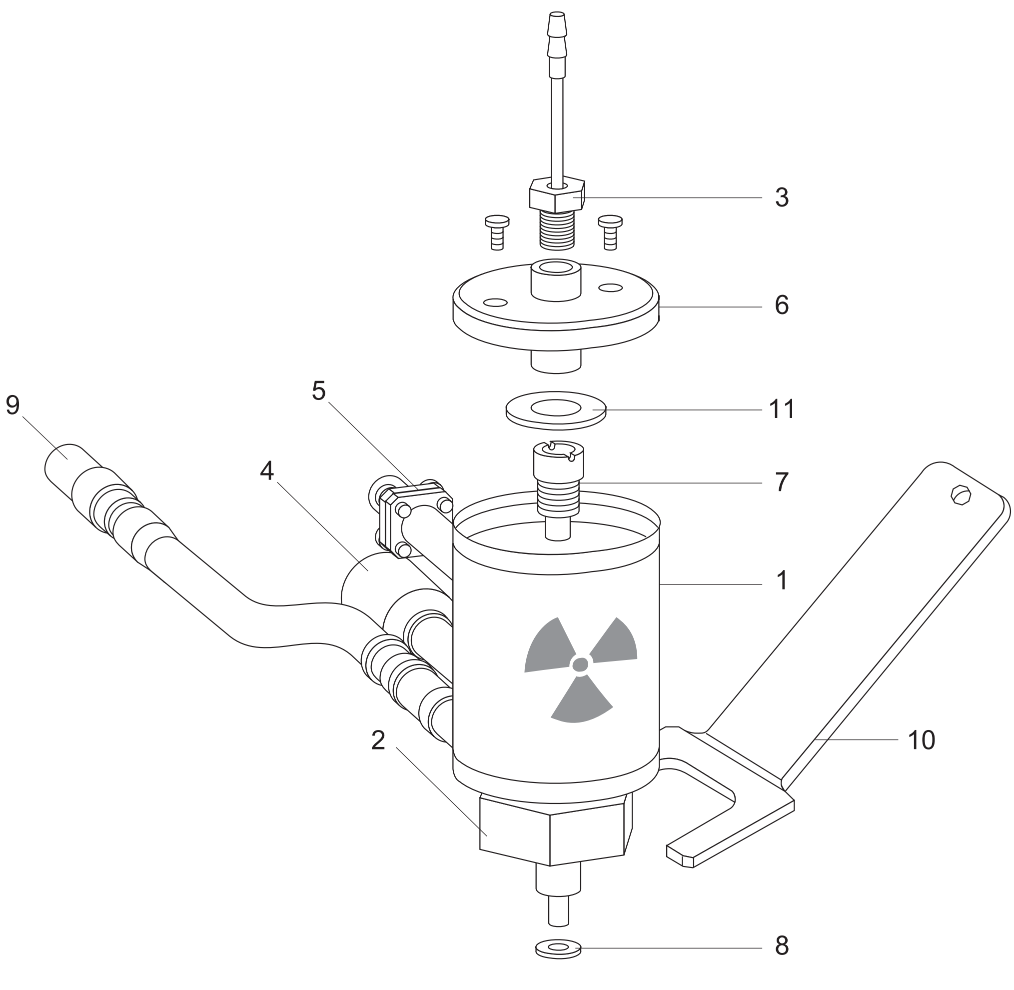


Table 12-1. Electron Capture Detector (ECD) parts identification table

No.	Description	P/N
1	Detector cell	
2	Fixing nut	
3	Chimney	
4	Excitation connector	
5	Signal connector	
6	Detector cover	
7	Collecting electrode	277 003 50
8	Silver seal	290 150 58
9	Temperature sensor/heater assembly	190 500 87
10	ECD fixing tool	205 021 50
11	Seal	290 326 08
	Maintenance kit	19050221

ECD Maintenance

The ECD, if properly used, has a good resistance against contamination thanks to its unique internal geometry. However, some critical operating conditions may give rise, in the long run, to contamination of the collecting electrode. This contamination is highlighted by an excessive increase of the base frequency and a baseline drift when the reference current or the pulse amplitude is changed. The collecting electrode, in these cases, can be easily removed and cleaned without disturbing the radioactive source.
If contamination of the whole cell is suspected, heat the ECD at the maximum operating temperature with carrier and make-up gases flowing through the detector (thermal cleaning). The decontamination process can be followed by monitoring the base frequency. Initially the frequency value tends to increase to very high values, and then it decreases to acceptable ones. If irreversible contamination of the cell is suspected, please contact your local Thermo Fisher Scientific Technical Service office for maintenance or replacement of the source, whichever is necessary. Strictly follow the existing regulations for the transport of radioactive material.

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 inserted in a cylindrical holder made of 6 mm thickness stainless steel. The holder is fixed to the detector body, also made of stainless steel, in order to be totally 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 prevents overheating.

In normal operating conditions, no dispersion of solid or gaseous radioactive material is involved. This eliminates any risk of direct or secondary radiation.

For no reason should the detector be opened or handled by the user.

Any maintenance or service operations involving even partial disassembling of the detector must be performed ONLY by Thermo Fisher Scientific Milan site or by a laboratory specifically licensed to handle radioactive material.

Wipe Test

Before leaving the factory, each ECD is leak tested for surface radio contamination by means of the **Wipe Test** method. Each detector is provided with a **Wipe Test Certificate** reporting the results of the values found and the procedure followed.

NOTE The users of the ECD detector in the United States are required to perform a Wipe Test on their ECD at intervals not exceeding 3 years (36 months), following the reported procedure.

For other Countries, please refer to the appropriate agency for equivalent requirements.

Wipe Test Certificate

ASSAY RESULTS*

Radionuclide	Counting Efficiency	Background c.p.m	c.p.m. net (**)	d.p.m. (**)	Activity Bq (**)
^{63} Ni					

(*) Method described overleaf (**) Sum of measurement taken

ASSAY OF RESULTS

The radioactive integrity of the detector is within the stated limits:

The detectable activity is less than 185 Bq.

Wipe Test performed by: Date:

Analysis performed by: Date:

ECD Wipe Test is to be carried out under a controlled procedure as reported below:

Procedure

Wet cotton wool swab with methanol.

Wipe the outer surface of the ECD.

Remove metal exit tube from top of ECD.

Wipe the bottom of the exit tube.

Remove the two screws retaining the tower cap.

Wipe the bottom of the cylindrical surface and place the swab in a marked vial.

Repeat twice steps 1-6 with new cotton wool swabs and place in clean marked vial.

Submit the marked vials containing the swabs for the radioactivity measurement using a liquid scintillation counter.

Notes:

The wipe test should be performed on the ECD at room temperature.

Radioactivity should be measured by a liquid scintillation spectrometer (TRI-CARB Packard or similar) discriminating at 1-70 keV to encompass the maximum beta energy
of 67 keV of ^{63}Ni. The test and background samples should be measured for 10 minutes.

Suitable precautions should be taken by the person performing the test.

The test should be carried out by authorized personnel

Limit

For a source containing 370 MBq of ^{63}Ni, the acceptable limit is 185 Bq.

OPERATING SEQUENCE

Clean the Collecting Electrode

Materials needed

- Screwdriver
- 2.5 mm Allen wrench
- Tweezers
- Ultrasonic cleaning bath
- GC-grade hexane
- GC-grade toluene
- Fine emery paper

WARNING! **Any maintenance or repair operations involving the radioactive source must be performed ONLY by qualified personnel duly authorized to handle radioactive material. Please contact your local Thermo Fisher Scientific Technical Service office.**

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

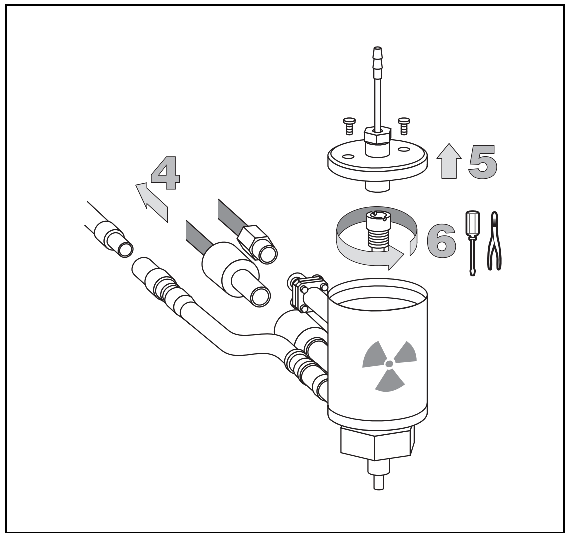
WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table. In the following example, an ECD installed on the right channel is considered.
2. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
3. Scroll to `Mkup(N2)` and press **OFF**.

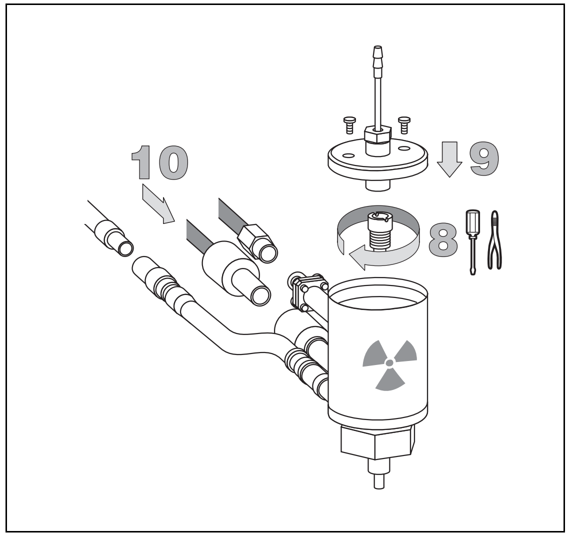
```
        RIGHT DETECTOR (ECD)
Base temp               250     Off <
ECD temp                250     Off
Ref current nA                  1.0
Freq kHz                     (2.15)
Pulse amp V                      25
Pulse width us                  1.0
Mkup (N2)                40     Off
```

4. Disconnect the signal, excitation and heater cables from the detector.
5. Remove the cover and the detector chimney unscrewing the two retaining Allen screws. Use a 1.5 mm Allen wrench.
6. Unscrew the collecting electrode and remove it using tweezers.



7. Clean the collecting electrode, washing it with toluene and hexane, preferably in an ultrasonic bath. In the most critical cases fine emery paper can be used.

8. Remount the electrode without touching it. Use tweezers.
9. Remount the detector cover with the chimney.
10. Reconnect the signal and ignition cables to the detector.
11. Reset the detector to the required operating conditions.



OPERATING SEQUENCE

Replace the Collecting Electrode

Materials needed

- Screwdriver
- 2.5 mm Allen wrench
- Tweezers
- Collecting Electrode (P/N 277 003 50)

WARNING! **Any maintenance or repair operations involving the radioactive source must be performed ONLY by qualified personnel duly authorized to handle radioactive material. Please contact your local Thermo Fisher Scientific Technical Service office.**

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

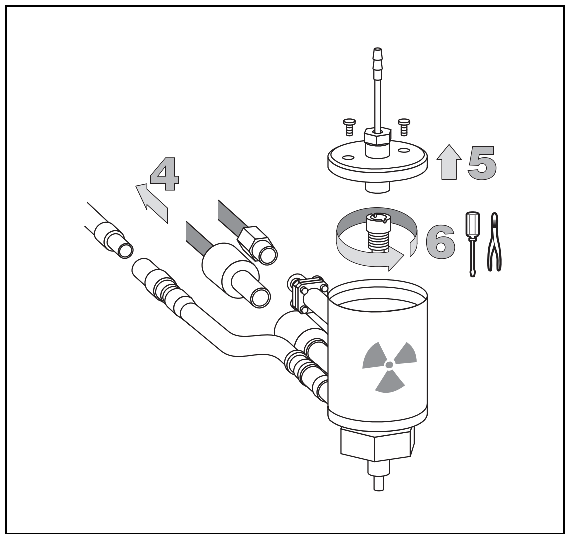
WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table. In the following example, an ECD installed on the right channel is considered.
2. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
3. Scroll to `Mkup(N2)` and press **OFF**.

```
      RIGHT DETECTOR (ECD)
Base temp             250    Off <
ECD temp              250    Off
Ref current nA               1.0
Freq kHz                   (2.15)
Pulse amp V                   25
Pulse width us               1.0
Mkup (N2)              40    Off
```

4. Disconnect the signal, excitation and heater cables from the detector.
5. Remove the cover and the detector chimney unscrewing the two retaining Allen screws. Use a 1.5 mm Allen wrench.
6. Unscrew the collecting electrode and remove it using tweezers.

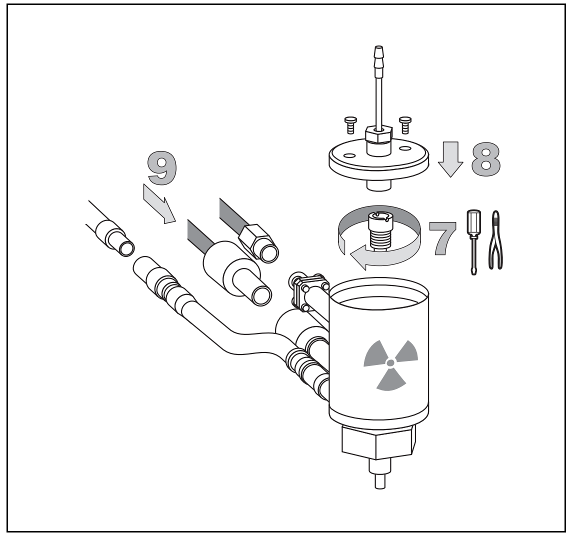


7. Using tweezers, replace the collecting electrode with a new one and tighten it.

8. Remount the cover with the chimney on the detector.

9. Reconnect the signal and ignition cables to the detector.

10. Reset the detector to the required operating conditions.



OPERATING SEQUENCE

Replace the Silver Seal

Materials needed

- Silver seal (P/N 290 150 58)
- ECD fixing tool (P/N 205 021 50)
- Tweezers

WARNING! **Any maintenance or repair operations involving the radioactive source must be performed ONLY by qualified personnel duly authorized to handle radioactive material. Please contact your local Thermo Fisher Scientific Technical Service office.**

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

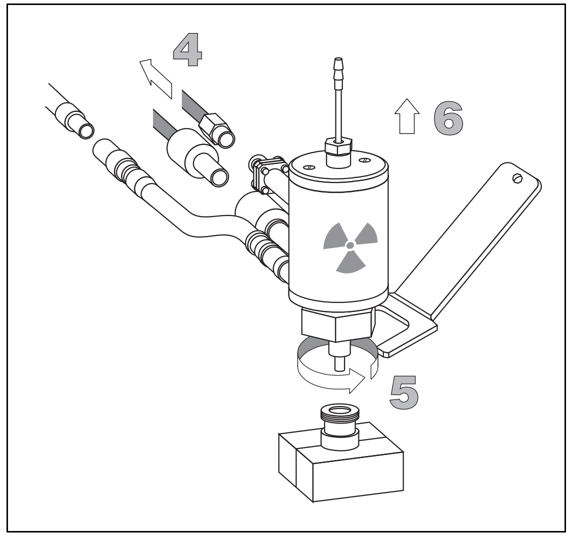
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table. In the following example, an ECD installed on the right channel is considered.
2. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
3. Scroll to `Mkup(N2)` and press **OFF**.

```
        RIGHT DETECTOR (ECD)
Base temp               250     Off <
ECD temp                250     Off
Ref current nA                  1.0
Freq kHz                     (2.15)
Pulse amp V                      25
Pulse width us                  1.0
Mkup (N2)                40     Off
```

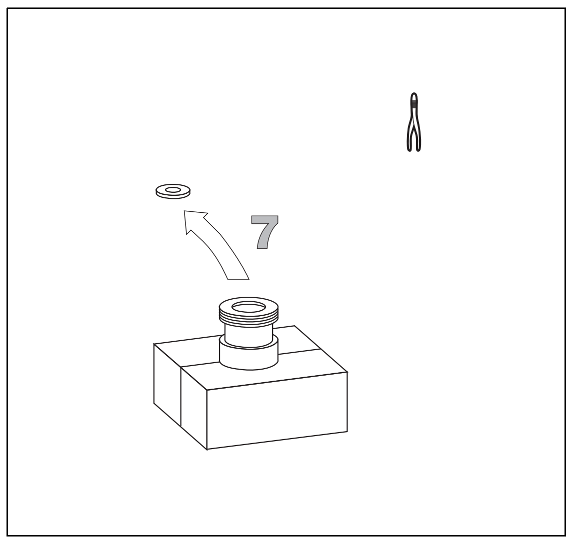
4. Disconnect the signal, excitation and heater cables from the detector.

5. Loosen the nut that fixes the detector to its base body with the proper tool contained in the standard outfit.

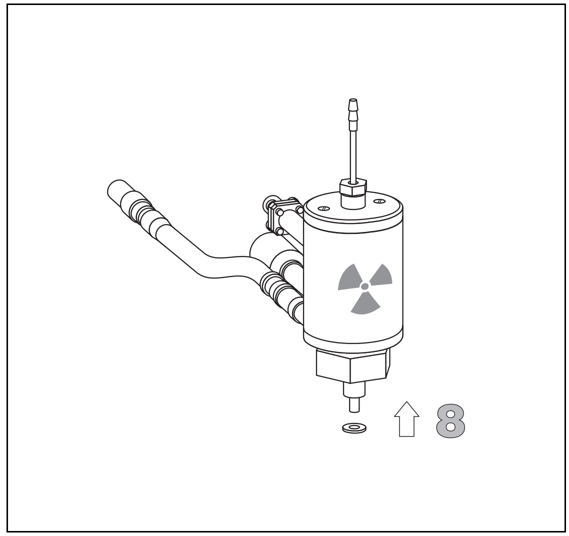
6. Remove the detector from its base body.



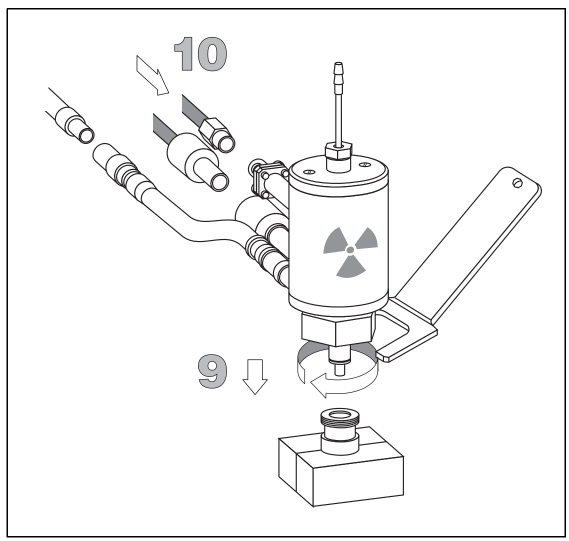
7. Using tweezers, remove the worn silver seal from its housing in the base body.



8. Insert a new silver seal on the lower part of the ECD detector.



9. Remount the detector on its base body, tightening the fixing nut with the proper tool.
10. Reconnect the signal and ignition cables to the detector.



11. Reset the detector to the required operating conditions.

ECD Troubleshooting

Change in Sensitivity

Temperature

The effect of a temperature change on the ECD response generally depends on the compounds analyzed.

Gases

The ECD behavior can be affected by a change of the total flow of the gases through the cell. The resulting response is also related to the nature and purity of the gases.

The gases normally used on the ECD are reported in Table 12-2

Table 12-2. Gases used for ECD

Carrier gas		Make-up gas
Capillary Columns	Packed Columns	
Helium Nitrogen Hydrogen	Nitrogen Argon-Methane (5-10%)	Nitrogen (recommended) Argon-Methane (5-10%)

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.

The carrier gas flow rate must be set according to the type of the gas used and to the diameter and the stationary phase of the capillary (or packed) column installed.

NOTE When packed columns are used, the make-up gas (as carrier gas type) is used only if strictly required.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to *Using Hydrogen* on page xvii.

Contamination

Detector contamination could be indicated by the following effects in the chromatogram:

- reduced signal to noise ratio
- high-noise baseline
- baseline drift with changing pulse voltage
- negative dips after peaks

Troubleshooting the ECD

Table 12-3. ECD Troubleshooting Table

Symptom	Diagnosis	Remedy
High base frequency	Impure gas supply.	Use high purity grade gases and filters to trap moisture and oxygen.
	No (or insufficient) flow of make-up gas.	Increase make-up gas flow rate.
	Excess column bleeding.	Column conditioning is needed.
	Leaks on carrier and/or make-up gas lines.	Run a leak test. Refer to the *Performing a Leak Check (Capillary Column)* Operating Sequence on page 181. for instructions.
	Contaminated collecting electrode	Clean the collecting electrode, as described in the *Clean the Collecting Electrode* Operating Sequence on page 226, or replace it.
	Chemically contaminated radioactive source.	Refer to the notes reported in the *ECD Maintenance* paragraph on page 222. Contact your customer support organization. Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices and affiliates worldwide.
	Pulse width not correctly set.	Set `Pulse width` to 1.0 µs for Nitrogen and 0.1 µs for Ar/CH_4.

Table 12-3. ECD Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Negative dips after peaks	Contaminated collecting electrode	Refer to the notes reported in the *ECD Maintenance* paragraph on page 222.
	Contaminated radioactive source.	Contact your customer support organization. Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices and affiliates worldwide.
Baseline drift with changing pulse voltage.	Contaminated collecting electrode.	Refer to the notes reported in the *ECD Maintenance* paragraph on page 222. Contact your customer support organization. Appendix B, *Customer Communication*, contains contact information for Thermo Fisher Scientific offices and affiliates worldwide.

13

Maintaining a Nitrogen Phosphorus Detector

Chapter at a Glance...

Operating Sequences

Nitrogen Phosphorus Detector (NPD)

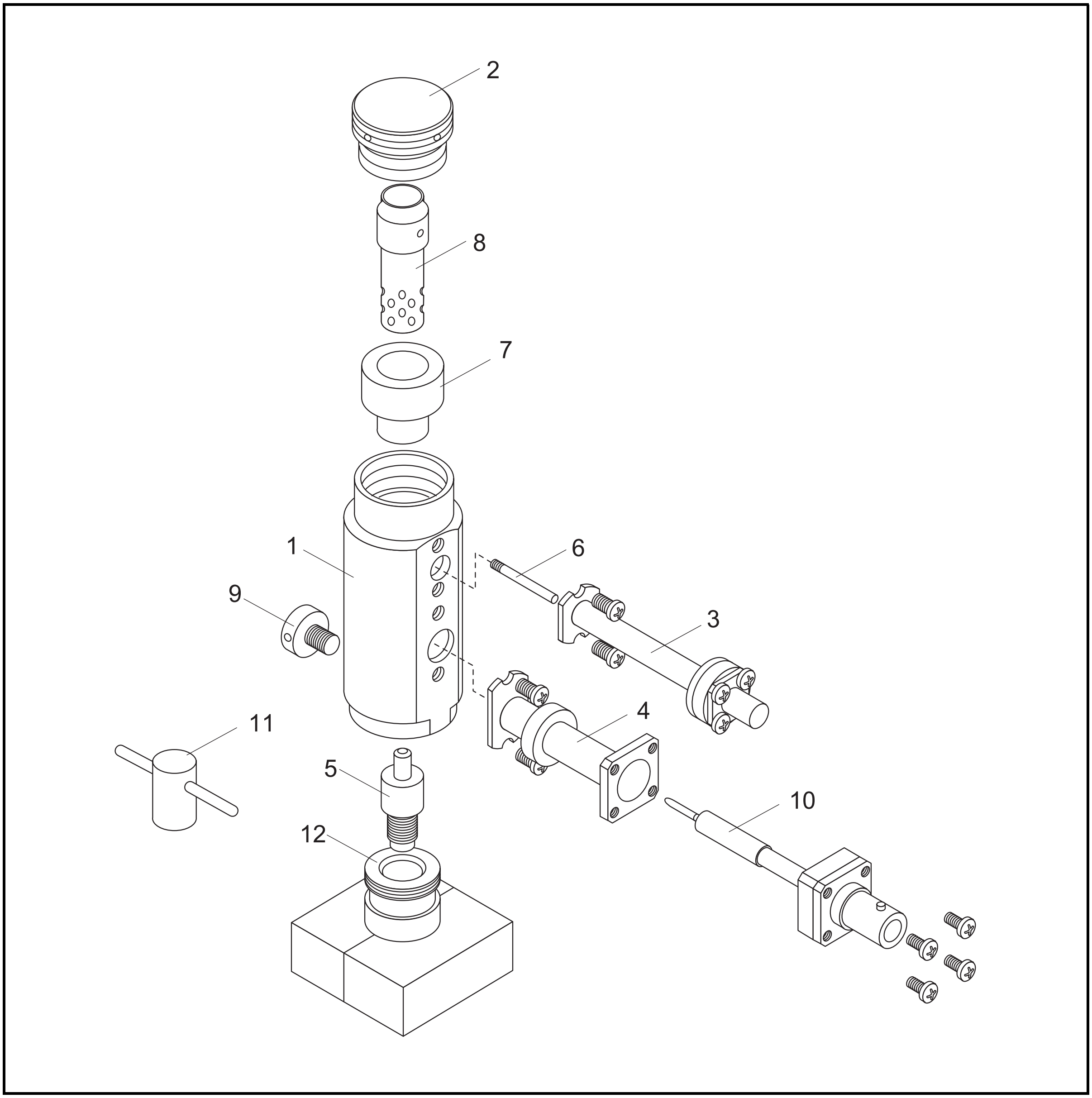


Table 13-1. Nitrogen Phosphorus Detector (NPD) parts identification table

No.	Description	P/N
1	Detector cell	
2	Cap	
3	Signal cable holder	
4	Thermionic source holder	
5	Jet	404 043 02
6	Contact pin	206 016 02
7	Ceramic insulator	302 023 10
8	Collecting electrode	259 011 10
9	Retaining screw	311 060 08
10	TS-2 thermionic source (standard)	465 002 55
	TS-1 thermionic source for ENS mode	465 002 50
11	Jet removing tool	205 019 00
12	Detector base body	

NPD Maintenance

To ensure optimum performance of the NPD, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector cleaning or thermionic source replacement could be necessary.

To properly maintain the Nitrogen-Phosphorus Detector, you should perform the following sequences:

- cleaning the jet.
- replacing the jet.
- cleaning the collecting electrode.
- replacing the collecting electrode.
- replacing the thermionic source.

Thermionic source

The thermionic source installed in the detector may be removed and replaced with a new one or with a source having different coating.

NOTE New thermionic sources may require slightly different values of source heating current to produce the same signal observed with a previous thermionic source.

OPERATING SEQUENCE

Clean the Jet

Materials needed

- Screwdriver
- Tool for extracting the jet (P/N 205 019 00)
- Forceps or tweezers
- Ultrasonic cleaning bath
- Liquid detergent
- Clean compressed air
- GC-grade methanol
- Clean paper towel

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

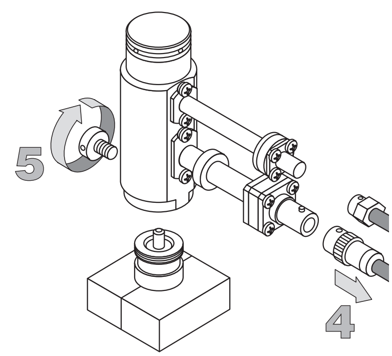
PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix B, *Reagents Safety Information* for more information.**

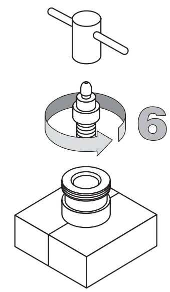
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the NPD detector, to access the NPD control table. In the following example, a NPD installed on the right channel is considered.
2. Scroll to `Base temp` and press **OFF**.
3. Scroll subsequently to `Source curr`, `H2`, `Air` and `Mkup` and press **OFF**.

RIGHT DETECTOR (NPD)		
Source cur,A		Off <
Base temp	300	Off
Signal pA		(10.2)
Polarizer V		3.5
H2 delay time		Off
H2	2.3	Off
Air	70	Off
Mkup N2	15	Off

4. Disconnect the signal and ignition polarization cables from the detector.
5. Loosen the fixing screw on the front of the detector cell and remove it.



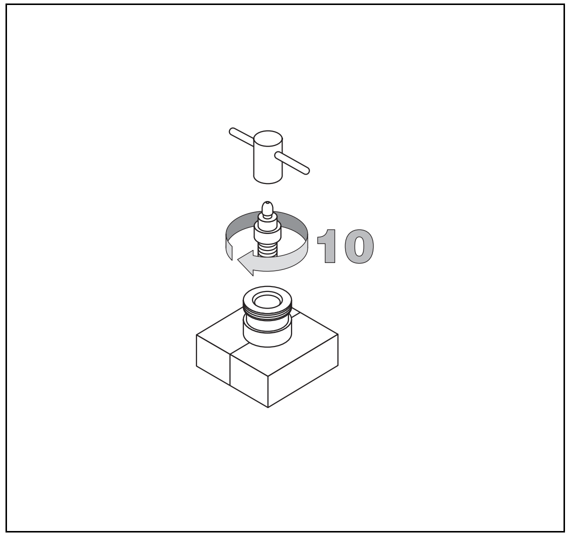
6. Using the tool provided with the GC, unscrew the metal jet.



7. Place the jet in the ultrasonic bath filled with liquid detergent and clean it for about five minutes.
8. Handling the jet with forceps or tweezers, rinse the jet with distilled water, then with methanol.
9. Place the jet on a paper towel and let it air dry.

PRECAUTIONS

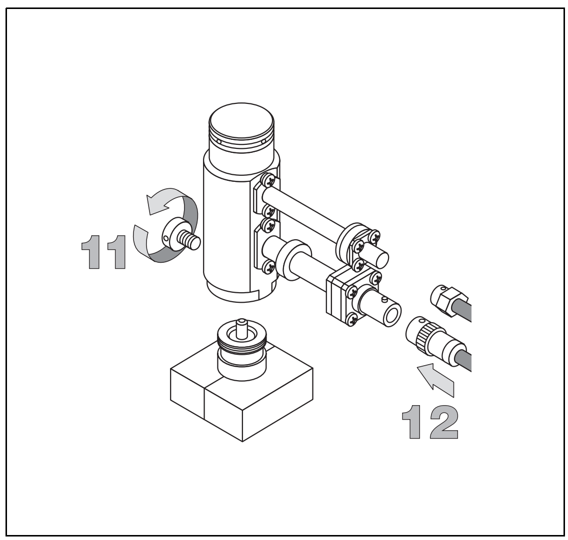
10. Place the jet into its housing and tighten it with the proper tool.



11. Place the detector cell on the detector base body and reconnect the fixing screw on the front of the detector cell.

12. Reconnect the signal and ignition cables to the detector.

13. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Jet

Materials needed

- Screwdriver
- Tool for extracting the jet (P/N 205 019 00)
- Forceps or tweezers
- Metal jet for NPD (P/N 404 043 02)

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

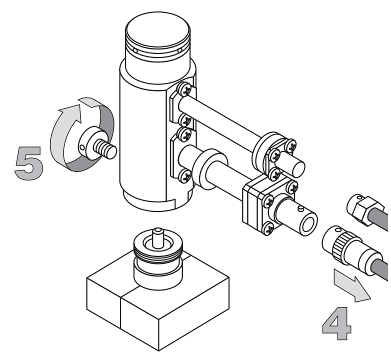
PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

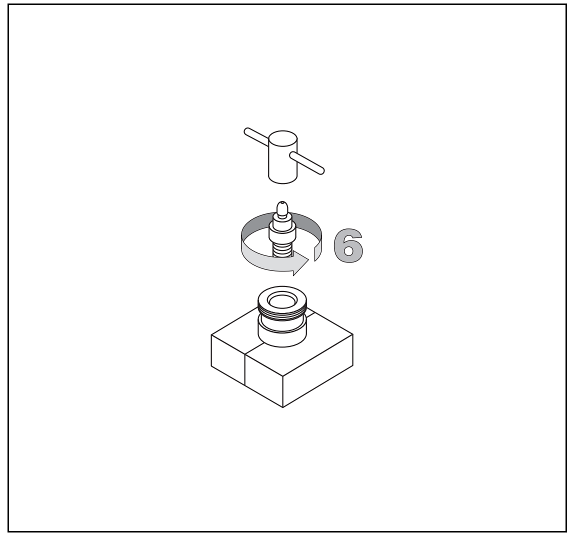
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the NPD detector, to access the NPD control table. In the following example, a NPD installed on the right channel is considered.
2. Scroll to `Base temp` and press **OFF**.
3. Scroll subsequently to `Source curr`, `H2`, `Air` and `Mkup` and press **OFF**.

RIGHT DETECTOR (NPD)		
Source cur,A		Off <
Base temp	300	Off
Signal pA		(10.2)
Polarizer V		3.5
H2 delay time		Off
H2	2.3	Off
Air	70	Off
Mkup N2	15	Off

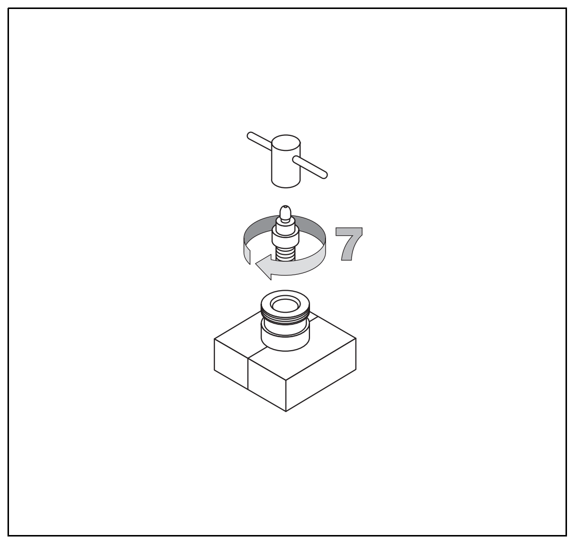
4. Disconnect the signal and ignition polarization cables from the detector
5. Loosen the fixing screw on the front of the detector cell and remove it.



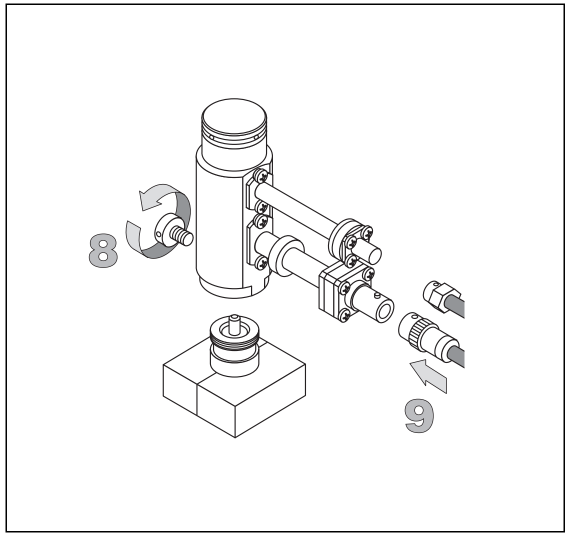
6. Using the tool provided with the GC, unscrew the worn jet from the detector base body.



7. Place a new jet into its housing and tighten it with the proper tool.



8. Reinsert the detector cell on the detector base body and tighten the fixing screw.
9. Reconnect the signal and ignition cables to the detector.
10. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Clean the Collecting Electrode

Materials needed

- Screwdriver
- Pliers
- Forceps or tweezers
- Ultrasonic cleaning bath
- Liquid detergent
- Clean compressed air
- GC-grade methanol
- Clean paper towel

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

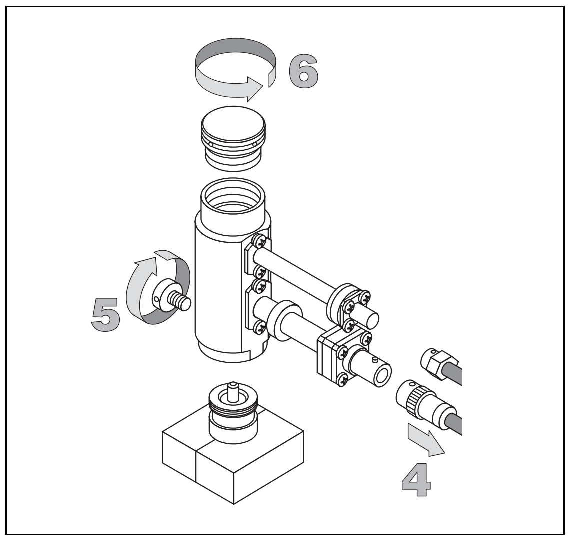
PRECAUTIONS

WARNING! When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.

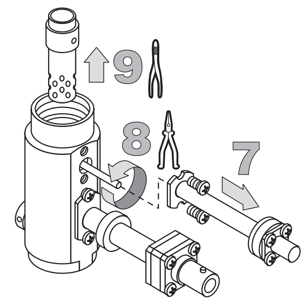
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the NPD detector, to access the NPD control table. In the following example, a NPD installed on the right channel is considered.
2. Scroll to `Base temp` and press **OFF**.
3. Scroll subsequently to `Source curr`, `H2`, `Air` and `Mkup` and press **OFF**.

RIGHT DETECTOR (NPD)		
Source cur,A		Off <
Base temp	300	Off
Signal pA		(10.2)
Polarizer V		3.5
H2 delay time		Off
H2	2.3	Off
Air	70	Off
Mkup N2	15	Off

4. Disconnect the signal and ignition polarization cables from the detector.
5. Loosen the fixing screw on the front of the detector cell and remove it.
6. Unscrew and remove the cap of the detector cell.



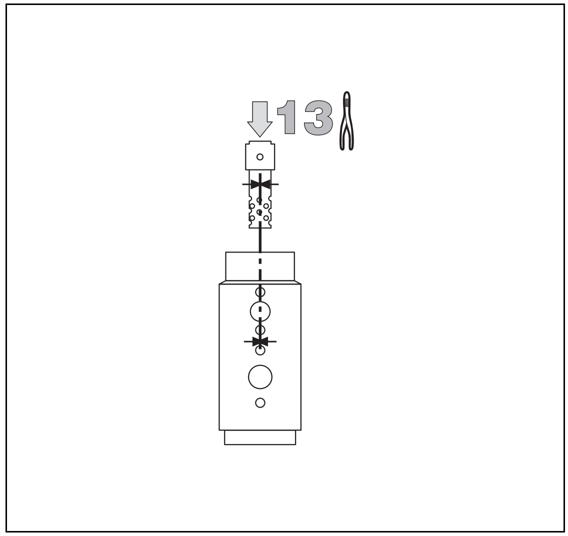
7. Remove the holder assembly of the signal cable by unscrewing the two screws that fix it on the detector cell.
8. Using forceps, unscrew and remove the contact pin.
9. Using tweezers, remove the collecting electrode through the top side of the detector cell.



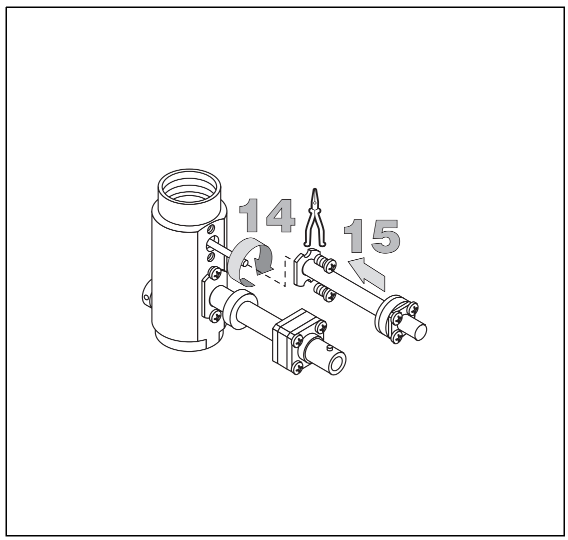
10. Place the collecting electrode in the ultrasonic bath filled with liquid detergent and clean for about five minutes.
11. Handling the collecting electrode with forceps or tweezers, rinse it using tap water, then methanol.
12. Place the collecting electrode on a paper towel and let it air dry.

PRECAUTIONS

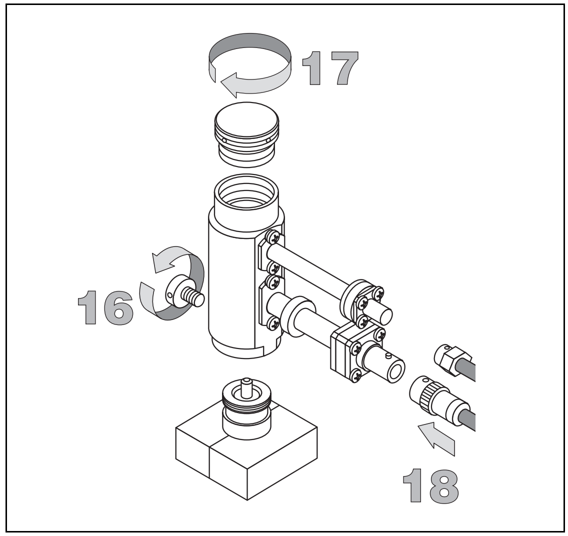
13. Place the collecting electrode into its housing. Ensure the insert hole is perfectly vertically aligned to the hole present on the cell.



14. Using forceps, reconnect the gold contact pin.
15. Remount the connector holder assembly verifying the contact efficiency.



16. Place the detector cell on the detector base body and tighten the fixing screw.
17. Screw the detector cap into its housing.
18. Reconnect the signal and ignition cables to the detector.
19. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Collecting Electrode

Materials needed

- Screwdriver
- Pliers
- Forceps or tweezers

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **Before replacing the collecting electrode, be sure that:**

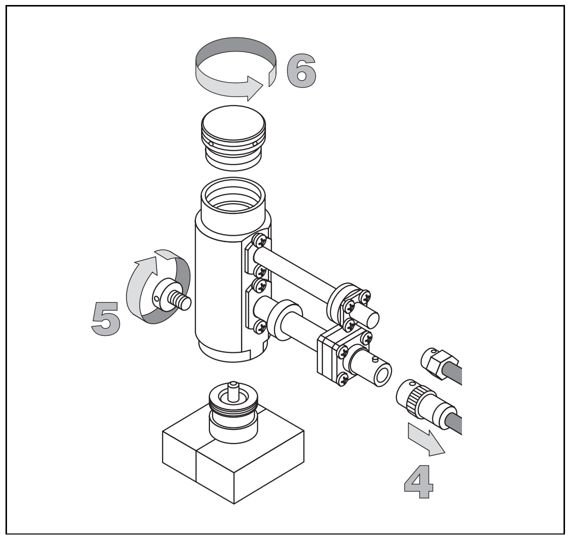
- **The flow of the detector gases has been interrupted**
- **The detector has been switched off**

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

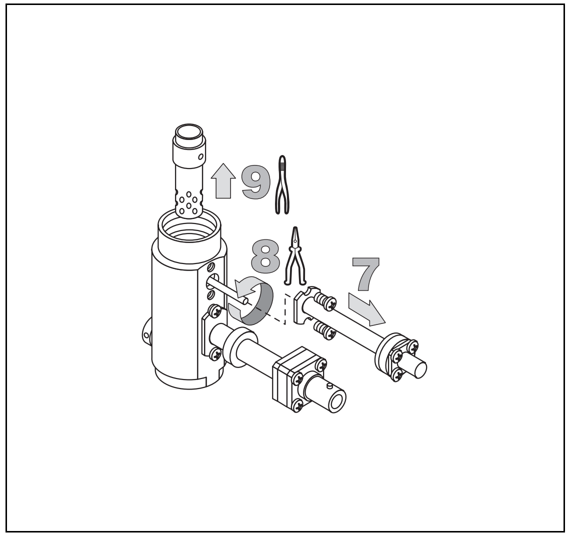
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the NPD detector, to access the NPD control table. In the following example, a NPD installed on the right channel is considered.
2. Scroll to `Base temp` and press **OFF**.
3. Scroll subsequently to `Source curr`, `H2`, `Air` and `Mkup` and press **OFF**.

RIGHT DETECTOR (NPD)		
Source cur,A		Off <
Base temp	300	Off
Signal pA		(10.2)
Polarizer V		3.5
H2 delay time		Off
H2	2.3	Off
Air	70	Off
Mkup N2	15	Off

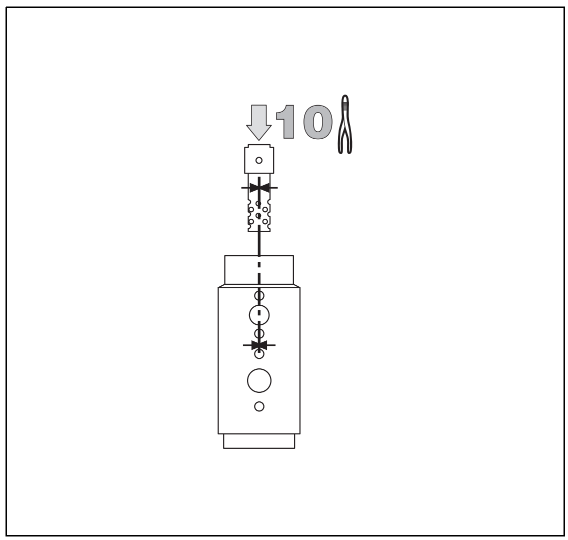
4. Disconnect the signal and source cables from the detector.
5. Loosen the fixing screw on the front of the detector cell and remove it.
6. Unscrew and remove the cap of the detector cell.



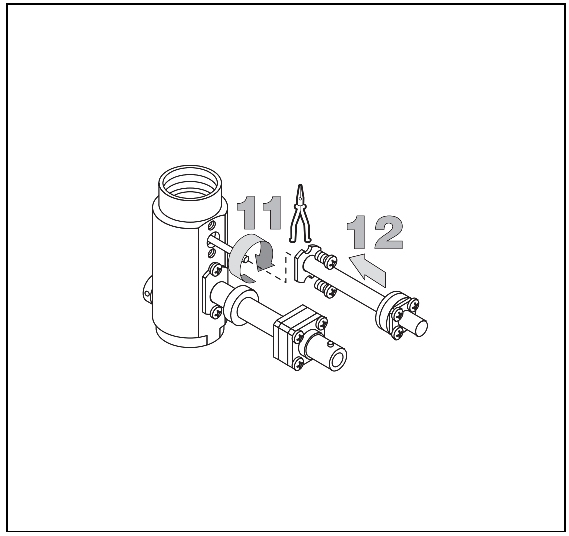
7. Remove the holder assembly of the signal cable by unscrewing the two screws that fix it on the detector cell.
8. Using forceps, unscrew and remove the contact pin.
9. Using forceps or tweezers, remove the collecting electrode through the top side of the detector cell.



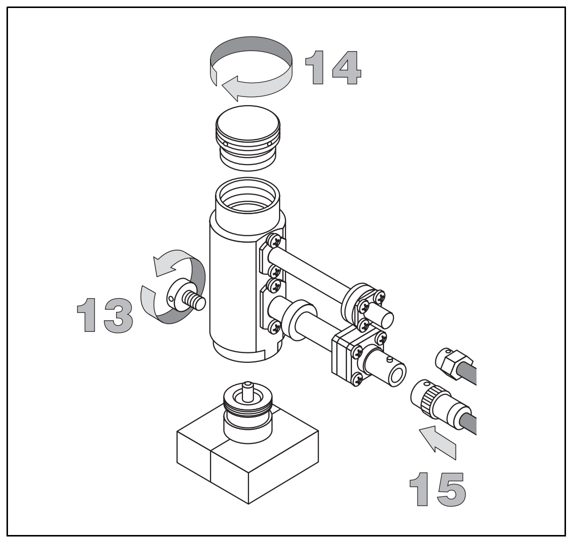
10. Place the new collecting electrode into its housing. Ensure the insert hole is perfectly vertically aligned to the hole present on the cell.



11. Using forceps, reconnect the gold contact pin.
12. Remount the connector holder assembly verifying the contact efficiency.



13. Place the detector cell on the detector base body and tighten the fixing screw.
14. Screw the detector cap into its housing.
15. Reconnect the signal and ignition cables to the detector.
16. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the NPD Thermionic Source

Materials needed:

- Screwdriver
- 1 mm Allen wrench
- Thermionic Source TS-1 (P/N 465 002 50) or TS-2 (P/N 465 002 55)

CAUTION **Thermionic sources are fragile. Handle them with care. Gently remove the plastic tube protecting the source ceramic during shipment.**

Especially avoid any excessive transverse force to the source ceramic.

Before inserting the new source into the holder check that the PTFE insulator is properly interposed.

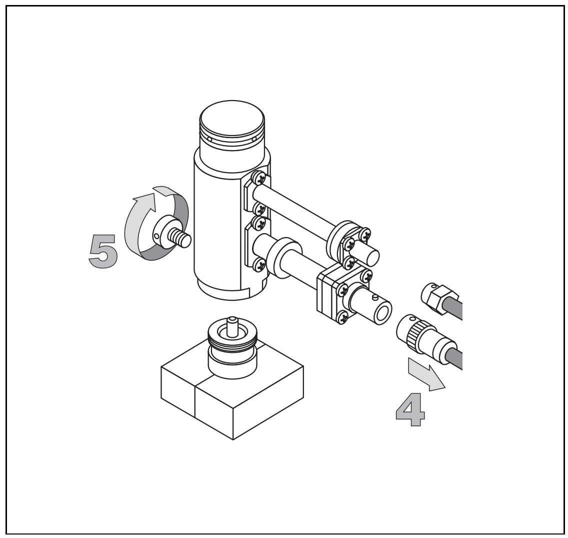
WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

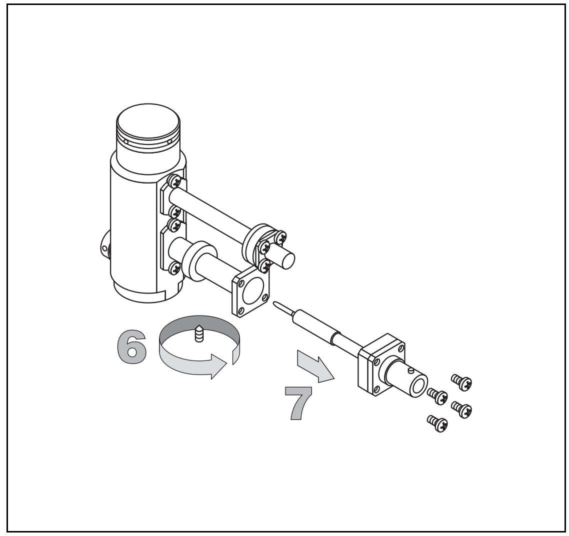
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the NPD detector, to access the NPD control table. In the following example, a NPD installed on the right channel is considered.
2. Scroll to `Base temp` and press **OFF**.
3. Scroll subsequently to `Source curr`, `H2`, `Air` and `Mkup` and press **OFF**.

RIGHT DETECTOR (NPD)		
Source cur,A		Off <
Base temp	300	Off
Signal pA		(10.2)
Polarizer V		3.5
H2 delay time		Off
H2	2.3	Off
Air	70	Off
Mkup N2	15	Off

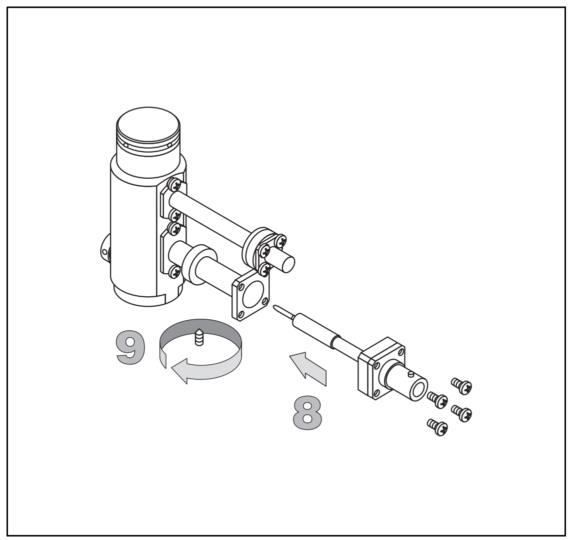
4. Disconnect the signal and source cables from the detector.
5. Loosen the fixing screw on the front of the detector cell and remove it.



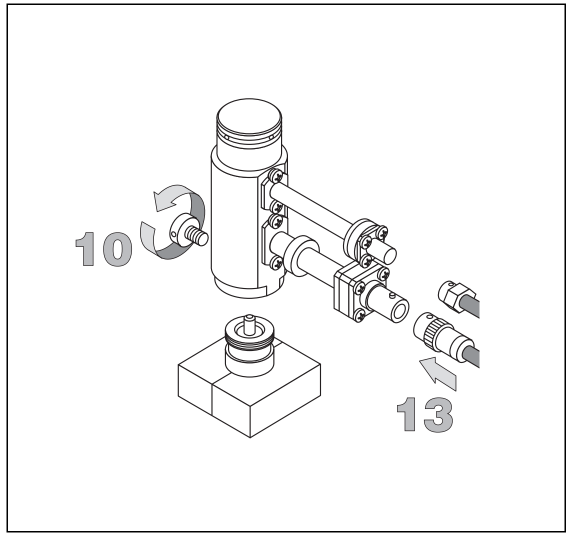
6. Unscrew the grub screw on the collar of the source holder.
7. Remove the four retaining screws of the source metallic socket and pull out the source.



8. Gently replace the thermionic source, pushing it in straight.
9. Fix the thermionic source into its housing, tightening the grub screw.



10. Place the detector cell on the detector base body and tighten the fixing screw.
11. Reconnect the signal and ignition cables to the detector.
12. Reset the detector to the required operating conditions. Refer to the relevant chapter of the TRACE™ GC Ultra *Operating manual* for more information.



NPD Troubleshooting

Sensitivity

The main cause of changes in sensitivity is related to the Thermionic source. Its gradual depletion results in a drop of sensitivity, that can be compensated by increasing the source current. Take into account that increasing current results in a shorter source life.
Loss of sensitivity are also related to source contamination, due to high boiling sample compounds not completely eliminated. Shift of detector temperature can reduce sensitivity.

Gases

The gases normally used for NPD are reported in Table 13-2. Consistent flows are necessary to maintain a constant and stable sensitivity.

Table 13-2. Gases used for NPD

Carrier gas		Detector Gas	Make-up gas Gas
Capillary Columns	Packed Columns		
Helium Nitrogen Hydrogen	Helium Nitrogen	Hydrogen + Air	Nitrogen Helium

Nitrogen is preferred over helium as make-up gas due to its lower thermal conductivity. Using nitrogen, the source requires a lower heating current.

NOTE When a packed column is used, make-up gas generally is not necessary.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to *Using Hydrogen* on page xvii.

Typical flow rates for detector gases are reported in Table 13-3.

Table 13-3. Typical NPD operating conditions

Gas	Flow rate
Hydrogen	2-4 mL/min
Air	40-80 mL/min
Make-up	10-20 mL/min

Troubleshooting the NPD

Table 13-4. NPD Troubleshooting Table

Symptom	Diagnosis	Remedy
No detector response	Source heating current too low.	Increase the heating current.
	No hydrogen flow.	Turn the hydrogen flow on and set it to a correct value.
	No air flow.	Turn the air flow on and set it to a correct value.
	Source turned off	Turn the source on.
	Source faulty	Replace the source
Detector response lower than expected	Lower source temperature	Check the base body temperature and source heating current.
	Air contamination in hydrogen line.	Turn the source off. Increase the hydrogen pressure for 10-20 minutes to purge the hydrogen line. Check the line tightness.
High background level	Heating current too high.	Set the correct operating parameter.
	Hydrogen flow too high.	Set the correct operating parameter.
	Air and/or Make-up gas flow too low.	Set the correct operating parameter.
	Excessive column bleed.	Column conditioning has to be performed.
FID-like response for solvent and other carbon based compounds	Hydrogen flow too high.	Reduce hydrogen flow to a lower value.

Table 13-4. NPD Troubleshooting Table (Continued)

Symptom	Diagnosis	Remedy
Large negative baseline upset at solvent elution with no recovery to the original baseline ("Solvent quenching effect")	Heating current too low.	Slightly increase the source heating current.
Unstable baseline	Background current level too high	Reduce the source heating current value.
	Hydrogen flow fluctuation.	Check pressure regulators on hydrogen line.
Low carbon rejection	Hydrogen flow too high.	Reduce hydrogen flow to the correct operative conditions.

14

Maintaining a Flame Photometric Detector

Chapter at a Glance...

Operating Sequences

Flame Photometric Detector (FPD)

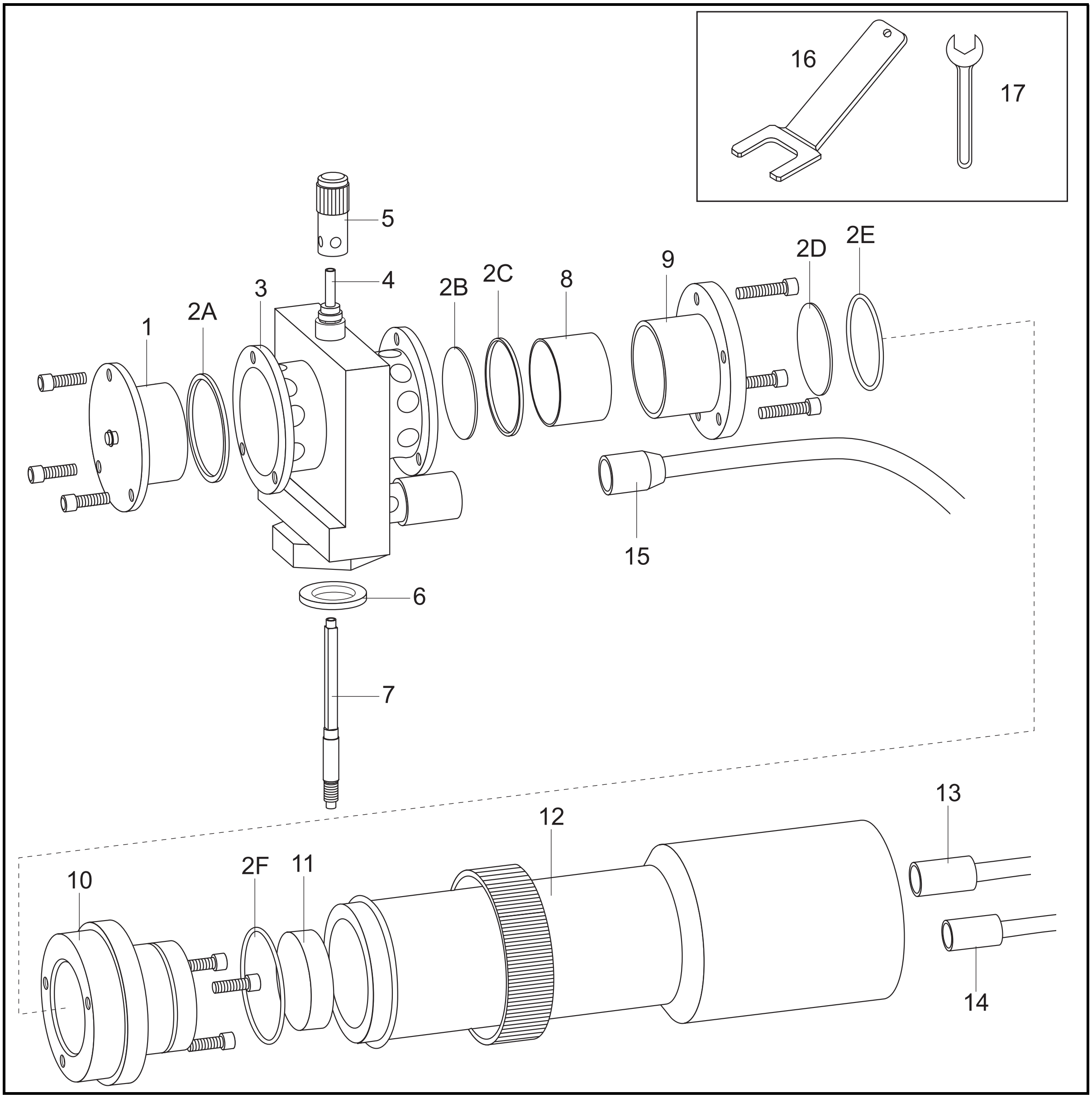


Table 14-1. Flame Photometric Detector (FPD) parts identification table

No.	Description	P/N
1	Mirror plug	350 053 50
2A	Graphite seal	190 045 89
2B	Heat shield (flame side)	
2C	Graphite seal	
2D	Heat shield (filter-side)	
2E	Viton™ O-ring	
2F	Viton™ O-ring	
3	Detector body	
4	Chimney	
5	Chimney cap	
6	Aluminium O-ring	290 326 30
7	Jet	404 045 11
8	Spacer	
9	Flange	
10	Filter support	
11	Interferential filter for phosphorus	281 071 00
	Interferential filter for sulphur	281 070 00
	Interferential filter for tin	281 070 01
12	Photomultiplier assembly	
13	Signal cable	
14	Excitation voltage cable	
15	Ignition/heating multipolar cable	
16	FPD fixing tool	205 021 50
17	5 mm wrench for jet	

FPD Maintenance

To ensure optimum performance of the FPD, you must keep it clean and free of dust and deposits. Symptoms such as reduced sensitivity and increased noise indicate that detector needs cleaning.

To properly maintain the FPD, you should perform the following cleaning or replacement sequences:

- cleaning the jet
- cleaning the interferential filter
- cleaning the mirror metal plug
- cleaning the flame-side heat shield
- replacing the jet
- replacing the interferential filter
- replacing the heat shields

OPERATING SEQUENCE

Clean the Jet

Materials needed:

- Detector fixing tool (P/N 205 021 50)
- 5 mm wrench
- Forceps or tweezers
- Ultrasonic cleaning bath
- Liquid detergent
- Clean compressed air
- GC-grade methanol
- Paper towels

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

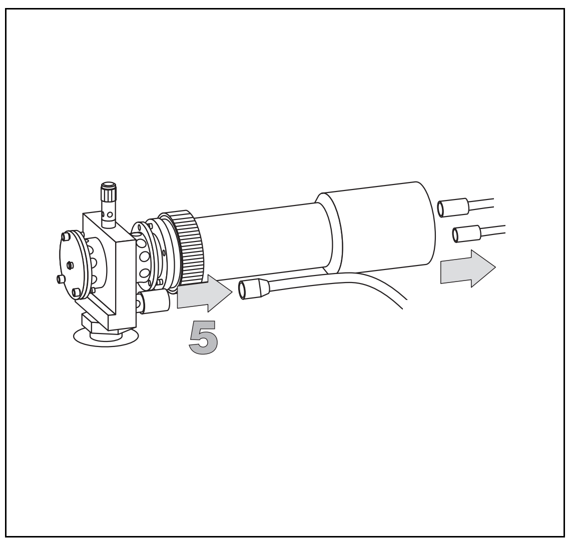
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

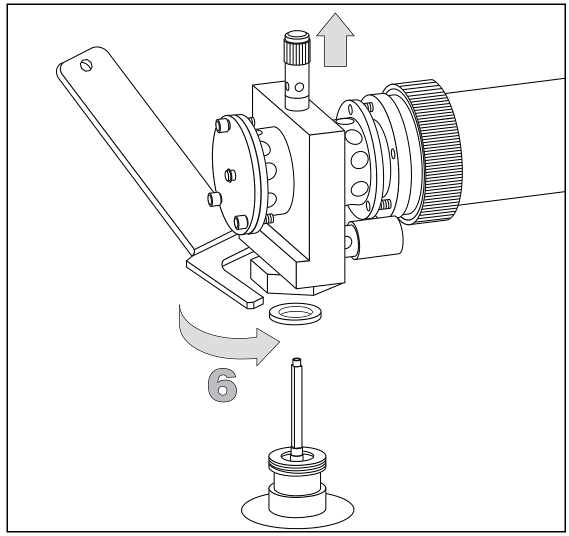
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.
4. Switch off the GC power supply.

WARNING! **Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.**

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.

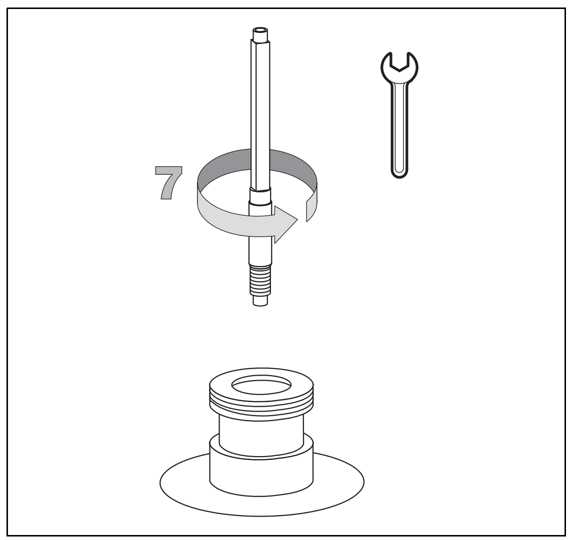


6. Using the tool provided with the GC, loosen the fixing nut on the base of the detector and remove it.



CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**

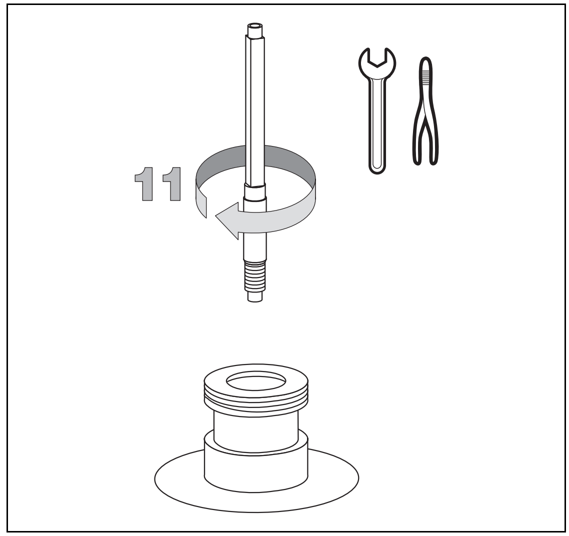
7. Using the 5 mm wrench provided with the GC, loosen the jet and remove it from the detector base body.



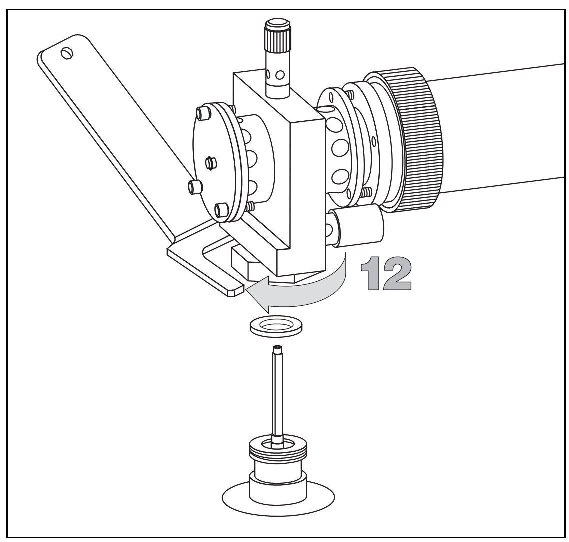
8. Place the jet in the ultrasonic bath filled with liquid detergent and clean it for about five minutes.
9. Handling the jet with forceps or tweezers, rinse the jet with distilled water, then with methanol.
10. Place the jet on a paper towel and let it air dry.

PRECAUTIONS

11. When the jet is dry, insert the jet into the detector base body and tighten it.

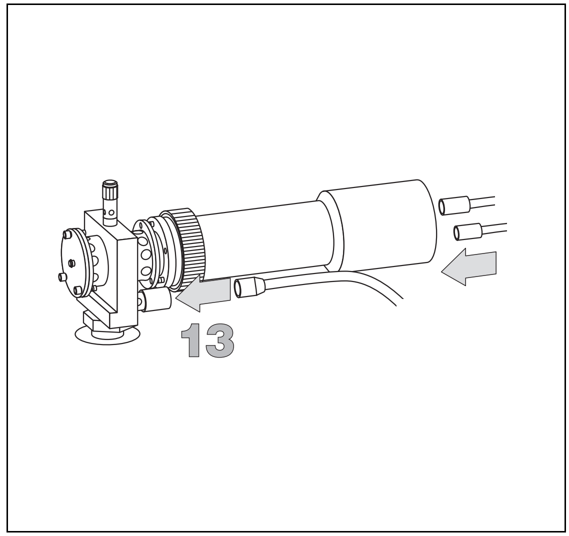


12. Place the detector on its base body, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



13. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.

14. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Clean the Interferential Filter

Materials needed

- Detector fixing tool (P/N 205 021 50)
- Paper towels
- GC-grade methanol

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

PRECAUTIONS

WARNING! When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.

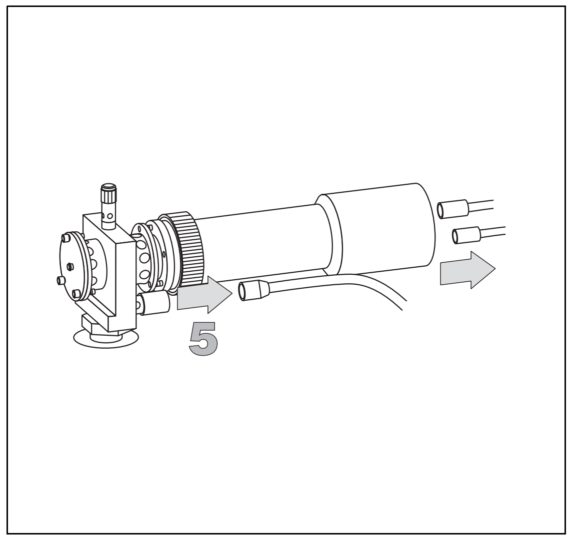
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.
2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

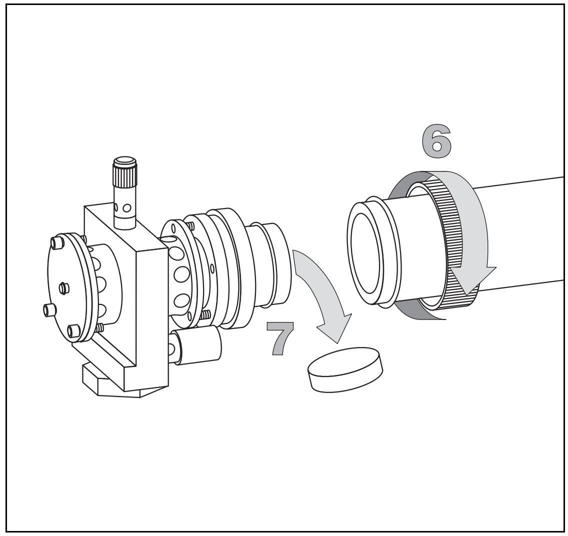
4. Switch off the GC power supply.

WARNING! **Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.**

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.



6. Loosen the knurled nut that fixes the photomultiplier tube and remove it from the detector body.



CAUTION **The photomultiplier tube could damage if exposed to ambient light with the excitation voltage** `On`**. Make sure the GC power supply has been switched off before disconnecting the tube from the detector body.**

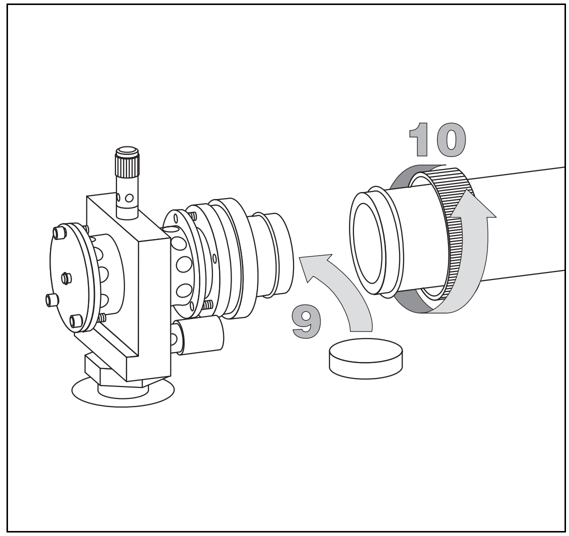
7. Remove the interferential filter from its housing, handling it very gently. Keep it using a clean paper towel.

CAUTION **Filters are fragile. Pay attention not to let the filter fall down and damage.**

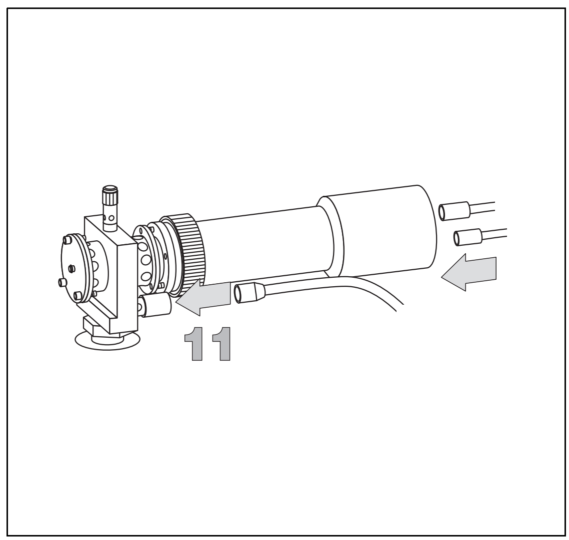
8. Using a clean paper towel, clean the filter on both faces.

CAUTION **Avoid touching the filter with your fingers. If you see fingertips on the filter, clean it using GC-grade methanol and air dry before remounting.**

9. Insert the cleansed filter into its housing. The mirror face must be oriented towards the flame.
10. Reassembly the photomultiplier tube and the detector body, then fix them together tightening the knurled nut.



11. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.
12. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Clean the Mirror Metal Plug

Materials needed

- Detector fixing tool (P/N 205 021 50)
- 1 mm Allen wrench
- Paper towels
- Methylene chloride or methanol

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

PRECAUTIONS

WARNING! When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.

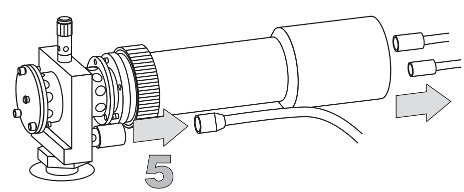
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.
4. Switch off the GC power supply.

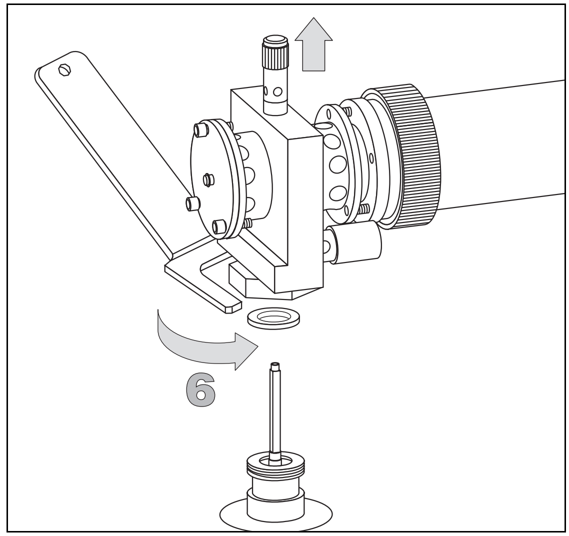
WARNING! Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.



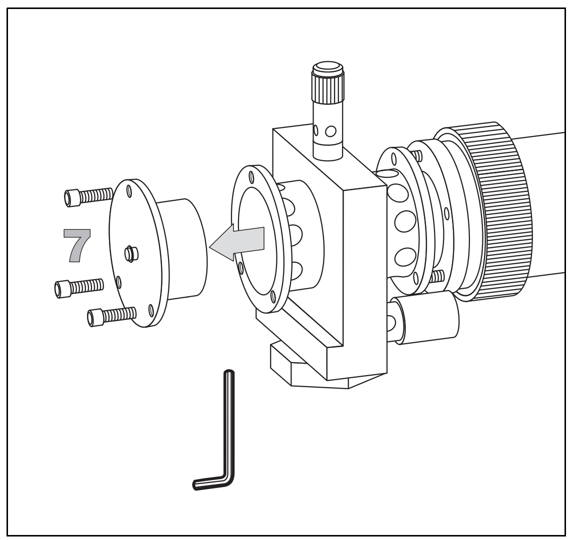
6. Using the tool provided with the GC, loosen the fixing nut on the base of the detector and remove it.

CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**

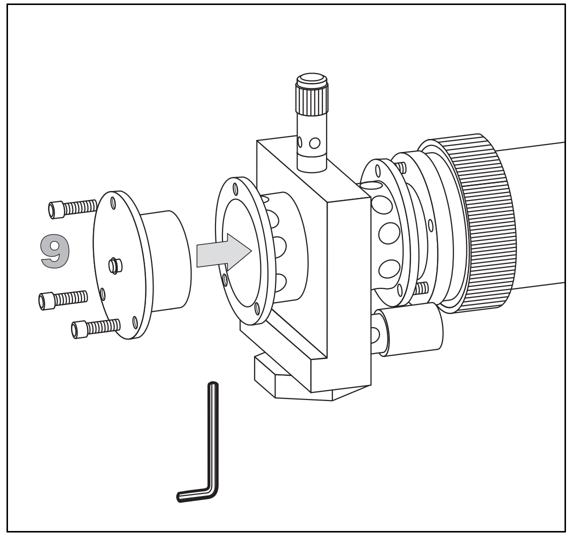


7. Loosen the Allen screws on the mirror plug and remove it.

8. Using a clean paper towel, clean the mirror surface of the plug. If necessary, use a solvent as methylene chloride or methanol to remove deposits and a metal polishing paste to restore it to the previous reflectivity.

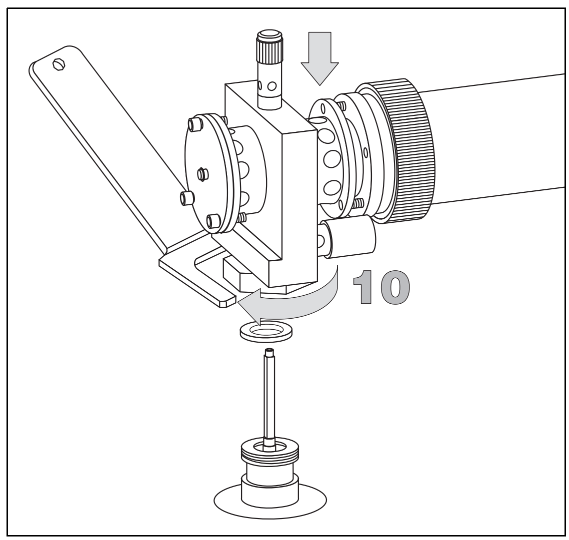


9. Insert the mirror plug in the detector body and fix it with the relevant Allen screws.

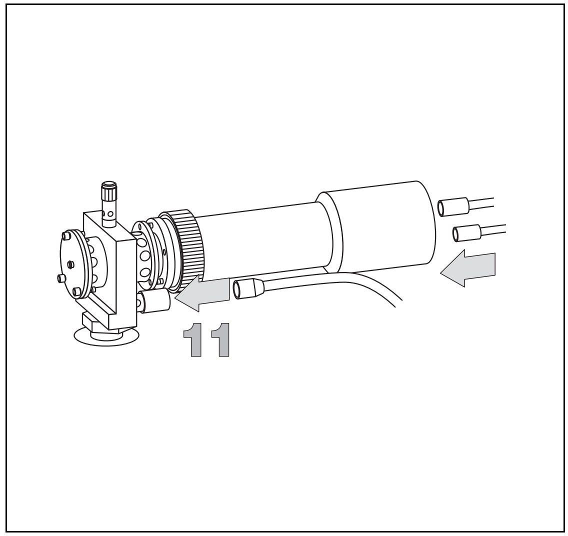


NOTE Before reinserting the plug, inspect the graphite seal inside the plug's housing. If the seal is damaged and could not ensure tightness, it must be removed and replaced with a new one.

10. Place the detector on its base body, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



11. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.



12. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE^{TM} GC Ultra *Operating manual* for more information.

OPERATING SEQUENCE

Clean the Flame-side Heat Shield

Materials needed

- Detector fixing tool (P/N 205 021 50)
- 1 mm Allen wrench
- Cotton sticks
- Paper towels
- Methylene chloride or methanol

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

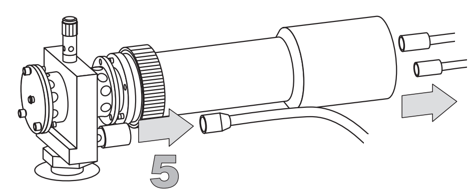
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.
4. Switch off the GC power supply.

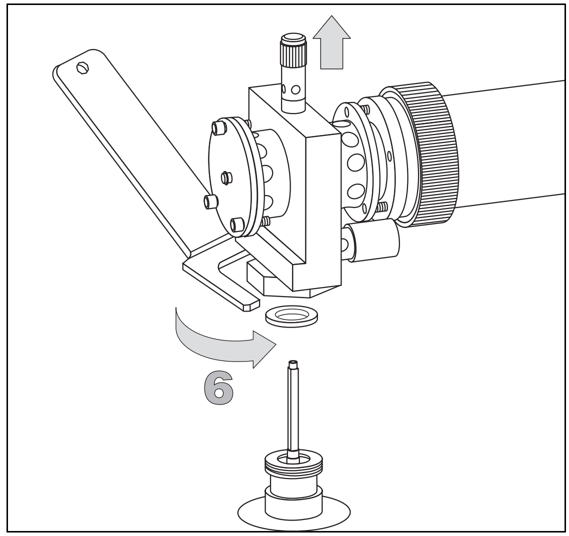
WARNING! **Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.**

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.



6. Using the tool provided with the GC, loosen the fixing nut on the base of the detector and remove it.

CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**

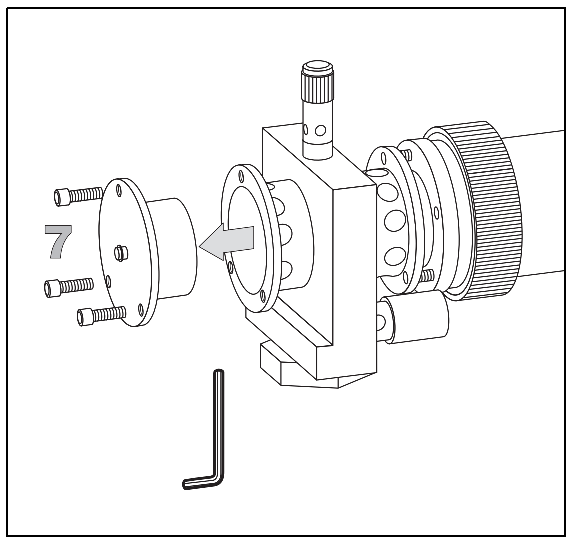


7. Loosen the Allen screws on the mirror plug and remove it.

8. With the aid of a cotton stick, clear the surface of the heat shield that faces the flame. If necessary, use a solvent like methylene chloride or methanol to help cleaning the heat shield.

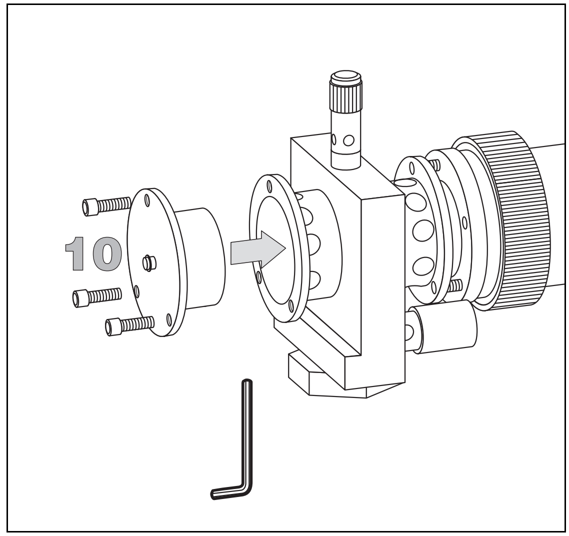
CAUTION **While cleaning the heat shield, pay attention not to damage the ignition coil.**

9. Using a clean paper towel, clean the mirror surface of the plug. If necessary, use a solvent as methylene chloride or methanol to remove deposits and a metal polishing paste to restore it to the previous reflectivity.

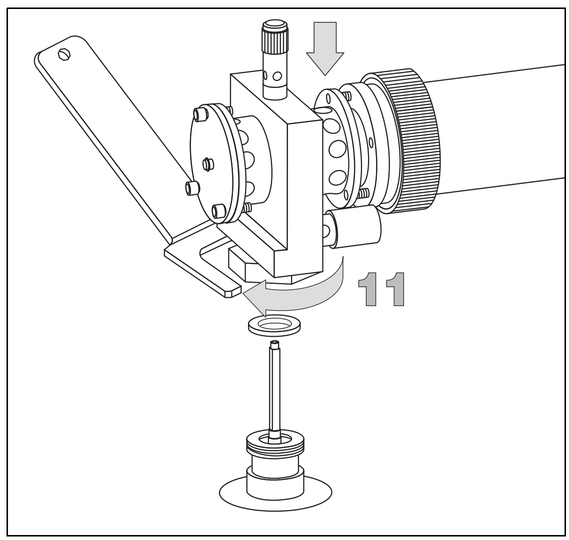


10. Insert the mirror plug in the detector body and fix it with the relevant Allen screws.

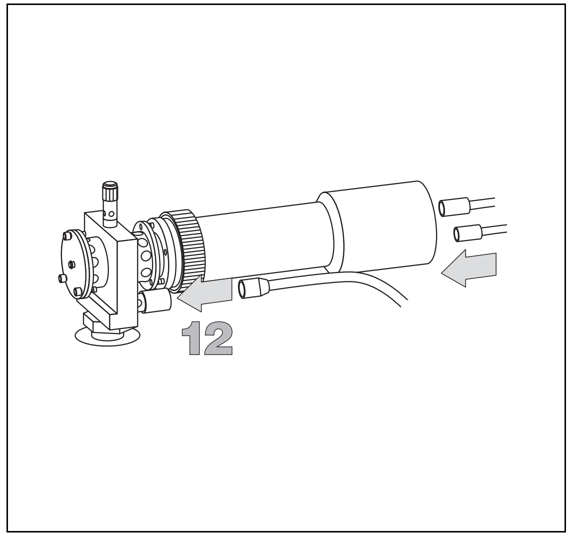
NOTE Before reinserting the plug, inspect the graphite seal inside the plug's housing. If the seal is damaged and could not ensure tightness, it must be removed and replaced with a new one.



11. Place the detector on its base body, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



12. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.
13. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Jet

Materials needed

- Detector fixing tool (P/N 205 021 50)
- 5 mm wrench
- Jet for FPD (P/N 404 045 11)

WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.

PRECAUTIONS

1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

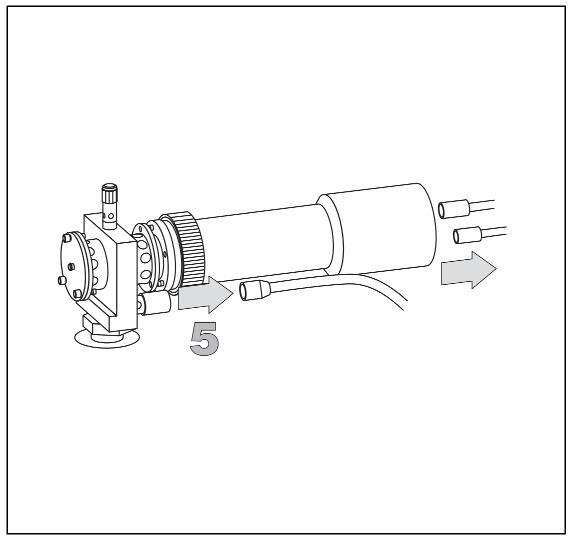
RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.

4. Switch off the GC power supply.

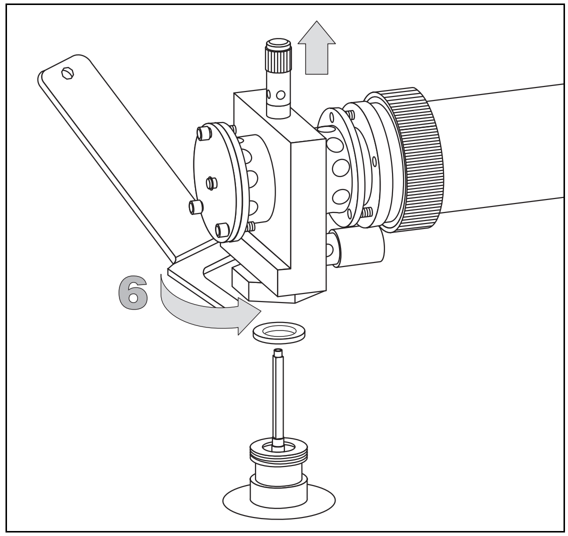
WARNING! **Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.**

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.

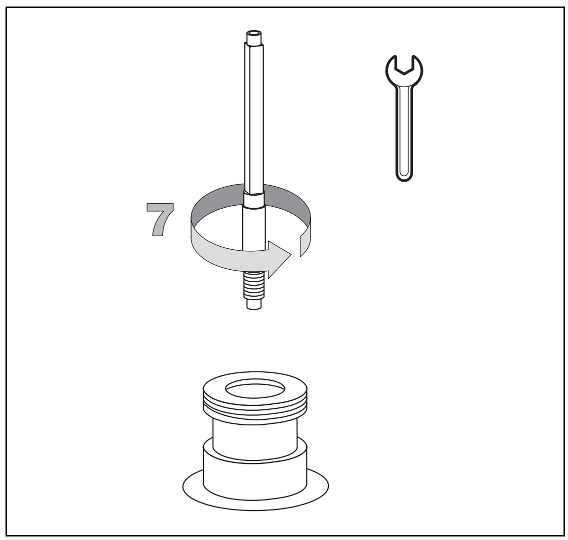


6. Using the tool provided with the GC, loosen the fixing nut on the base of the detector and remove it.

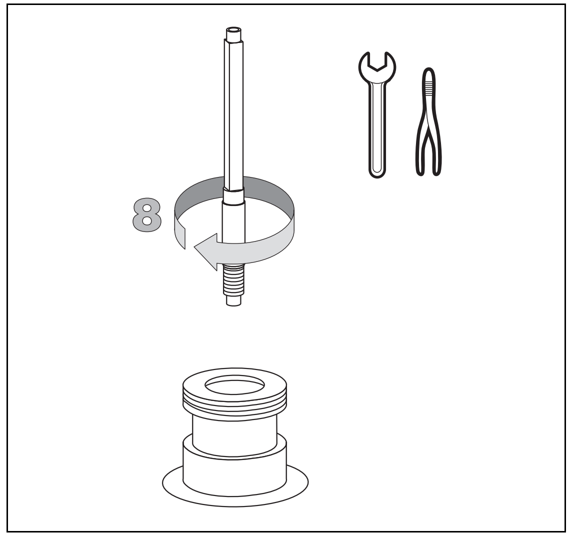
CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**



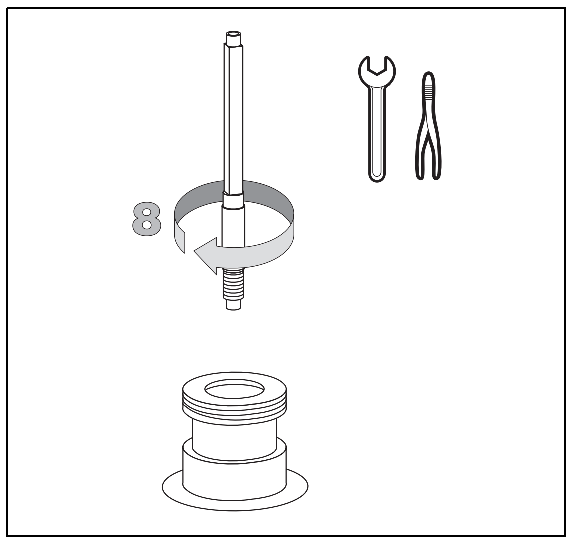
7. Using the 5 mm wrench provided with the GC, loosen the worn jet and remove it from the detector base body.



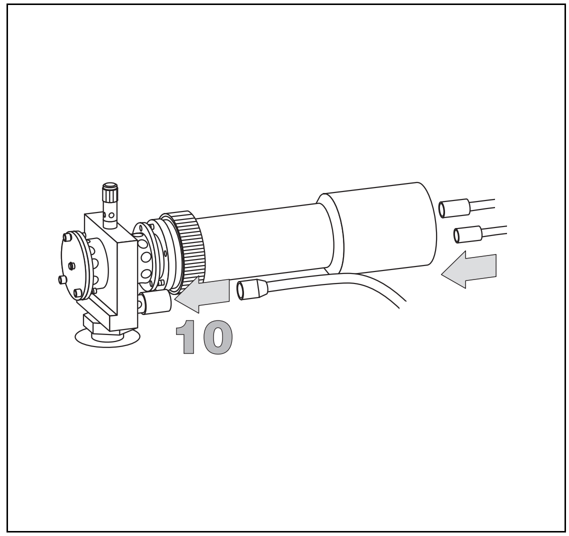
8. Place a new jet into the housing and tighten it.



9. Place the detector on its base body, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



10. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.
11. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Interferential Filter

Materials needed

- Detector fixing tool (P/N 205 021 50)
- Interferential filter (see Table 14-2 for available filters)
- Paper towels

Table 14-2. Available interferential filters for the FPD detector

	P/N
Phosphorus	281 071 00
Sulphur	281 070 00
Organotin	281 070 01

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

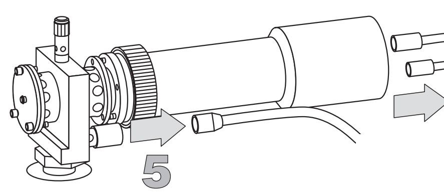
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `FPD temp`, `H2` and `Air` and press **OFF**.
4. Switch off the GC power supply.

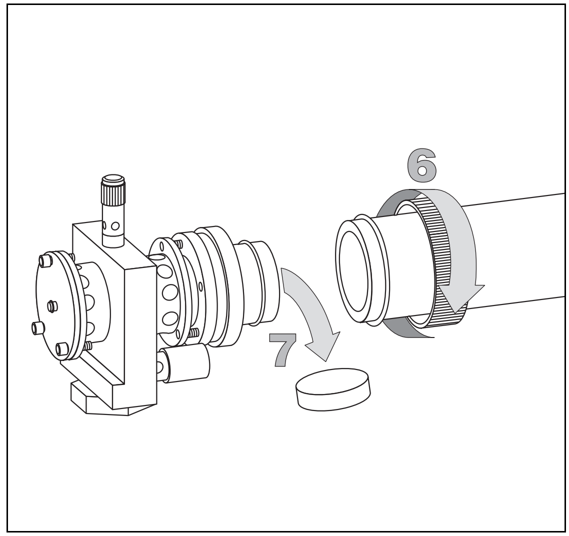
WARNING! **Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.**

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.



6. Loosen the knurled nut that fixes the photomultiplier tube and remove it from the detector body.

CAUTION **The photomultiplier tube could damage if exposed to ambient light with the excitation voltage set to `On`. Make sure the GC power supply has been switched off before disconnecting the tube from the detector body.**

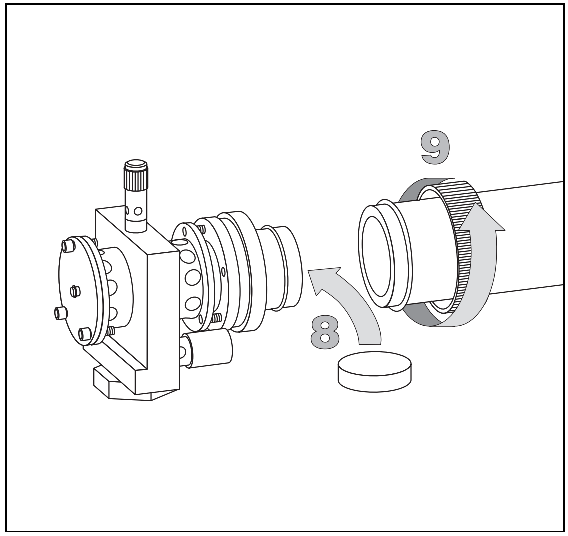


7. Remove the interferential filter from its housing, handling it very gently. Keep it using a clean paper towel.

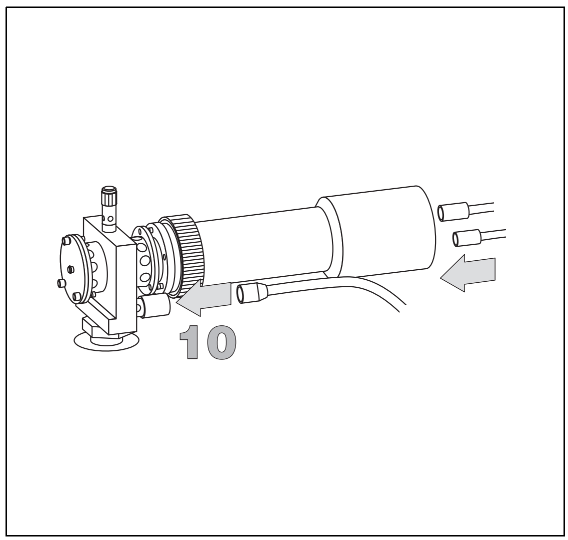
CAUTION **Filters are fragile. Pay attention not to let the filter fall down and damage.**

Avoid touching the filter with your fingers. If you see fingertips on the filter, clean it using GC-grade methanol and air dry before remounting.

8. Insert the new filter into its housing. The mirror face must be oriented towards the flame.
9. Reassembly the photomultiplier tube and the detector body, then fix them together tightening the knurled nut.



10. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.
11. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



OPERATING SEQUENCE

Replace the Heat Shields

Materials needed

- Detector fixing tool (P/N 205 021 50)
- FPD maintenance kit (P/N 190 045 89)
- 1 mm Allen wrench
- Screwdriver
- Methylene chloride or methanol
- Paper towels

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detector must be cooled to room temperature.**

PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

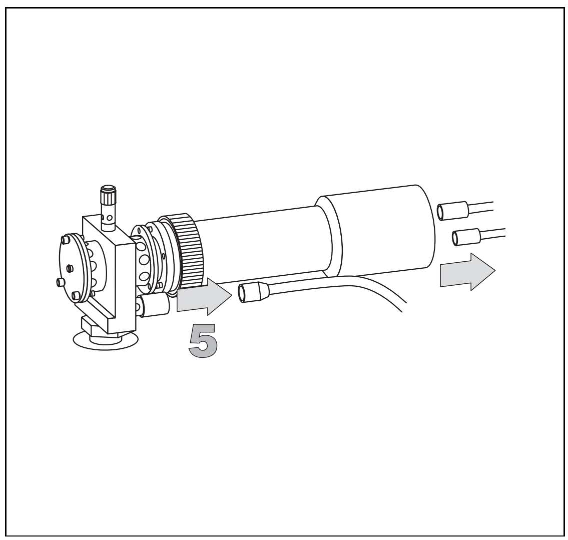
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the FPD detector, to access the FPD control table. In the following example, a FPD installed on the right channel is considered.

RIGHT DET (FPD)		
Flame		Off <
Base temp	300	Off
FPD temp	150	Off
Signal pA		(xx)
High voltage mode?		N
H2	90	Off
Air	115	Off
Mkup(N2)	0	0

2. Scroll to `Flame` and press **OFF**.
3. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.
4. Switch off the GC power supply.

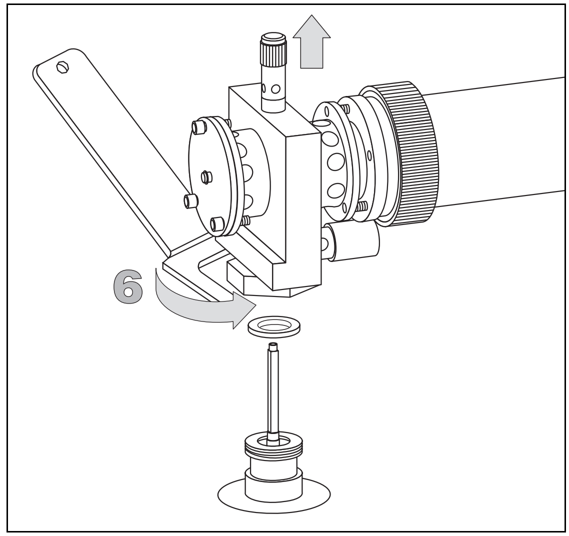
WARNING! Before disconnecting the cables or disassembling the detector, switching off the GC power supply is indispensable to avoid electrical shocks and damages to the instrument.

5. Disconnect the signal, excitation voltage and ignition/heating cables from the detector.



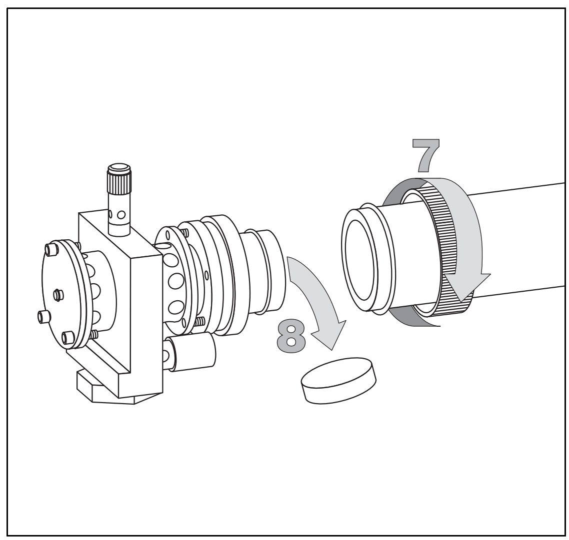
6. Using the tool provided with the GC, loosen the fixing nut on the base of the detector and remove it.

   CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**



7. Loosen the knurled nut that fixes the photomultiplier tube and remove it from the detector body.

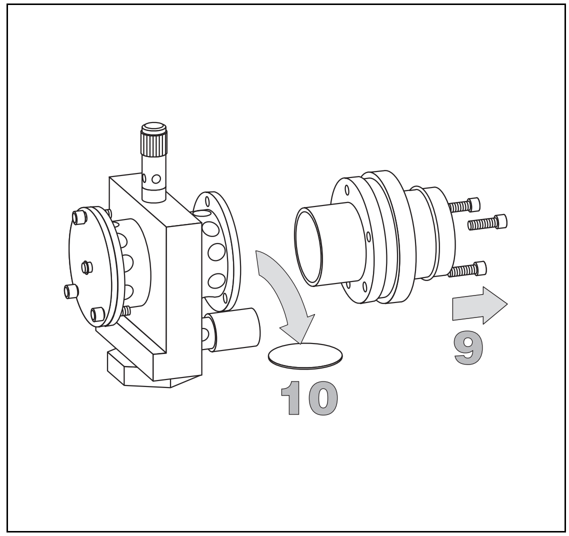
   CAUTION **The photomultiplier tube could damage if exposed to ambient light with the excitation voltage set to `On`. Make sure the GC power supply has been switched off before disconnecting the tube from the detector body.**



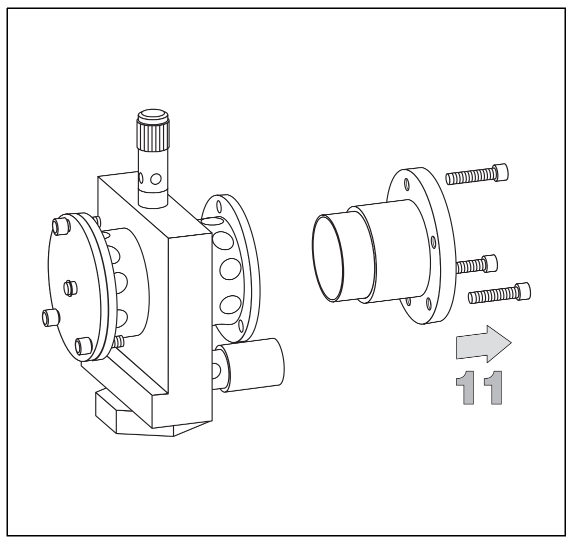
8. Remove the interferential filter from its housing, handling it very gently. Keep it with a clean paper towel and put it aside.

   CAUTION **Filters are fragile. Pay attention not to let the filter fall down and damage.**

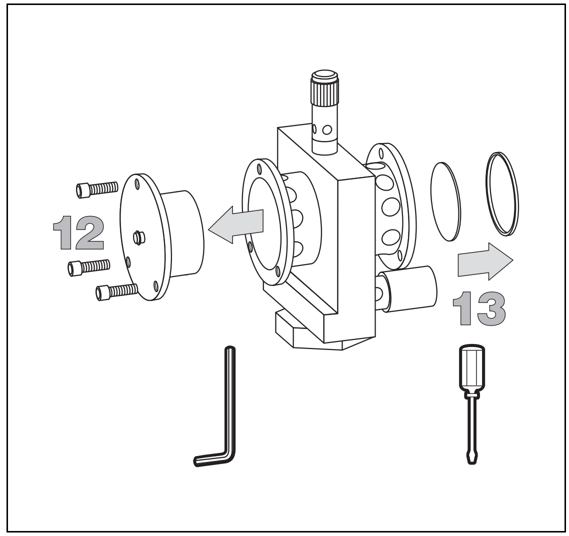
9. Loosen the three Allen screws that fix the filter support to the flange of the detector body and remove it.
10. Turn down slowly the detector body and let the filter-side heat shield come out from the flange. Pay attention not to lose the O-ring inserted in the filter support.



11. If you want to replace only the filter-side heat shield, go to # 21. If you want to replace the flame-side heat shield, loosen the three Allen screws that fix the flange to the detector body and remove it with the spacer that should remain inserted in the flange. Now you could access the flame-side heat shield and the relevant graphite seal.

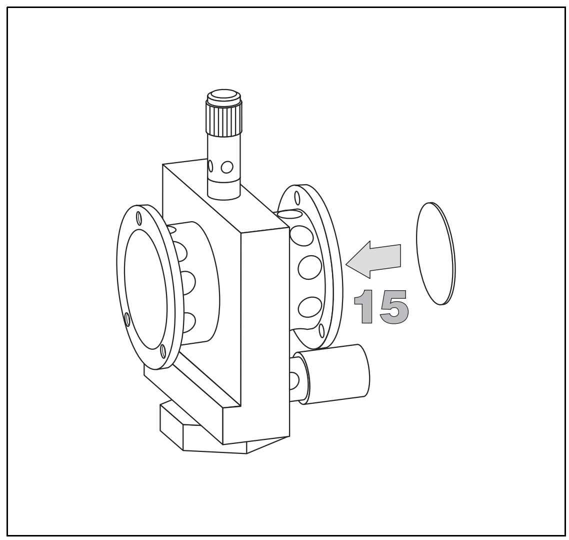


12. Loosen the Allen screws that fix the mirror plug and remove it.
13. Insert the handle of a screwdriver or other unsharpened tool in the combustion chamber and push the flame-side heat shield and its graphite seal out from its housing. Act gently to avoid breaking of the heat shield.

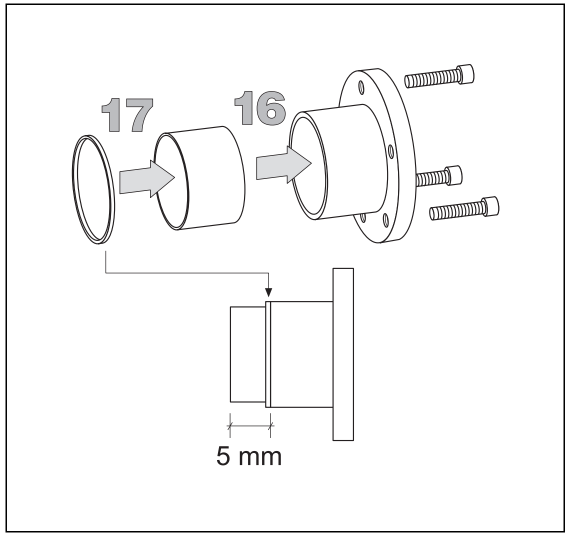


CAUTION **While pushing out the heat shield, pay attention not to damage the ignition coil.**

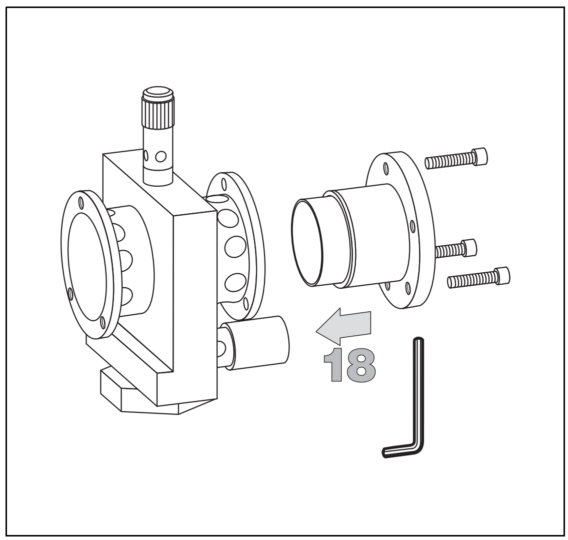
14. Remove with care traces of graphite due to the breaking of the seal.
15. Insert a new heat shield into its housing inside the detector body.



16. Insert the spacer into the flange, letting it stand out for about 5 mm.
17. Insert a new graphite seal on the spacer, pushing it slowly until it touches the flange.

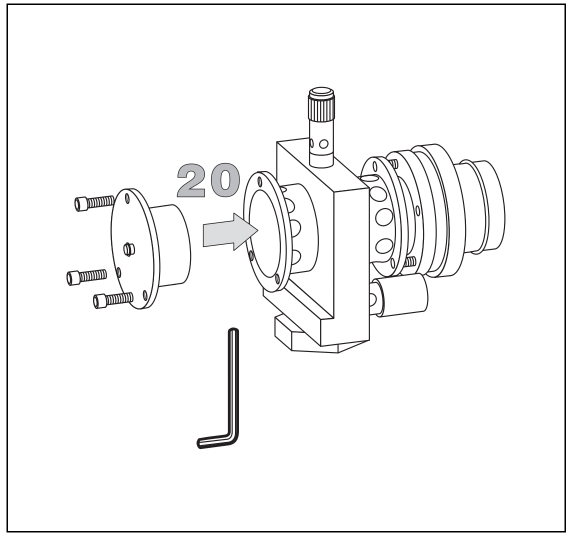


18. Insert the flange into the detector body and fix it tightening the three Allen screws.
19. Using a clean paper towel, clean the mirror surface of the plug. If necessary, use a solvent as methylene chloride or methanol to remove deposits and a metal polishing paste to restore it to the previous reflectivity.

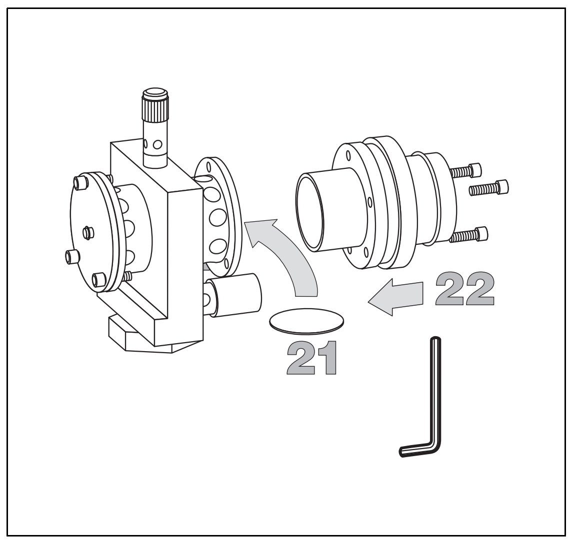


20. Insert the mirror plug in the detector body and fix it with the relevant Allen screws.

NOTE Before reinserting the plug, inspect the graphite seal inside the plug's housing. If the seal is damaged and could not ensure tightness, remove it and replace with a new one.

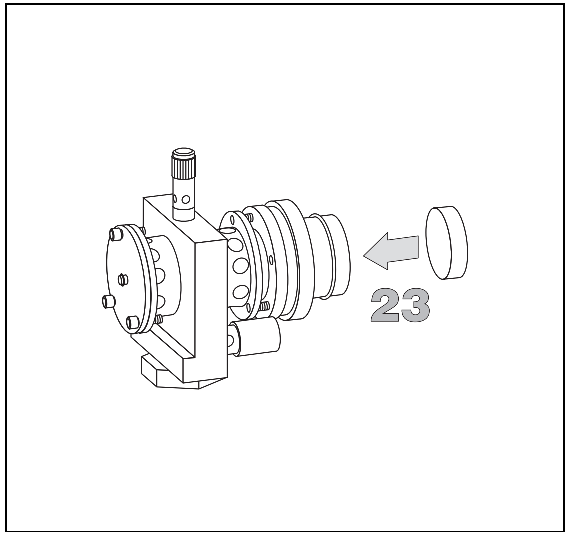


21. Insert a new heat shield into its housing inside the flange.
22. Insert the filter support into the flange and fix it tightening the three Allen screws. Check the O-ring that fixes the heat shield is in place before tightening the screws.

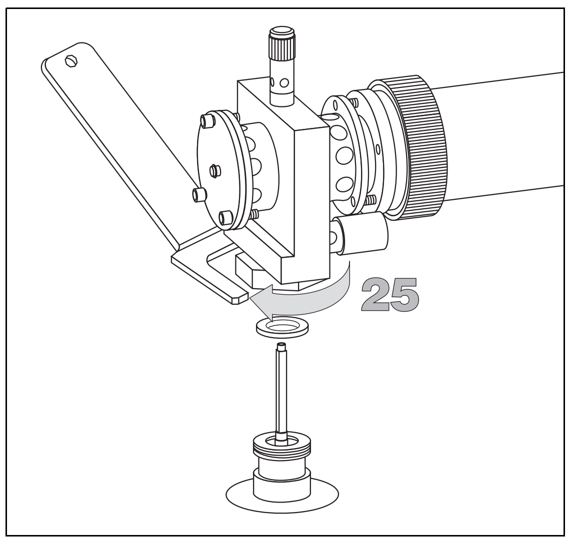


23. Reinsert the filter into the support. The mirror face must be oriented towards the flame

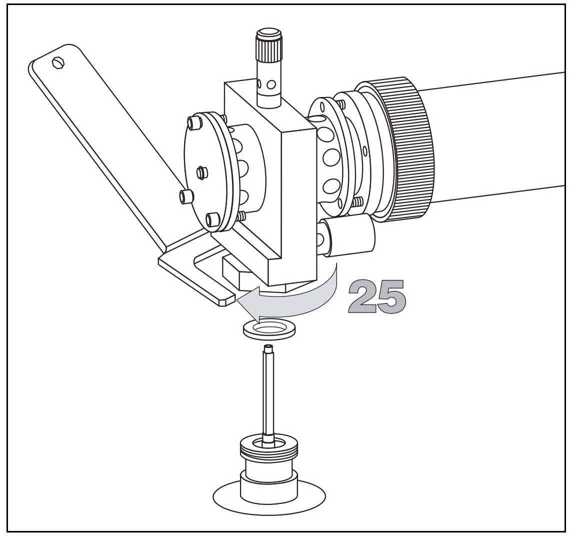
CAUTION **Avoid touching the filter with your fingers. If you see fingertips on the filter, clean it using a clean paper towel and, if needed, GC-grade methanol before remounting.**



24. Reassembly the photomultiplier tube and the detector body, then fix them together tightening the knurled nut.

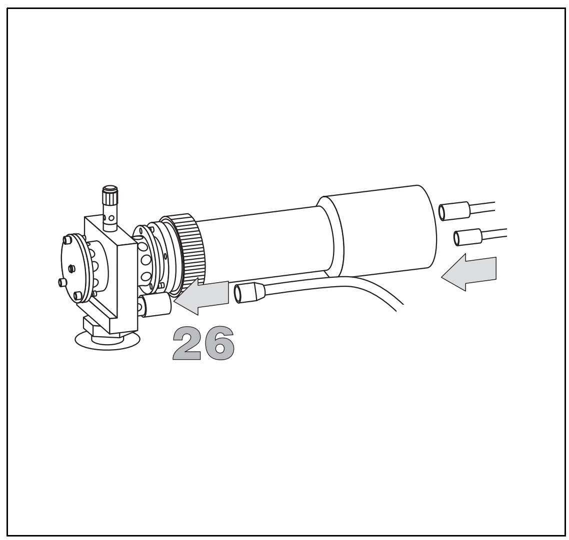


25. Place the detector on its base body, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



26. Reconnect the signal, excitation voltage and ignition/heating cables to the detector.

27. Switch on the GC power supply and set the detector to the required operating conditions. Refer to the TRACE™ GC Ultra *Operating manual* for more information.



FPD Troubleshooting

Sensitivity

The FPD sensitivity is affected by the temperature. The FPD response decreases with an increase in the detector temperature. In addition, the detector response can be severely reduced if a non-sulphur or non-phosphorus compound is partially or fully eluted with the analyte of interest (**quenching**).

Keep clean the interferential filter and the heat shields as described in the maintenance sequences, to avoid reducing sensitivity. Replace the heat shields if the deposits due to contamination cannot be eliminated.

Gases

A right choice of the flow rates of hydrogen and air (fuel gases) is of primary importance in determining FPD sensitivity. Flow rates will affect also selectivity and, to a great extent, peak shapes.

Optimum carrier gas flow rate depends on the type of gas used and on the diameter of the column installed.

The gases normally used with FPD are reported in the Table 14-3

Table 14-3. Gases used for FPD

		Fuel gas	Make-up gas
Capillary Columns	Packed Columns		
Helium Nitrogen Hydrogen	Helium Nitrogen Argon	Hydrogen + Air	Generally not required

The following values of flow rate are recommended:

Table 14-4. Recommended gas flow rates for FPD

	Capillary columns	Packed columns
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 found experimentally, setting a proper value for the hydrogen and performing some analyses on a standard mixture at different air flow rates.

WARNING! It is the customer's responsibility to ensure compliance with all local safety regulations concerning gas supplies.

Hydrogen is a potentially dangerous gas. Mixed with air it may give rise to an explosive mixture. The use of hydrogen requires the operator's extreme caution due to the risks involved. For further details concerning hydrogen, please refer to *Using Hydrogen* on page xvii.

Troubleshooting the FPD

Table 14-5. FPD troubleshooting table

Symptom	Diagnosis	Remedy
High standing current and noise	Detector conditioning not completed	Increase detector conditioning time
	Air flow rate set too high	Reduce air flow rate
	Detector not light-tight	Tighten the chimney cap and the knurled nut that fixes the photomultiplier tube
	Interferential filter missing	Check the interferential filter has been correctly installed
No standing current	Signal or power cables incorrectly connected	Check connections
	Faulty photomultiplier tube	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
Unstable and excessively noisy baseline	Faulty photomultiplier tube	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
FPD temperature does not reach the set point	Power cable not properly connected	Check cable connection
	Faulty heater	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

Table 14-5. FPD troubleshooting table (Continued)

Symptom	Diagnosis	Remedy
Low sensitivity	Low photomultiplier excitation voltage	Set `High Voltage Mode` to `Yes` in the FPD control table
Low sensitivity	Inadequate hydrogen flow rate	Set the hydrogen flow rate to a proper value. Refer to Table 14-4, *Recommended gas flow rates for FPD* on page 311
	Inadequate air flow rate	Set the air flow rate to a proper value. Refer to Table 14-4, *Recommended gas flow rates for FPD* on page 311 and relevant note
	Dirty interferential filter	Clean the interferential filter. Refer to *Clean the Interferential Filter* Operating Sequence on page 278
	The mirror of the combustion chamber is dirty	Clean the mirror. Refer to *Clean the Mirror Metal Plug* Operating Sequence on page 282
	The flame-side heat shield is dirty	Clean the flame-side heat shield. Refer to *Clean the Flame-side Heat Shield* Operating Sequence on page 287
	Reduced transparency of the heat shields	Replace the heat shields. Refer to *Replace the Heat Shields* Operating Sequence on page 301
Low sensitivity. Water droplets generate between the heat shields	Leak at the flame-side heat shield	Replace the heat shields. Refer to *Replace the Heat Shields* Operating Sequence on page 301 If the symptom does not disappears, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

15

Maintaining a Photoionization Detector

Chapter at a Glance…

Operating Sequences

Photoionization Detector (PID)

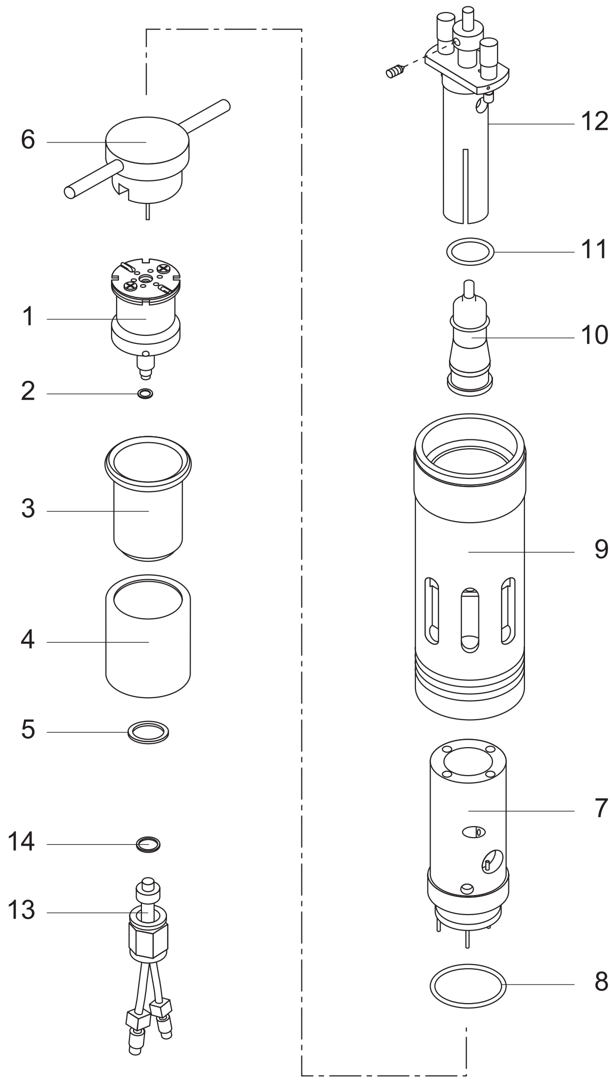


Table 15-1. Photoionization Detector (PID) parts identification table

No.	Description	P/N
1	Detector cell assembly	231 050 20
2	Seal	290 150 58
3	Stainless steel bell	
4	Insulation jacket	
5	Seal	290 147 01
6	Fixing tool	205 031 00
7	Lamp housing	
8	Viton™ O-ring for lamp holder	290 203 25
9	Heat sink	
10	UV lamp 8.4 eV	305 030 13
	UV lamp 9.6 eV	305 030 14
	UV lamp 10.6 eV	305 030 15
	UV lamp 11.8 eV	305 030 16
11	Viton™ O-ring for UV lamp	290 203 38
12	Lamp holder	
13	Capillary adapter	350 438 25
14	Silver seal	290 150 59

PID Maintenance

The Photoionization Detector requires relatively little maintenance. To properly maintaining the detector, you should perform the following sequences:

- UV lamp replacement
- Cleaning the UV lamp window

The UV Lamp must be replaced when exhausted, faulty or when a different class of compounds is to be analyzed. Table 15-2 reports the available lamps and their application field.

Table 15-2. Available UV lamps for PID detection

	Application
8.4 eV	Determination of amines and polycyclic aromatic compounds.
9.6 eV	Determination of low-boiling aromatic compounds (BTEX analyses)
10.6 eV	General applications. Filled with krypton, emits at 10.0 and 10.6 eV.
11.8 eV	Determination of aldehydes and ketones.

NOTE Keep always a spare UV lamp in your laboratory because the lamp could collapse suddenly, without warning signs.

CAUTION **Make sure to have appropriate filters installed on the carrier and make-up gas lines, and replace them regularly.**

Leaks

Checking for leaks a system fitted with a PID detector requires, beside the usual operations that are described in the relevant chapter (see *Ensuring Tightness* on page 179), an additional test. This operation, that must be executed with the detector installed, is reported in *Checking for Leaks*, page 328.

OPERATING SEQUENCE

Replace the UV Lamp

Materials needed

- UV Lamp (see Table 15-2 on page page 318)
- Viton™ O-ring for UV lamp (P/N 290 203 38)
- Cotton gloves

WARNING! **UV radiation can cause injuries to your eyes. Therefore, before beginning the sequence, make sure the UV lamp has been switched off.**

PRECAUTIONS

WARNING! **The lamp replacement can be carried out at operating temperature, without removing the detector from its base body. Nevertheless, keep in mind that parts of the detector are hot and could cause injuries to your hands.**

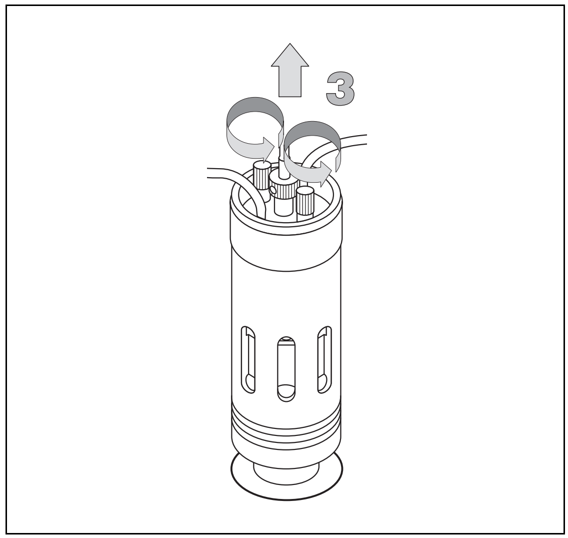
PRECAUTIONS

1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the PID detector, to access the PID control table. In the following example, a PID installed on the right channel is considered.

RIGHT DET (PID)		
Lamp		Off <
Base temp	250	250
High current		N
Signal pA		(xx)
Mkup(N2)	7	Off
Sheath gas	40	Off

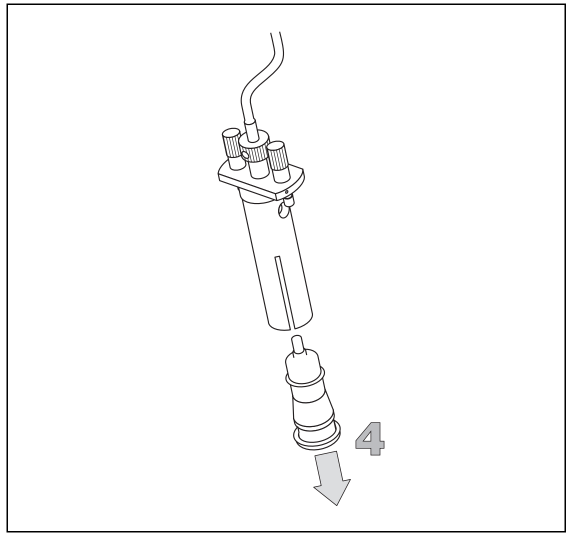
2. Scroll subsequently to `Lamp`, `Mkup` and `Sheath gas` and press **OFF**.

3. Loosen the two knurled screws that fix the lamp holder to the lamp housing and remove it.



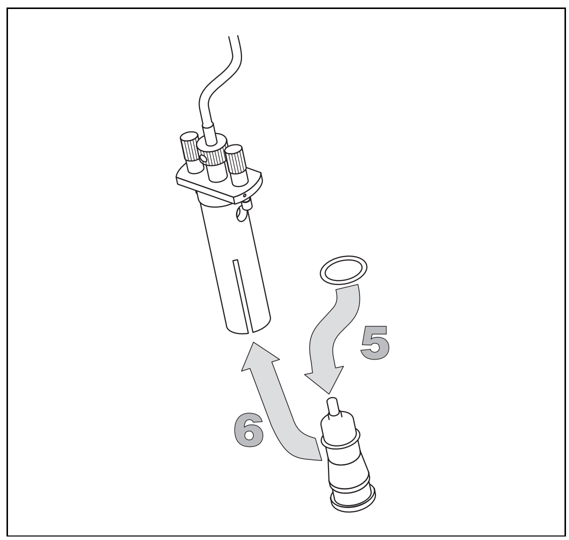
4. Remove the lamp with its Viton™ O-ring from the lamp holder and store it.

CAUTION **Avoid touching the lamp and particularly the lamp window and the O-ring with your fingers. Use cotton gloves during this operation.**

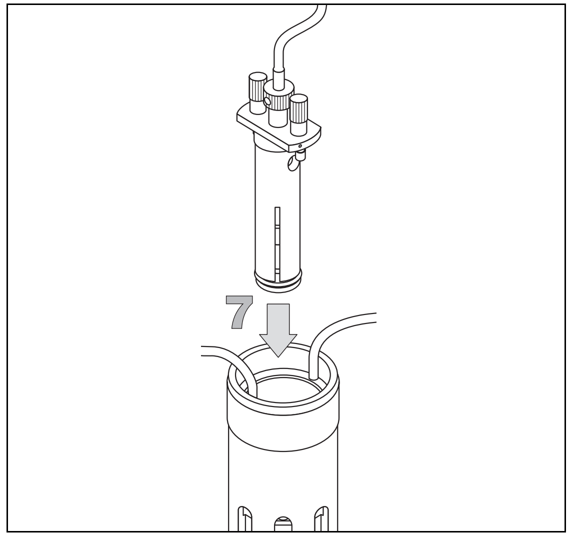


5. Place the proper Viton™ O-ring on the new lamp so to contact its flange.
6. Insert the lamp into the holder, pushing it in until it stops.

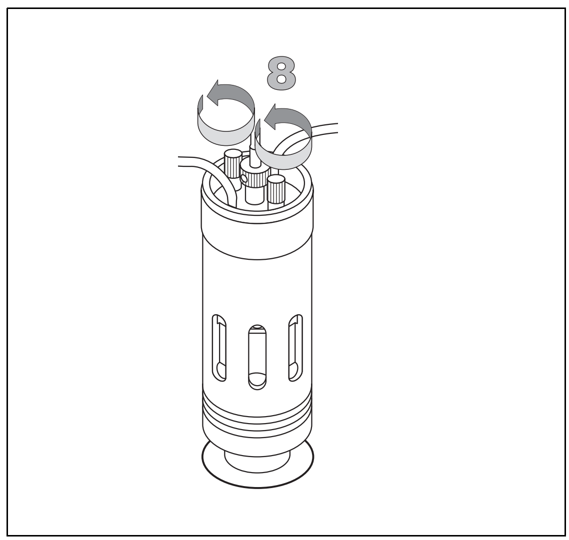
CAUTION **While inserting the lamp, pay attention not to damage the contact pin inside the holder.**



7. Insert the lamp holder into the lamp housing



8. Tighten the two knurled screws to fix the lamp holder to its housing.



9. In the `RIGHT DETECTOR (PID)` control table scroll subsequently to `Mkup`, `Sheath Gas` and `Lamp` and press **ON**.

CAUTION **Before starting a new analysis wait about 30 minutes so the lamp output can stabilize.**

OPERATING SEQUENCE

Clean the Lamp Window

Materials needed

- Lamp Polishing kit (P/N 190 048 00)
- GC-grade methanol or acetone
- Paper towels
- Cotton gloves

WARNING! **UV radiation can cause injuries to your eyes. Therefore, before beginning the sequence, make sure the UV lamp has been switched off.**

PRECAUTIONS

WARNING! **The cleaning of the lamp window can be carried out without removing the detector from its base body. Nevertheless, keep in mind that parts of the detector are hot and could cause injuries to your hands.**

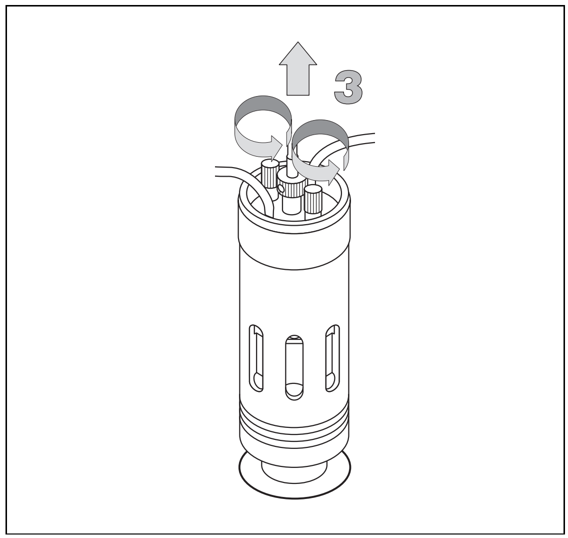
PRECAUTIONS

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

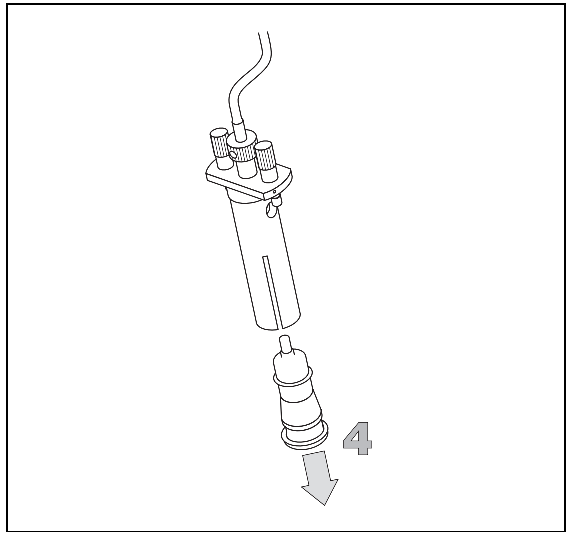
1. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the PID detector, to access the PID control table. In the following example, a PID installed on the right channel is considered.
2. Scroll subsequently to `Lamp`, `Mkup` and `Sheath Gas` press **OFF**.

RIGHT DET (PID)		
Lamp		Off <
Base temp	250	250
High current		N
Signal pA		(xx)
Mkup(N2)	7	Off
Sheath gas	40	Off

3. Loosen the two knurled screws that fix the lamp holder and remove it from the lamp housing.

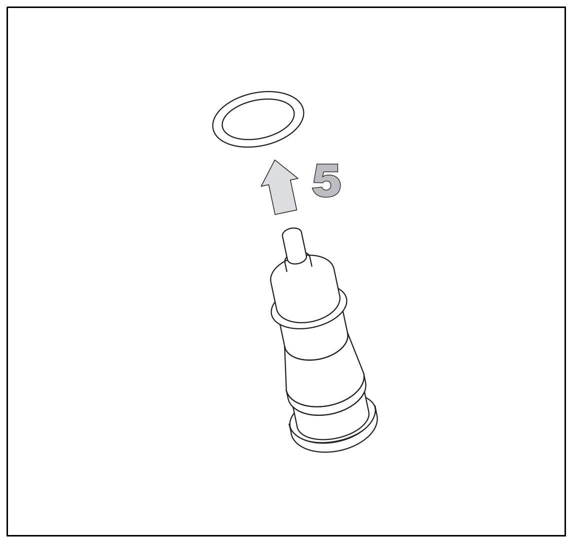


4. Remove the lamp with its Viton™ O-ring from the lamp holder.



WARNING! **Avoid touching the lamp and particularly the lamp window and the O-ring with your fingers. Use cotton gloves during this operation.**

5. Remove the Viton™ O-ring from the lamp.



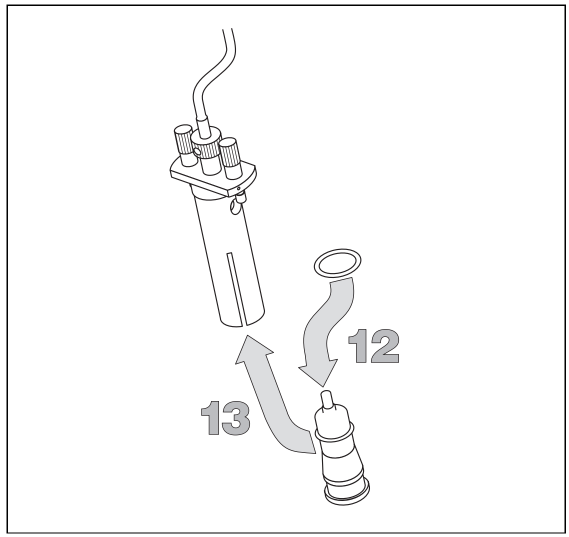
6. Wet the cotton tip of the applicator (included in the lamp polishing kit) with tap water and dip it into the polishing powder, so to pick-up a small amount of it.
7. Swab the lamp window in a circular motion until the UV absorbing film deposited on its surface has been removed.

CAUTION **Apply only a light pressure on the applicator while polishing the window. Heavy pressure could cause abrasion of the quartz surface, resulting in a loss of UV transmission.**

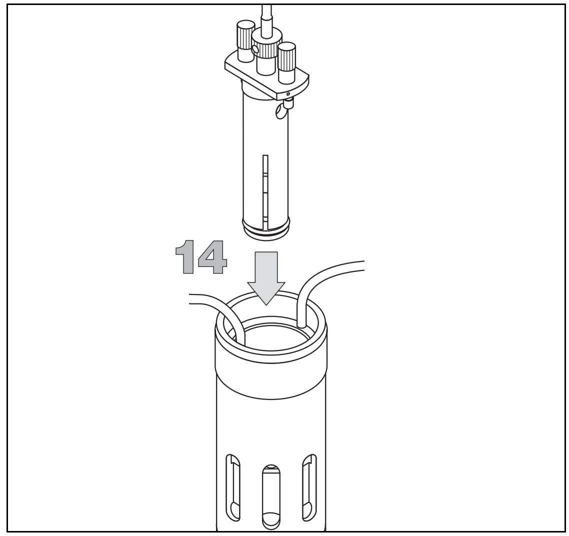
8. Rinse the lamp window with running tap water to remove the residues of polishing powder.

9. Rinse the lamp window with GC-grade methanol or acetone.
10. Wipe the window using a clean paper towel.
11. Air dry the lamp.
12. Insert the Viton™ O-ring on the lamp, pushing it in until it touches the flange.
13. Insert the lamp into the holder, pushing it in until it stops.

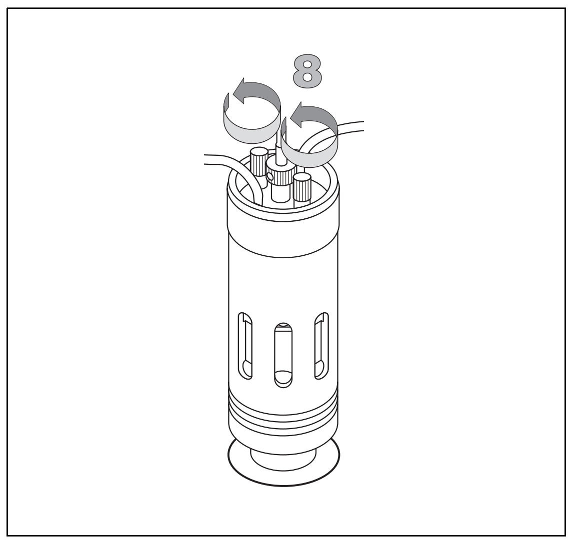
CAUTION **While inserting the lamp, pay attention not to damage the contact pin inside the holder.**



14. Insert the lamp holder into the lamp housing.



15. Tighten the two knurled screws to fix the lamp holder to its housing.
16. In the `RIGHT DETECTOR (PID)` control table scroll subsequently to `Mkup`, `Sheath Gas` and `Lamp` and press **ON**.



OPERATING SEQUENCE

Checking for Leaks

Materials needed

- Thermo Scientific GLD Pro leak detector (or equivalent)
- PID fixing tool (P/N 205 031 00)

WARNING! **UV radiation can cause injuries to your eyes. Therefore, before beginning the operation, make sure the UV lamp has been switched off.**

PRECAUTIONS

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the procedure, the detector must be cooled to room temperature.**

PRECAUTIONS

1. Insert the end of the exit line into a piece of silicon rubber, disconnecting it from the base body of the second detector (if any).
2. Close the purge and split valves (if present) following the instructions reported for the relevant injector in the TRACE™ GC Ultra Operating Manual.

3. Press **LEFT CARRIER** or **RIGHT CARRIER** depending on which injector is operating.
4. Scroll to `Flow mode` and set `con pres`.
5. Scroll to `Pressure` and set `200`.

```
        XXX CARRIER (XXX)
Col. flow          2.5       <
Pressure           100    200
Flow mode            con pres
```

6. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the PID detector, to access the PID control table.
7. Set `Lamp` to `Off`.
8. Set `Mkup(N2)` to `Off`.
9. Set `Sheath` gas to `200`.

```
          XXX DET (PID)
Lamp                      Off <
Base temp          250    250
High current                N
Signal pA                (xx)
Mkup(N2)            30    Off
Sheath gas          40    200
```

10. Wait some minutes until the pressure in the system is equilibrated.
11. Set `Sheath gas` to `Off`.

```
          XXX DET (PID)
Lamp                      Off <
Base temp          250    250
High current                N
Signal pA                (xx)
Mkup(N2)            30    Off
Sheath gas          40    Off
```

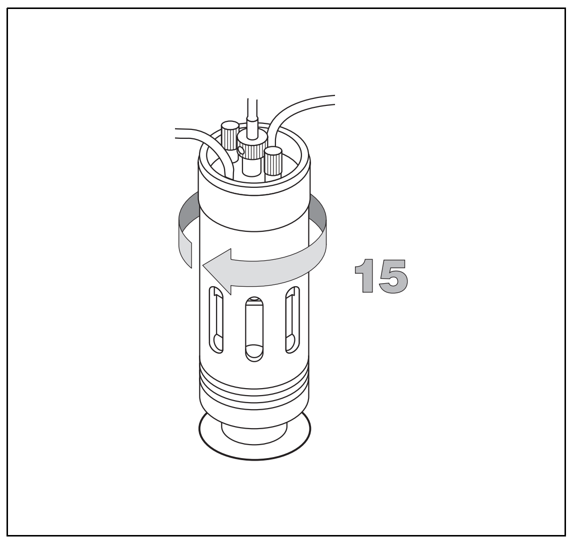
12. Set the carrier pressure to `Off`.

    The actual value should not change more than 5% during two or three minutes. If this occur, you can consider the system free from leaks.

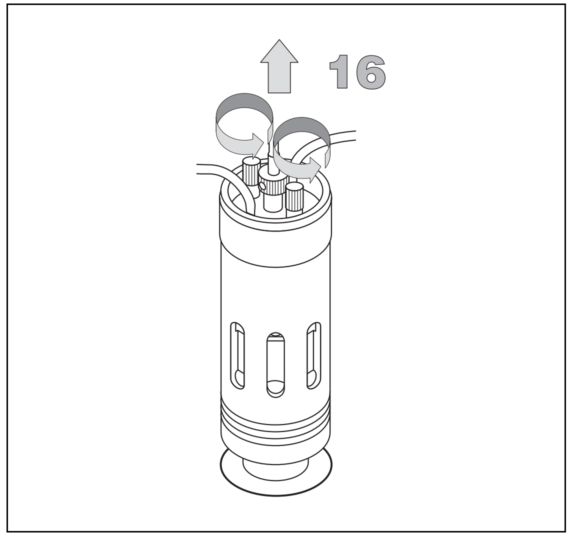
    On the contrary, if the pressure begins to drop immediately, one or more leaks are present.

```
        XXX CARRIER (XXX)
Pressure            30.0    Off
Col. flow           3.00       <
Lin. Veloc.              (60.9)
```

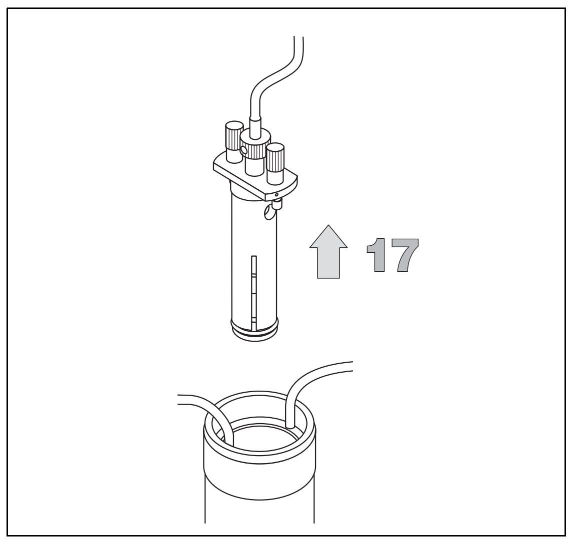
13. Using the leak detector, check the connection between the capillary adapter and the detector, located inside the oven.
14. If a leak is detected, replace the seal of the capillary adapter (#14 in the exploded view reported on page 316). In this case repeat the leak test to ensure complete tightness.
15. If the detector localizes the leaks on the detector body, cool the temperature of the detector to ambient and try to correct them tightening the heat sink, then check again using the detector.



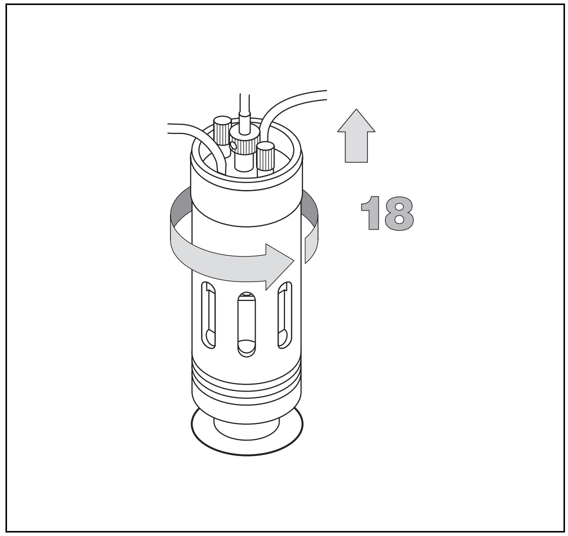
16. If leaks are still detected, loosen the two knurled nuts that fix the lamp holder to the lamp housing.



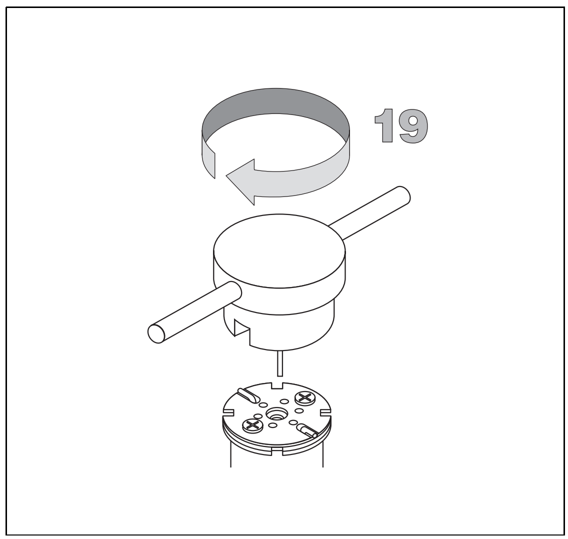
17. Remove the lamp housing.



18. Remove the heat sink.



19. Tighten the detector cell using the fixing tool provided.



20. Reassemble the detector and repeat the leak test.

21. If leaks are still present, disassemble completely the detector and replace the two seals (#5 and #2 of the exploded view reported on page 316).
22. Reassemble the detector and repeat the leak test to ensure complete tightness.

PID Troubleshooting

Sensitivity

Lamp

The most common cause of loss of sensitivity is condensation of the effluent on the lamp window, resulting in a progressive increase of absorption of the UV. For long life, the window must be cleaned periodically according to the operating sequence described in the *Clean the Lamp Window* Operating Sequence on page 323.

A `Lamp failure` message will be displayed when the lamp does not work. One of the possible causes is the breakdown of the lamp. In this case replace the lamp following the instructions reported in the in the *Replace the UV Lamp* Operation Sequence on page 319.

Contamination

An abnormally high baseline may indicate contamination of the detector. In this case, turning off the lamp will not make the symptom disappear. This means that the detector cell has been contaminated.

Try to solve the problem by baking the cell for two hours at 400°C.

If the symptom does not disappear, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

If you suspect contaminants are carried into the detector cell by the gas lines, try reducing the flow rate in each line, one at a time, to identify the contamination source.

To avoid detector contamination install gas filters between the gas cylinders and the detector and replace them periodically.

NOTE To face more effectively possible chromatographic problems, you should carry out an analysis using the test mixture and evaluate the results.

Troubleshooting the PID

Table 15-3. PID troubleshooting table

Symptom	Diagnosis	Remedy
UV Lamp does not light immediately	The lamp has not been used for a long time.	Set `High current mode?` to `Y` until the lamp lights, the reset to `N`
UV Lamp does not light at all. **Message:** `Lamp failure`	Lamp not installed	Install a suitable UV lamp. Refer to the *Replace the UV Lamp* Operating Sequence on page 319.
	Lamp not correctly installed in the lamp holder	Reinstall the UV lamp. Refer to the *Replace the UV Lamp* Operating Sequence on page 319.
	Lamp holder not correctly fixed to the lamp housing	Tighten the two knurled screws that fix the lamp holder to the housing.
	Lamp power cable disconnected	Check the connection
	Polarization cable disconnected	Check the connection
	Safety interlock circuit is faulty	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
	UV lamp is exhausted	Replace UV lamp. Refer to the *Replace the UV Lamp* Operating Sequence on page 319.
No standing current	Power and signal cables not properly connected	Check the connections of the cables
Tailing of solvent peak	Insufficient make-up gas flow rate	Increase the flow rate.

Table 15-3. PID troubleshooting table (Continued)

Symptom	Diagnosis	Remedy
Tailing of sample peaks	Adsorption effects caused by degradation products inside the cell	Remove deposits by baking the cell for two hours at 400°C. If the symptom does not disappears, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
	Contamination of the cell	Remove contaminants by baking the cell for two hours at 400°C. If the symptom does not disappears, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
	Inadequate gas flow rate settings	Set gas flow rates to proper values.
	Leaks on the gas lines	Perform a leak test. Refer to the *Performing a Leak Check (Capillary Column)* on page 181.
Low sensitivity	Lamp window fogged by UV-absorbing deposits	Clean the lamp window. Refer to the *Clean the Lamp Window* Operating Sequence on page 323.
	Flow rate of the make-up gas is set too high	Reduce the flow rate of the make-up gas down to10 ml/min or less.
	UV lamp is not adequate to the compounds to be analyzed	Replace with an appropriate one.

16

Maintaining a Thermal Conductivity Detector

Chapter at a Glance…

Thermal Conductivity Detector (TCD)

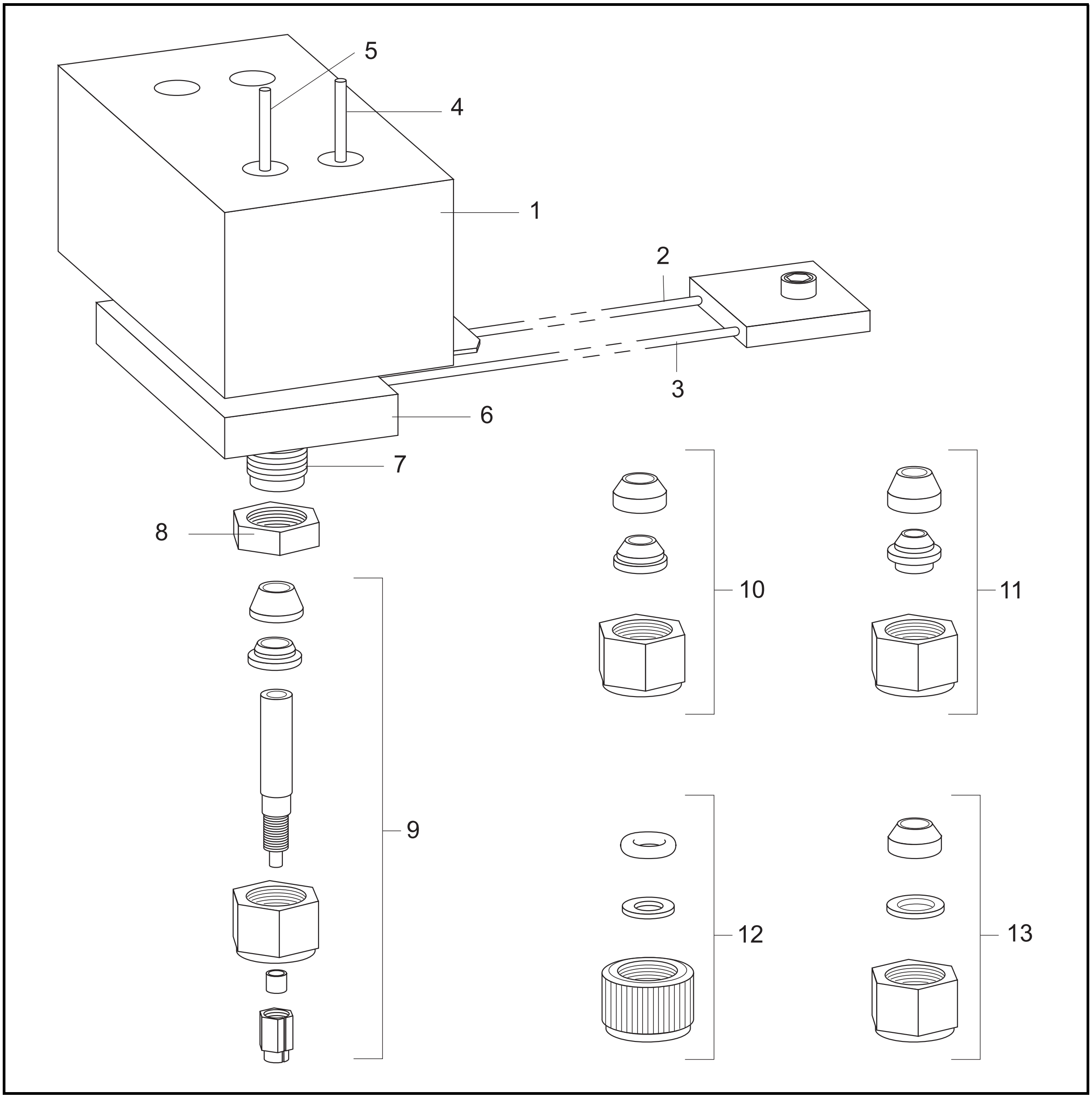


Table 16-1. Thermal Conductivity Detector (TCD) parts identification table

No.	Description	P/N
1	Detector insulated box	
2	Reference gas line	
3	Make-up gas line	
4	Reference gas exhaust	
5	Carrier + make-up gas exhaust	
6	Transfer line heater	
7	Column fitting	
8	Fixing nut	
9	Kit for connecting capillary columns	347 095 42
10	Retaining nut for 6 mm OD column and ferrule	350 201 17
11	Retaining nut for 4 mm OD column and ferrule	350 201 18
12	Knurled nut, O-ring and washer for 6 mm OD glass column	350 201 19
13	Nut, ferrule and washer for 6 mm OD glass column	350 201 22

TCD Maintenance

The TCD detector does not usually need current maintenance. Nevertheless, if you follow a few simple sequences, you will avoid troubles and prolong the detector’s lifetime. Pay a special attention to avoid contamination or damage of the filaments.

Follow these simple rules:

- Avoid to activate/deactivate the bridge when not necessary. This operation would considerably reduce the filaments lifetime.
- Avoid injecting samples that contain halogenated or acid compounds at high concentrations.
- Ensure that oxygen (air) could not enter into the filaments cells. Oxidation would irreversibly damage the filaments. Install traps for moisture and oxygen on the gas lines to reduce the hazard.

WARNING! **Set** `Filament power` **to** `Off` **before disconnecting the column from the detector. When the column is disconnected, air will enter into the cell and the filament, if powered, will burn.**

For the same reason set `Filament power` **to** `On` **only if the column has been connected. It is a good practice to let the reference and make-up gases flow through the cells for 10-15 minutes before powering the filaments.**

TCD Troubleshooting

Sensitivity

Sensitivity is related to the detector temperature (increasing temperature reduce sensitivity) and to the flow rate of the carrier, reference and make-up gas. A gain in sensitivity can be obtained increasing the gap between the temperature of the block and that of the filaments or increasing the filaments voltage.

Sensitivity is strictly related to the state of the filaments and to their operating conditions. A significant reduction of the detector sensitivity may be caused by the contamination of the filaments due to degradation of high molecular weight compounds inside the cell or to contaminated gases.

Low temperatures of the detector block may cause high boiling compounds condensate on the filaments reducing sensitivity.

Baseline drift

A small baseline drift normally occurs during a temperature program and does not indicate a trouble. This effect is due to the decrease of the carrier gas flow rate as temperature increases.

A baseline that suddenly goes out of scale and a rapid growth of the signal could indicate that filaments are likely to be burnt and have to be replaced.

Negative peaks

Negative peaks are normally generated by the sample components that have a thermal conductivity higher than carrier gas. For instance, using nitrogen or argon as carrier gas, negative peaks are obtained for helium, hydrogen or methane.

To revert the polarity of the detector, refer to the TRACE™ GC Ultra *Operating Manual* for instructions.

Troubleshooting the TCD

Table 16-2. TCD troubleshooting table

Symptom	Diagnosis	Remedy
Baseline fluctuations	Unstable regulation of the flow rate of the gases	Check the controllers of the carrier, reference and make-up gases work well.
	Leaks on the gas lines	Check the connections of the carrier, reference and make-up gas are tight.
	Inlet pressure of gases set too low	Set the pressure of the carrier, reference and make-up gas to a proper value. Refer to the TRACE™ GC Ultra *Operating Manual* for instructions.
	`Trans temp` set too high	Set `Trans temp` to a proper value (about 10 to 20°C below the `Block temp` value).
	Faulty temperature regulation	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
Baseline drift	Unstable regulation of the flow rate of the gases	Check the controllers of the carrier, reference and make-up gases work well.
	Leaks on the gas lines	Check the connections of the carrier, reference and make-up gas are tight.
	Column conditioning not correctly performed	Recondition the column according to the manufacturer’s instructions.
	Septum bleeding	Check the operating temperature of the septum is adequate. Replace the septum if necessary.

Table 16-2. TCD troubleshooting table (Continued)

Symptom	Diagnosis	Remedy
Low sensitivity	Leaks on the gas lines	Check the connections of the carrier, reference and make-up gas are tight.
	Leak due to septum wearing	Replace the septum.
	Operating conditions of the detector not properly set	Optimize working parameters of the detector according to the actual operating mode (Constant Voltage, Constant Temperature). Refer to the TRACE GC Ultra *Operating Manual* for instructions.
	Thermal conductivity of the carrier gas is too close to the one of the compound to be analyzed	Use a different carrier gas. Refer to the TRACE GC Ultra *Operating Manual* for instructions.
	Contaminated filaments	Remove contamination by baking the filaments for one hour at a temperature higher than the boiling point of the most high-boiling compound. Perform baking twice if necessary. If the symptom does not disappears, contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

Table 16-2. TCD troubleshooting table (Continued)

Symptom	Diagnosis	Remedy
Baseline drift	Faulty temperature control	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
Low sensitivity	Oxidized filaments	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.
The detector does not work. **Message:** `Filament power Off`	Lack of carrier, make-up or reference gas or pressure too low	Check the feed of the carrier, make-up and reference gas.
	Filament burnt	Contact your customer support organization. Refer to Appendix B, *Customer Communication*, for contact information.

17

Maintaining Detectors in Stack Configuration

Chapter at a Glance...

Operating Sequences

ECD adapted for Stack Configuration

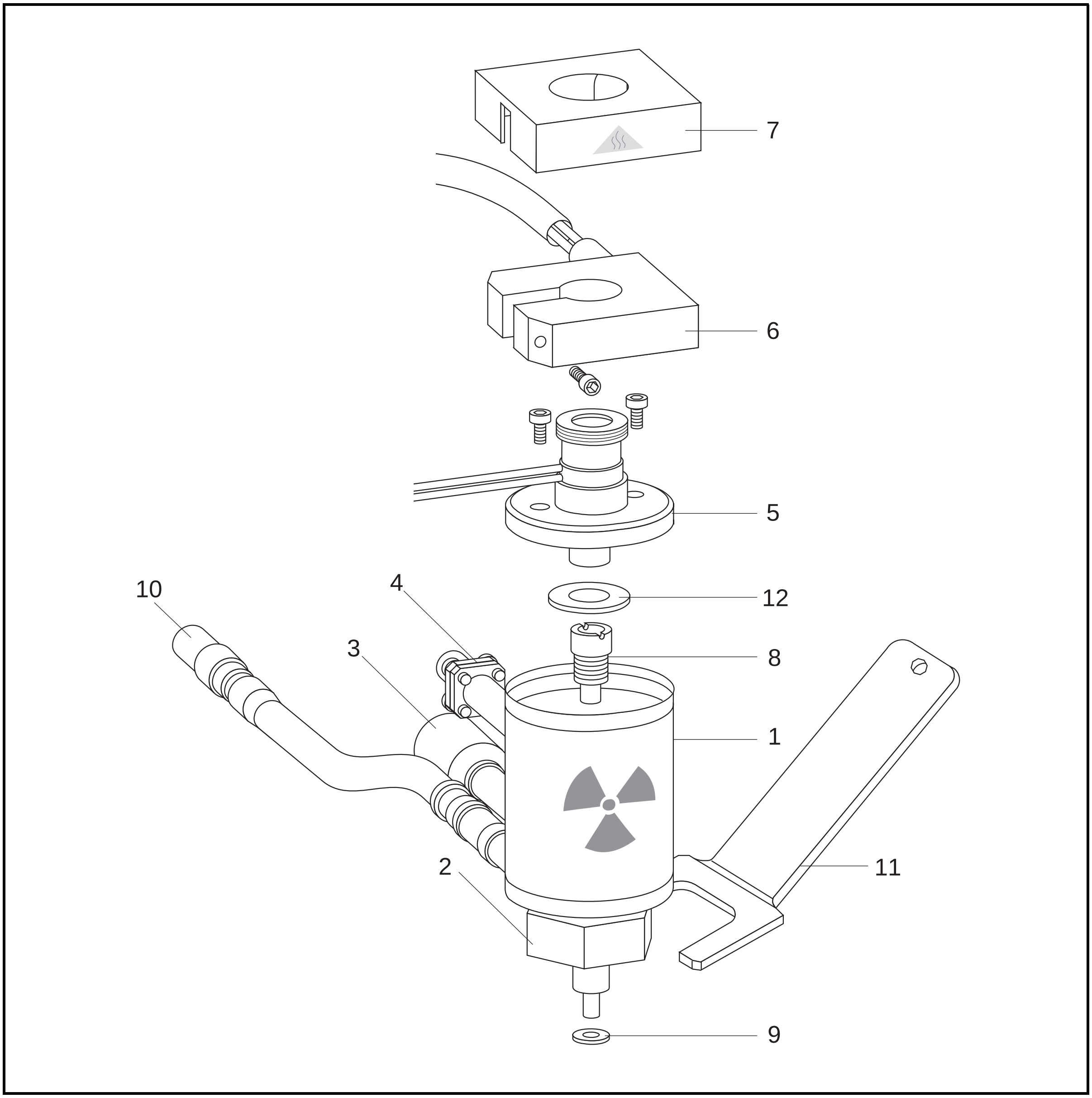


Table 17-1. Electron Capture Detector (ECD) parts identification table (Stack configuration version)

No.	Description	P/N
1	Detector cell	
2	Fixing nut	
3	Excitation connector	
4	Signal connector	
5	Detector adapter	347 090 07
6	Heating block	354 250 64
7	Heat shield	241 044 49
8	Collecting electrode	277 003 50
9	Silver seal	290 150 58
10	Temperature sensor/heater assembly	190 500 87
11	ECD fixing tool	205 021 50
12	Seal	290 326 08

Guidelines for Maintenance

A FID, NPD or FPD detector can be installed on the top of an ECD with the aid of a specially designed adapter (see *ECD adapted for Stack Configuration*, page 346) to operate as an auxiliary detector.

Maintenance of the auxiliary detector will be performed as it would be mounted directly on the gas chromatograph. You will find instructions in the relevant chapters of this manual.

In this chapter you will find instructions on how to perform ECD maintenance when installed in stack configuration respectively with a FID, NPD and FPD detector.

To properly maintain the ECD, you should perform the following cleaning or replacement sequences:

- cleaning the collecting electrode
- replacing the collecting electrode
- replacing the silver seal

WARNING! The Electron Capture Detector (ECD) contains a ^{63}Ni beta-emitting radioactive source of 370 MBq (10 mCi). Read with care the paragraph *ECD Maintenance* on page 222 and follow the instructions reported to avoid radioactive contamination.

OPERATING SEQUENCE

Clean or Replace the Collecting Electrode (FID on ECD)

Materials needed

- Screwdriver
- 2.5 mm Allen wrench
- 3 mm Allen wrench
- Tweezers

for replacement:

- Collecting electrode (P/N 277 003 50)

for cleaning:

- Ultrasonic cleaning bath
- GC-grade hexane
- GC-grade toluene
- Fine emery paper (optional)

WARNING! **Any maintenance or repair operations involving the radioactive source of the ECD must be performed ONLY by qualified personnel duly authorized to handle radioactive material. Please contact your local Thermo Fisher Scientific Technical Service office.**

WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.**

PRECAUTIONS

1. Press **AUX**.
2. Scroll to `Detector` and press **ENTER**.

```
              AUXILIARY
Signal                                <
Detector
Temperature Zones
Pressure Channel
```

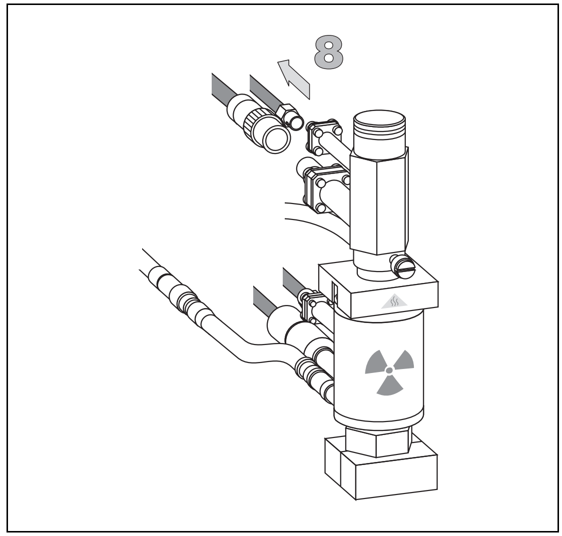
3. In the `AUX DETECTOR (FID)` table, scroll to `Flame` and press **OFF**.
4. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

```
        AUX DETECTOR (FID)
Flame                          Off <
Base temp              300     Off
Signal pA                    (5.4)
Ign. tresh                     2.0
Flameout retry                 Off
H2                      35     Off
Air                    350     Off
```

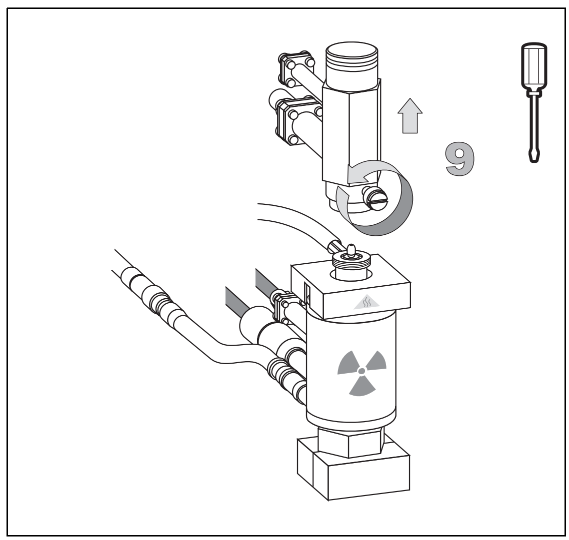
5. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table.
6. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
7. Scroll to `Mkup(N2)` and press **OFF**.

```
        XXX DETECTOR (ECD)
Base temp              250     Off <
ECD temp               250     Off
Ref current nA                 1.0
Freq kHz                    (2.15)
Pulse amp V                     25
Pulse width us                 1.0
Mkup (N2)               40     Off
```

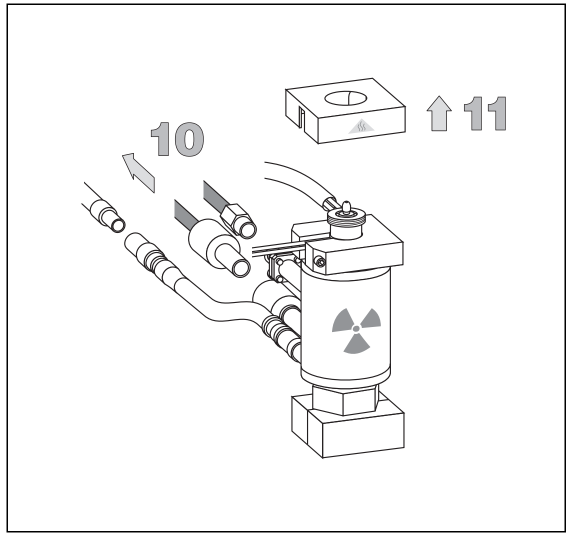
8. Wait until the detectors temperature is near to ambient, then disconnect the signal and ignition polarization cables from the FID.



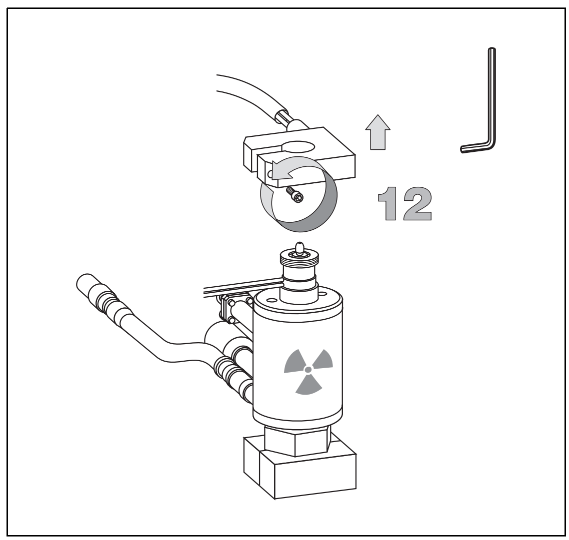
9. Loosen the fixing screw on the front of the FID cell and remove it.



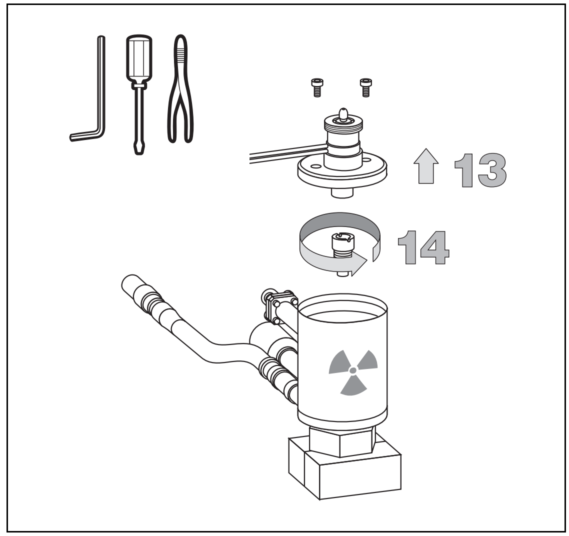
10. Disconnect the signal, excitation and heater cables from the ECD detector
11. Remove the heat shield.



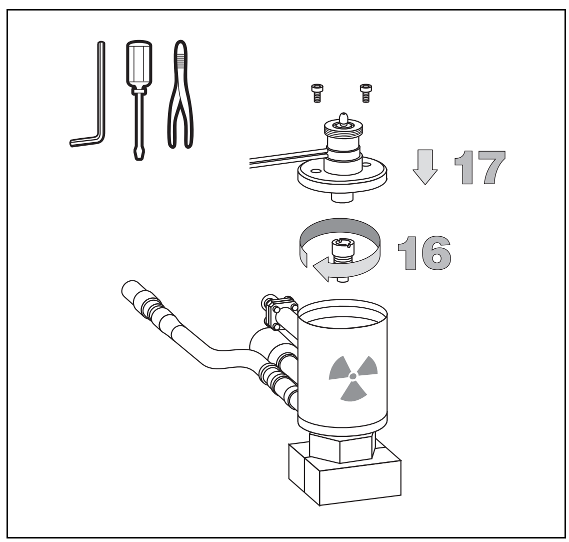
12. Remove the heater from the top of the ECD detector unscrewing and removing the 4 mm Allen fixing screw. Use a 3 mm Allen wrench.



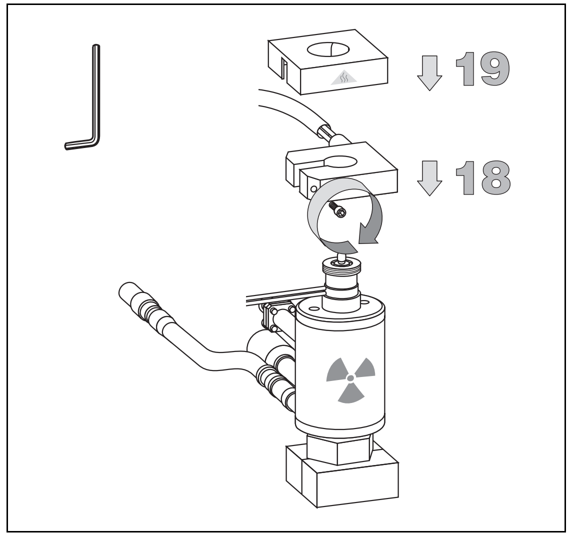
13. Remove the detector adapter unscrewing the two 3 mm Allen screws. Use a 2.5 mm Allen wrench.
14. Unscrew the collecting electrode and remove it using tweezers.



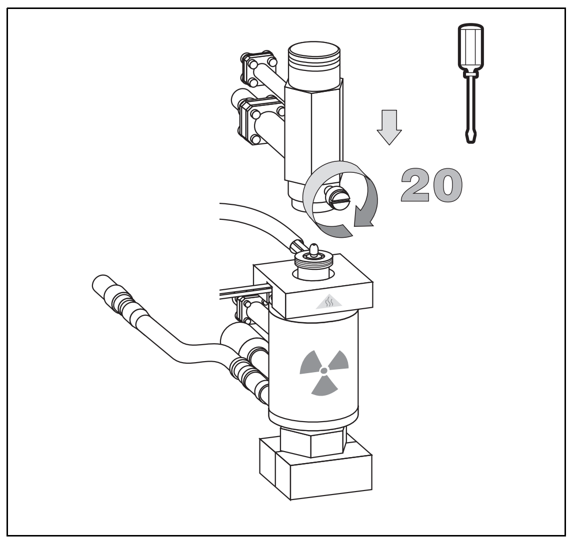
15. If you are going to replace the collecting electrode with a new one, go to step 16. If you are going to clean it, put it into an ultrasonic bath filled with a toluene/hexane mixture and sonicate it. In the most critical cases, fine emery paper can be used.
16. Mount the new (or cleaned) electrode without touching it. Use tweezers.
17. Remount the detector adapter and tighten the Allen screws.



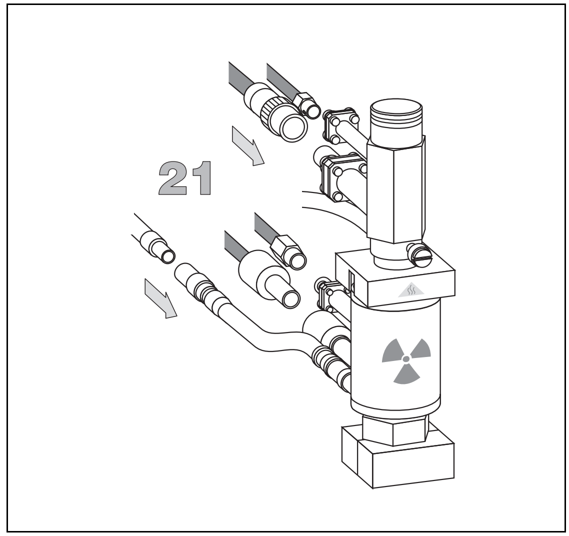
18. Reinsert the heater on the top of the ECD detector and fix it with its Allen screw.
19. Slip the heat shield onto the heater



20. Remount the FID detector on the top of the ECD and tighten the fixing screw.



21. Reconnect the signal and ignition cables to the FID and the signal, excitation and heater cables to the ECD detector.



22. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and reset the ECD to the required operating conditions.
23. Press **AUX**.
24. Scroll to `Detector` and press **ENTER**.
25. In the `AUX DETECTOR (FID)` table, reset the auxiliary FID detector to the required operating conditions.

OPERATING SEQUENCE

Clean or Replace the Collecting Electrode (NPD on ECD)

Materials needed

- Screwdriver
- 2.5 mm Allen wrench
- 3 mm Allen wrench
- Tweezers

for replacement:

- Collecting electrode (P/N 277 003 50)

for cleaning:

- Ultrasonic cleaning bath
- GC-grade hexane
- GC-grade toluene
- Fine emery paper (optional)

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WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.**

PRECAUTIONS

1. Press **AUX**.
2. Scroll to `Detector` and press **ENTER**.

```
              AUXILIARY
Signal                              <
Detector
Temperature Zones
Pressure Channel
```

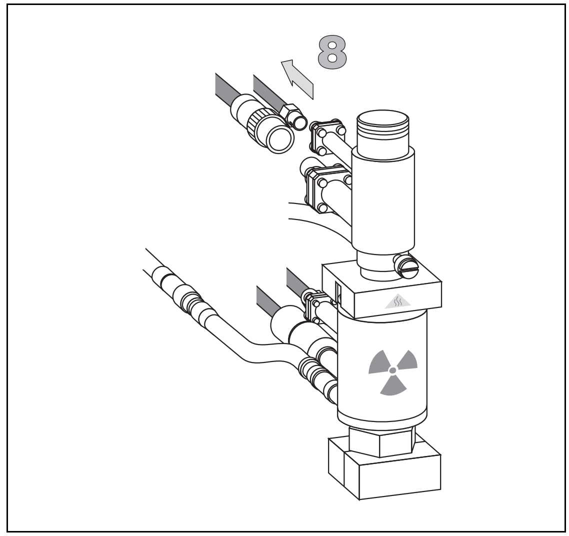
3. In the `AUX DETECTOR (NPD)` table, scroll to `Base temp` and press **OFF**.
4. Scroll subsequently to `Source curr`, `H2`, `Air` and press **OFF**.

```
       AUX DETECTOR (NPD)
Source cur,A                  2.740 <
Base temp             300       Off
Signal pA                    (10.2)
Polarizer V                     3.5
H2 delay time                   Off
H2                      2         2
Air                    60        60
```

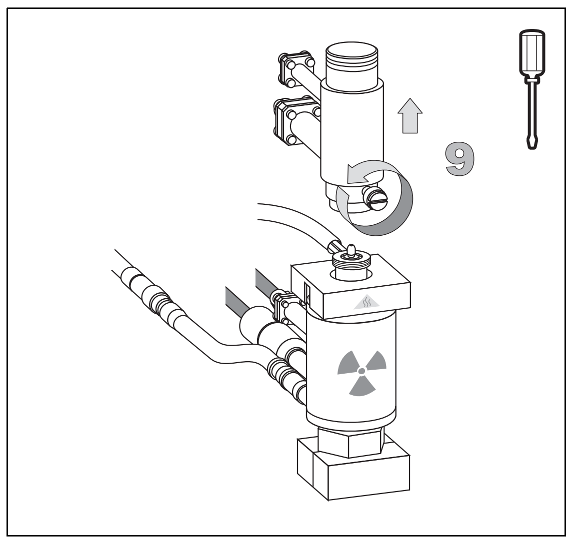
5. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table.
6. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
7. Scroll to `Mkup(N2)` and press **OFF**.

```
       XXX DETECTOR (ECD)
Base temp             250       Off <
ECD temp              250       Off
Ref current nA                  1.0
Freq kHz                     (2.15)
Pulse amp V                      25
Pulse width us                  1.0
Mkup (N2)              40       Off
```

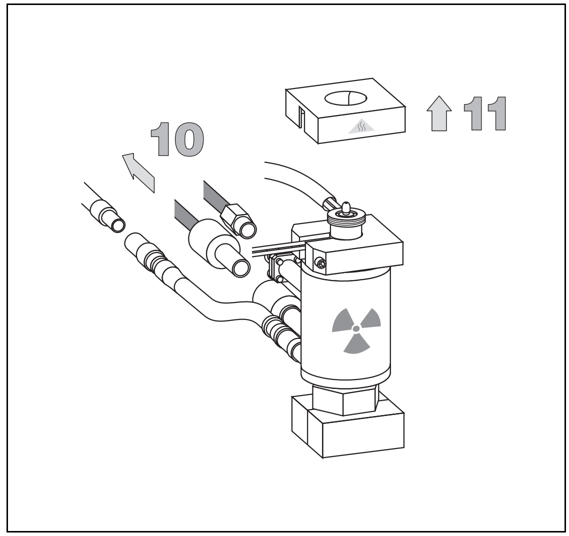
8. Wait until the detectors temperature is near to ambient, then disconnect the signal and ignition polarization cables from the NPD.



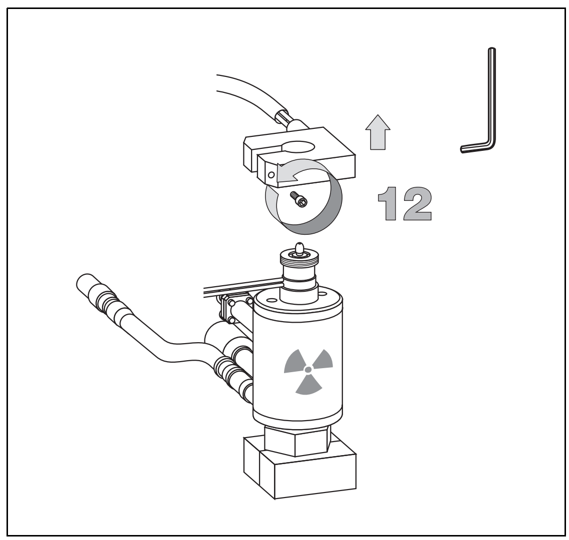
9. Loosen the fixing screw on the front of the NPD cell and remove it.



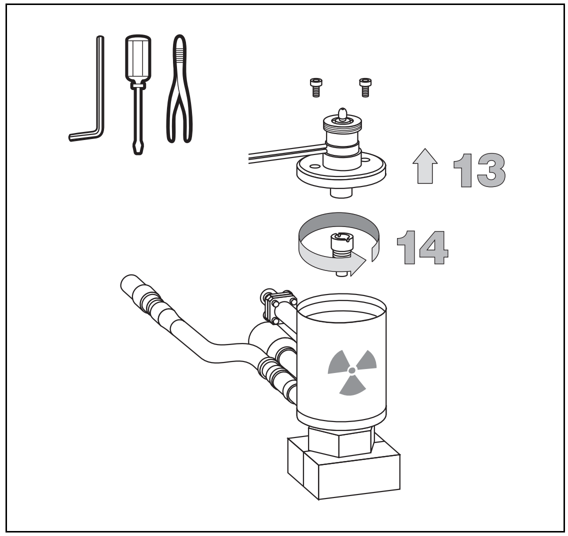
10. Disconnect the signal, excitation and heater cables from the ECD detector
11. Remove the heat shield.



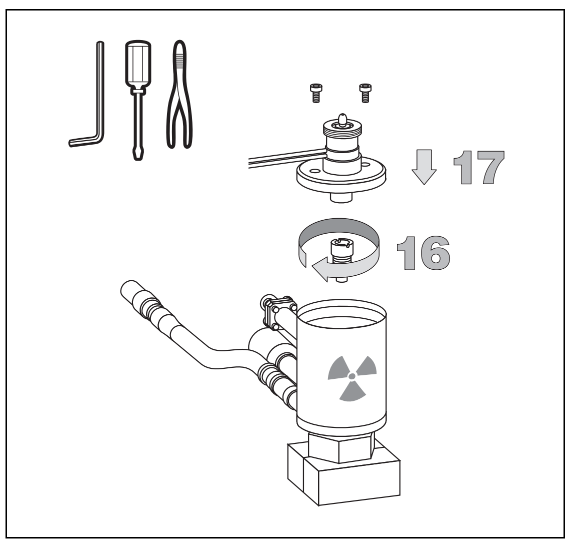
12. Remove the heater from the top of the ECD detector loosening the Allen fixing screw. Use a 3 mm Allen wrench.



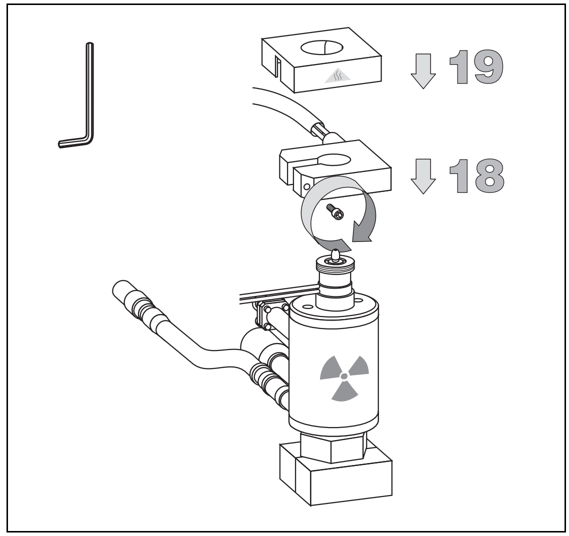
13. Remove the detector adapter unscrewing the two retaining Allen screws. Use a 2.5 mm Allen wrench.
14. Unscrew the collecting electrode and remove it using tweezers.



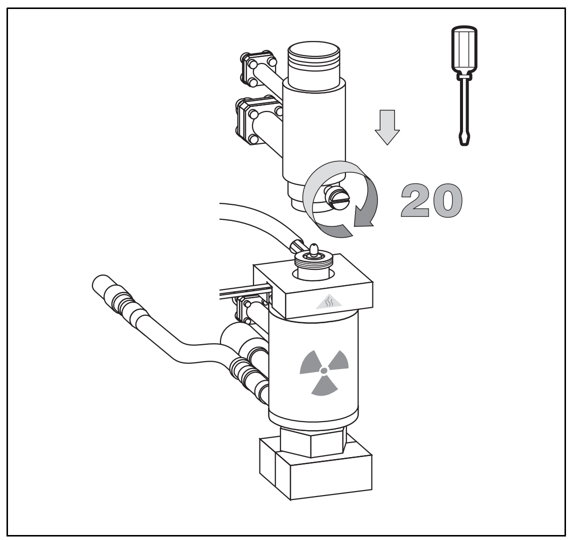
15. If you are going to replace the collecting electrode with a new one, go to step 16. If you are going to clean it, put it into an ultrasonic bath filled with a toluene/hexane mixture and sonicate it. In the most critical cases, fine emery paper can be used.
16. Mount the new (or cleaned) electrode without touching it. Use tweezers.
17. Remount the detector adapter and tighten the Allen screws.



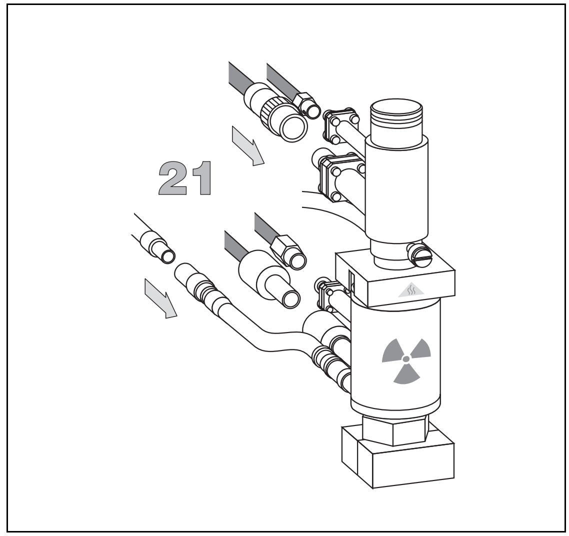
18. Reinsert the heater on the top of the ECD detector and fix it with its Allen screw.
19. Slip the heat shield onto the heater



20. Remount the NPD detector on the top of the ECD and tighten the fixing screw.



21. Reconnect the signal and ignition cables to the NPD and the signal, excitation and heater cables to the ECD detector.



22. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and reset the ECD to the required operating conditions.
23. Press **AUX**.
24. Scroll to `Detector` and press **ENTER**.
25. In the `AUX DETECTOR (NPD)` table, reset the auxiliary NPD detector to the required operating conditions.

OPERATING SEQUENCE

Clean or Replace the Collecting Electrode (FPD on ECD)

Materials needed

- Screwdriver
- 2.5 mm Allen wrench
- 3 mm Allen wrench
- 5 mm wrench
- Tweezers
- FPD removing tool (P/N 205 021 50)

for replacement:

- Collecting electrode (P/N 277 003 50)

for cleaning:

- Ultrasonic cleaning bath
- GC-grade hexane
- GC-grade toluene
- Fine emery paper (optional)

WARNING! Any maintenance or repair operations involving the radioactive source of the ECD must be performed ONLY by qualified personnel duly authorized to handle radioactive material. Please contact your local Thermo Fisher Scientific Technical Service office.

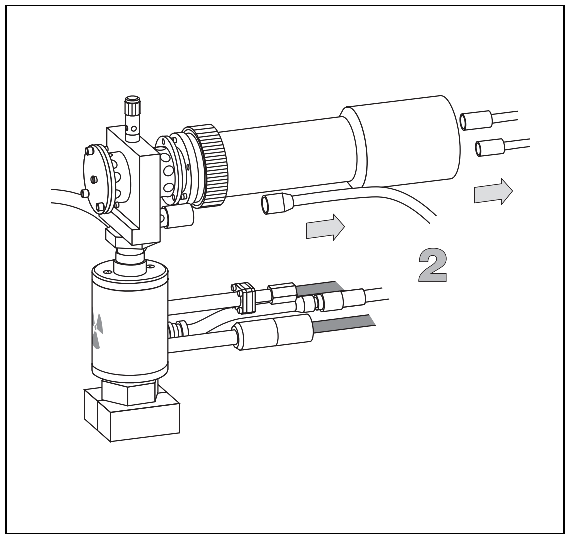
WARNING! This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.

PRECAUTIONS

WARNING! **Before disconnecting the cables or disassembling an FPD detector, switching off the GC power supply is imperative to avoid electrical shocks and damages to the instrument.**

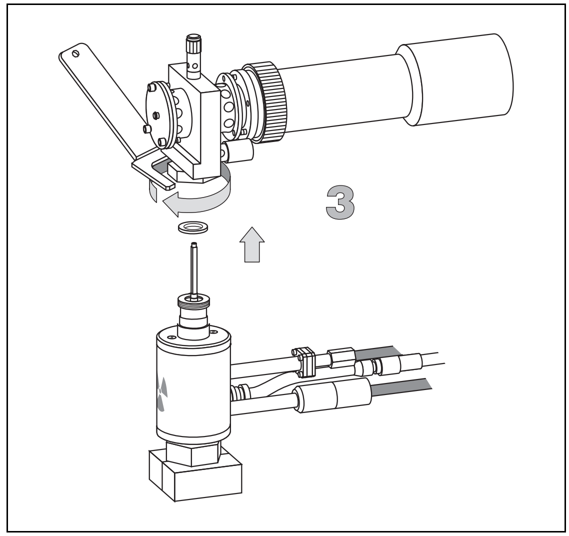
WARNING! **When handling organics solvents you must take precautions to avoid health hazards. Refer to Appendix A, *Reagents Safety Information* for more information.**

1. Switch off the GC power supply and wait until the detectors have cooled to room temperature.
2. Disconnect the signal, excitation voltage and ignition/heating cables from the FPD detector.

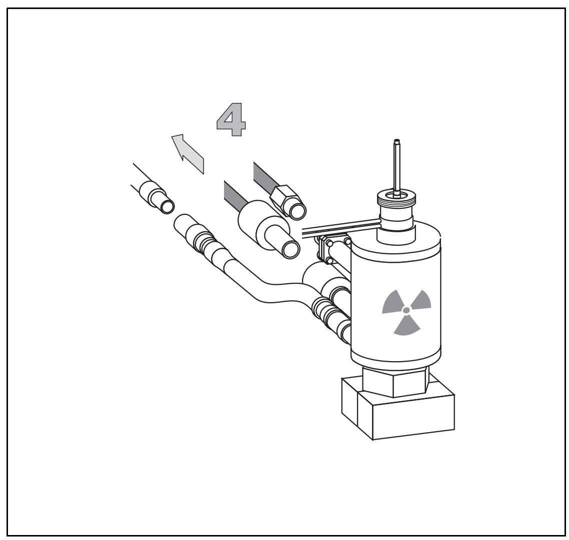


3. Using the tool provided with the GC, loosen the fixing nut on the base of the FPD detector and remove it.

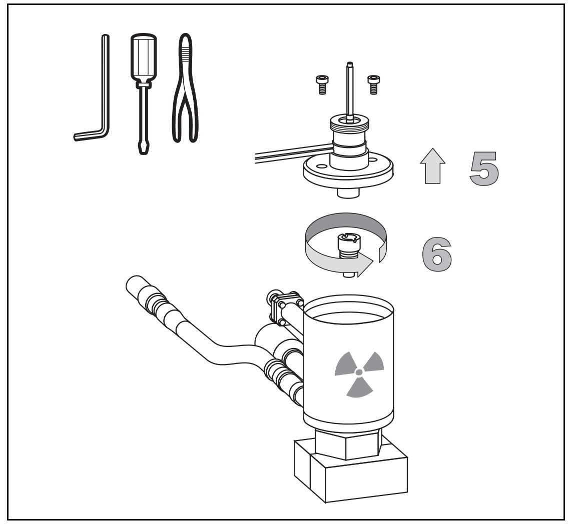
CAUTION **Pay attention not to lose the aluminium ring inserted between the detector head and the base body.**



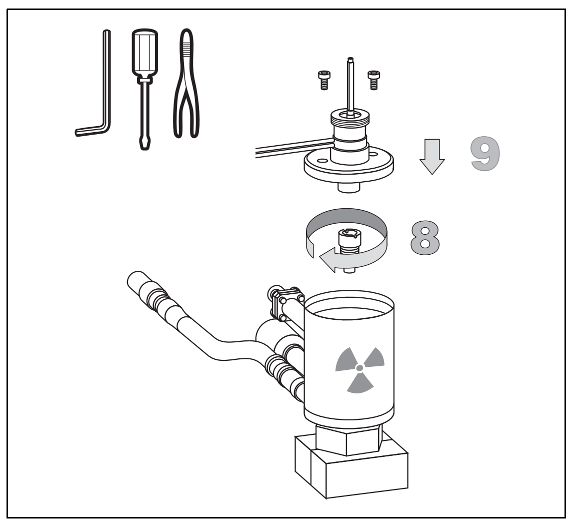
4. Disconnect the signal, excitation and heater cables from the ECD detector.



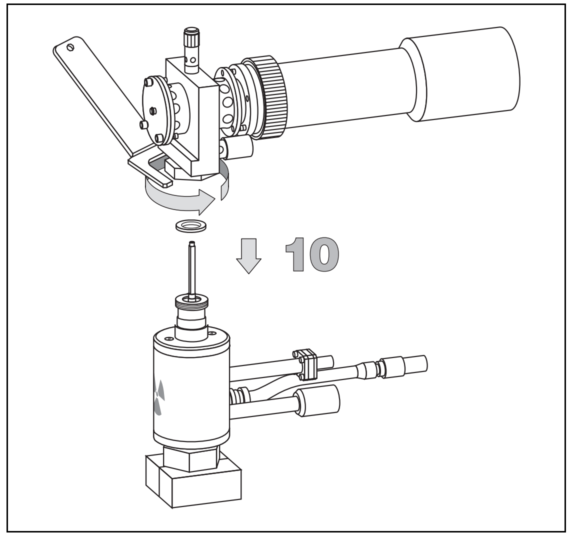
5. Remove the detector adapter unscrewing the two retaining Allen screws. Use a 2.5 mm Allen wrench.
6. Unscrew the collecting electrode and remove it using tweezers.
7. If you are going to replace the collecting electrode with a new one, go to step 8. If you are going to clean it, put it into an ultrasonic bath filled with a toluene/hexane mixture and sonicate it. In the most critical cases, fine emery paper can be used.



8. Mount the new (or cleaned) electrode without touching it. Use tweezers.
9. Remount the detector adapter and tighten the Allen screws.

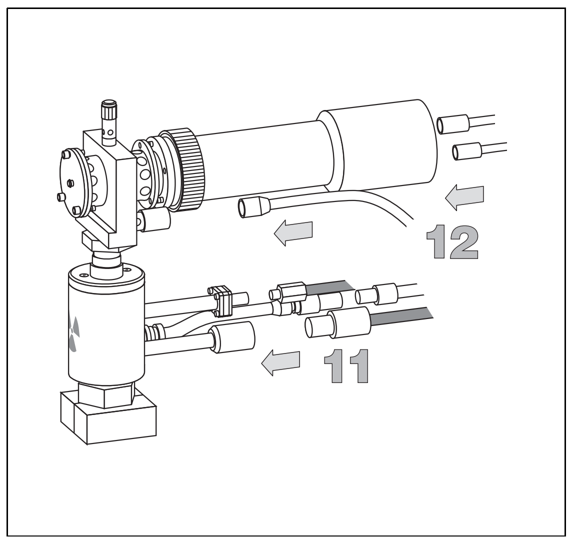


10. Place the FPD detector on the ECD top cover, paying attention that the aluminium ring has been inserted in the correct position, then tighten the fixing nut.



11. Reconnect the excitation and heater cables to the ECD detector.

12. Reconnect the signal, excitation voltage and ignition/heating cables to the FPD.



13. Switch on the GC power supply.

14. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and set the ECD to the required operating conditions.
15. Press **AUX**.
16. Scroll to `Detector` and press **ENTER**.
17. In the `AUX DETECTORS` table, set the FPD detector to the required operating conditions.

OPERATING SEQUENCE

Replace the Silver Seal (FID on ECD)

Materials needed

- Silver seal (P/N 290 150 58)
- ECD fixing tool (P/N 205 021 50)
- Tweezers

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WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.**

PRECAUTIONS

1. Press **AUX**.
2. Scroll to `Detector` and press **ENTER**.

```
           AUXILIARY
Signal                          <
Detector
Temperature Zones
Pressure Channel
```

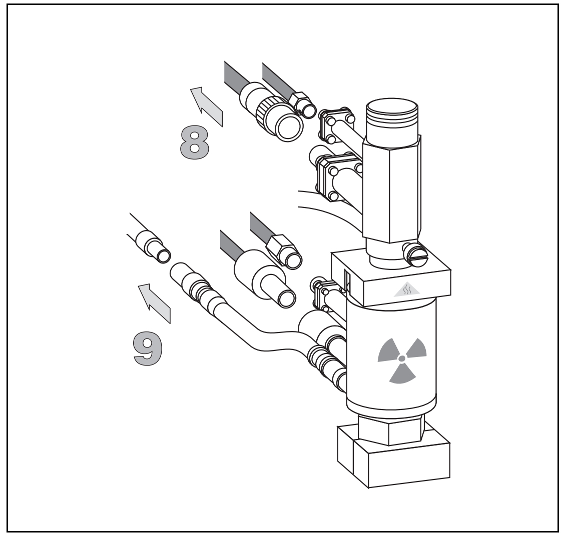
3. In the `AUX DETECTOR (FID)` table, scroll to `Flame` and press **OFF**.
4. Scroll subsequently to `Base temp`, `H2` and `Air` and press **OFF**.

AUX DETECTOR (FID)		
Flame		Off <
Base temp	300	Off
Signal pA		(5.4)
Ign. tresh		2.0
Flameout retry		Off
H2	35	Off
Air	350	Off

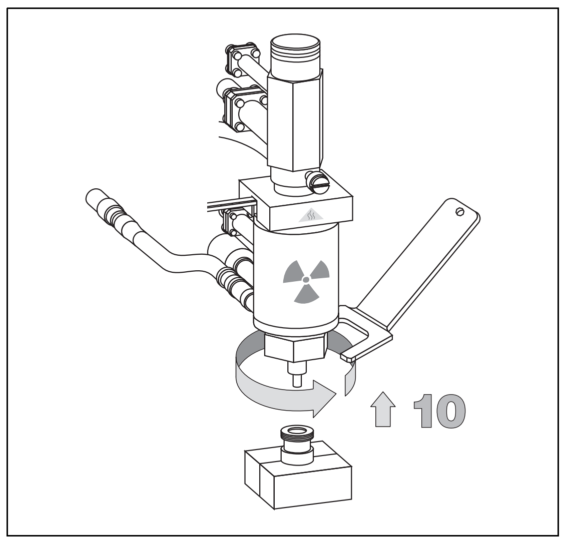
5. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table.
6. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
7. Scroll to `Mkup(N2)` and press **OFF**.

XXX DETECTOR (ECD)		
Base temp	250	Off <
ECD temp	250	Off
Ref current nA		1.0
Freq kHz		(2.15)
Pulse amp V		25
Pulse width us		1.0
Mkup (N2)	40	Off

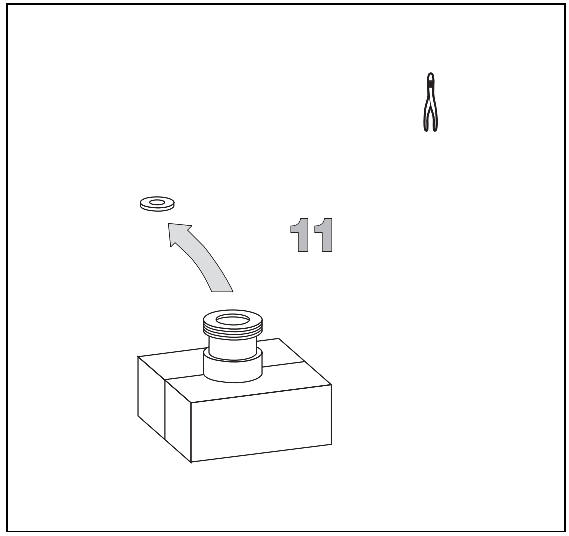
8. Disconnect the signal and ignition polarization cables from the FID.
9. Disconnect the signal, excitation and heater cables from the ECD detector



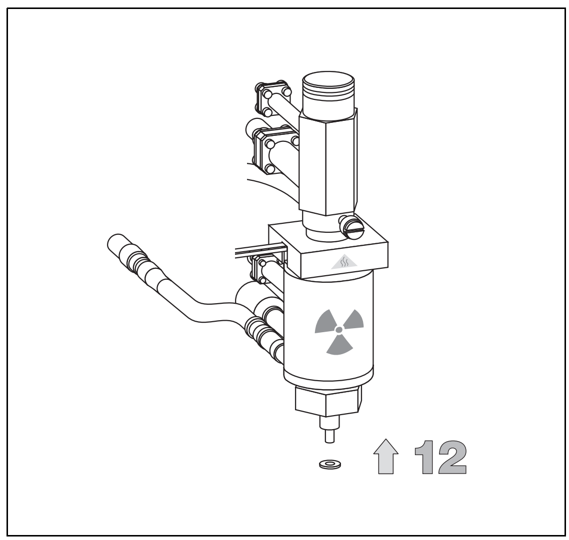
10. Loosen the nut that fixes the ECD detector to its base body with the proper tool contained in the standard outfit and remove the detectors from the ECD base body.



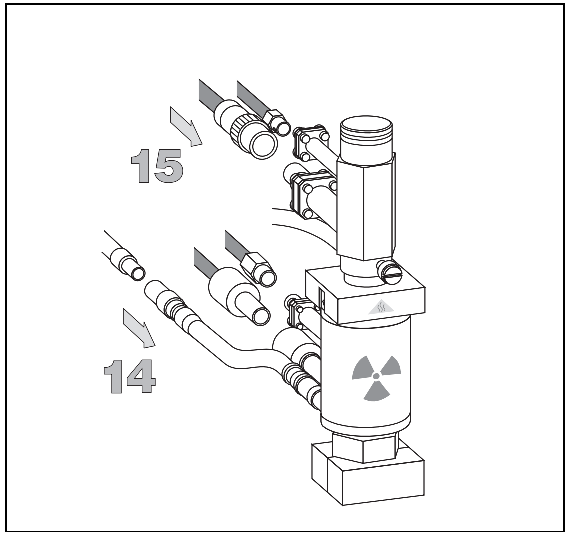
11. Using tweezers, remove the worn silver seal from its housing in the base body.



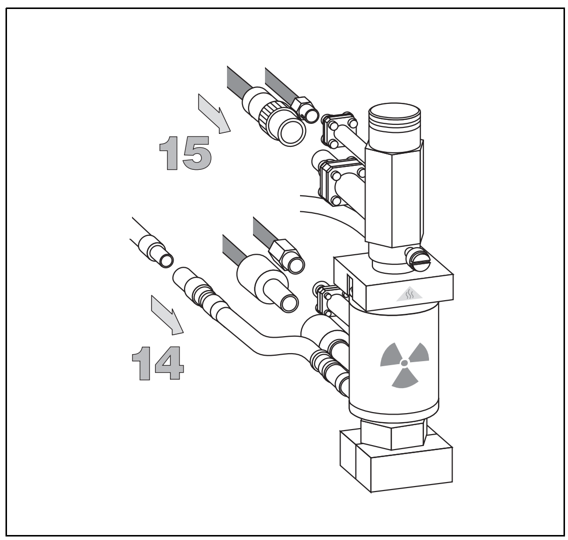
12. Insert a new silver seal on the lower part of the ECD detector.



13. Remount the detector on its base body, tightening the fixing nut with the proper tool.



14. Reconnect the signal, excitation and heater cables to the ECD detector.
15. Reconnect the signal and ignition polarization cables to the FID



16. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and reset the ECD to the required operating conditions.
17. Press **AUX**.
18. Scroll to `Detector` and press **ENTER**.
19. In the `AUX DETECTOR (FID)` table, set the FID detector to the required operating conditions.

OPERATING SEQUENCE

Replace the Silver Seal (NPD on ECD)

Materials needed

- Silver seal (P/N 290 150 58)
- ECD fixing tool (P/N 205 021 50)
- Tweezers

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WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.**

PRECAUTIONS

1. Press **AUX**.
2. Scroll to `Detector` and press **ENTER**.

```
             AUXILIARY
Signal                            <
Detector
Temperature Zones
Pressure Channel
```

3. In the `AUX DETECTOR (NPD)` table, scroll to `Base temp` and press **OFF**.
4. Scroll subsequently to `Source curr`, `H2`, `Air` and press **OFF**.

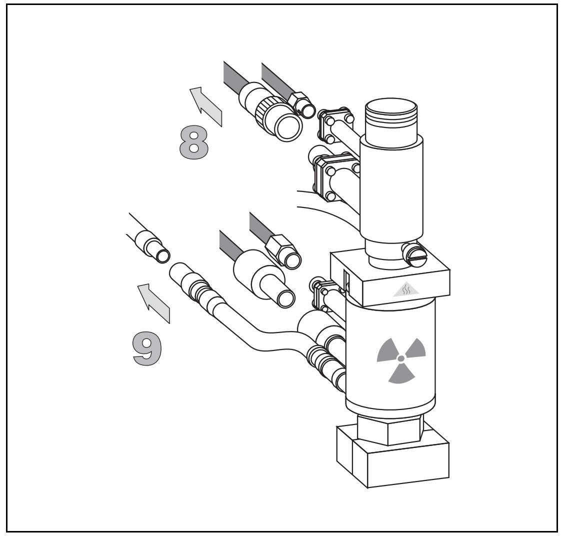
AUX DETECTOR (NPD)			
Source cur,A		2.740	<
Base temp	300	Off	
Signal pA		(10.2)	
Polarizer V		3.5	
H2 delay time		Off	
H2	2	Off	
Air	60	Off	

5. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, to access the ECD control table.
6. Scroll subsequently to `Base temp` and `ECD temp` and press **OFF**.
7. Scroll to `Mkup (N2)` and press **OFF**.

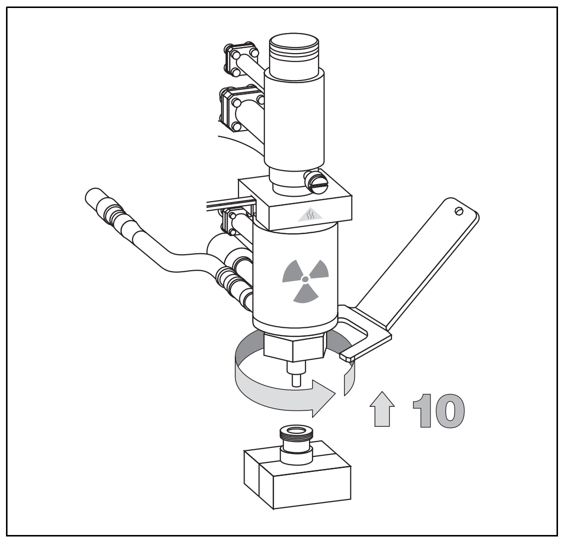
XXX DETECTOR (ECD)			
Base temp	250	Off	<
ECD temp	250	Off	
Ref current nA		1.0	
Freq kHz		(2.15)	
Pulse amp V		25	
Pulse width us		1.0	
Mkup (N2)	40	Off	

8. Disconnect the signal and ignition polarization cables from the NPD.

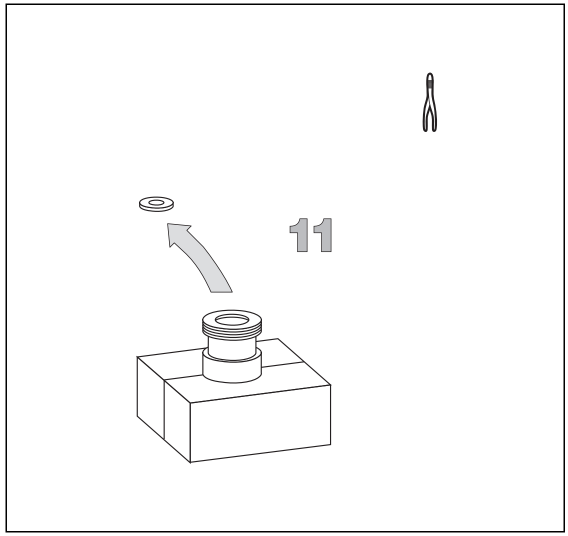
9. Disconnect the signal, excitation and heater cables from the ECD detector



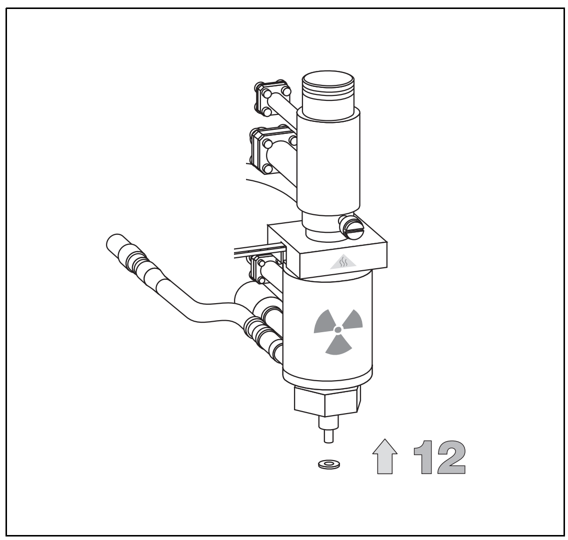
10. Loosen the nut that fixes the ECD detector to its base body with the proper tool contained in the standard outfit and remove the detectors from the ECD base body.



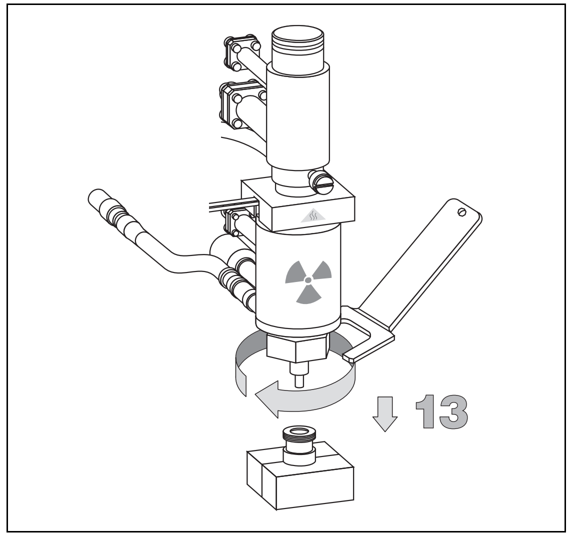
11. Using tweezers, remove the worn silver seal from its housing in the base body.



12. Insert a new silver seal on the lower part of the ECD detector.

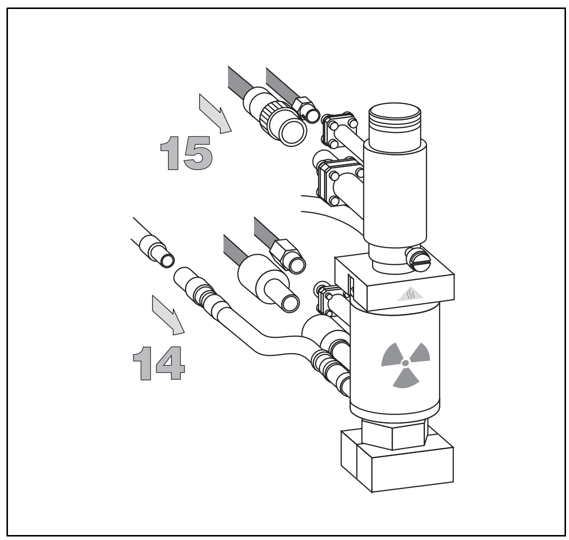


13. Remount the detector on its base body, tightening the fixing nut with the proper tool.



14. Reconnect the signal, excitation and heater cables to the ECD detector.

15. Reconnect the signal and ignition polarization cables to the NPD



16. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and reset the ECD to the required operating conditions.
17. Press **AUX**.
18. Scroll to `Detector` and press **ENTER**.
19. In the `AUX DETECTOR (NPD)` table, set the NPD detector to the required operating conditions.

OPERATING SEQUENCE

Replace the Silver Seal (FPD on ECD)

Materials needed

- Silver seal (P/N 290 150 58)
- ECD fixing tool (P/N 205 021 50)
- Tweezers

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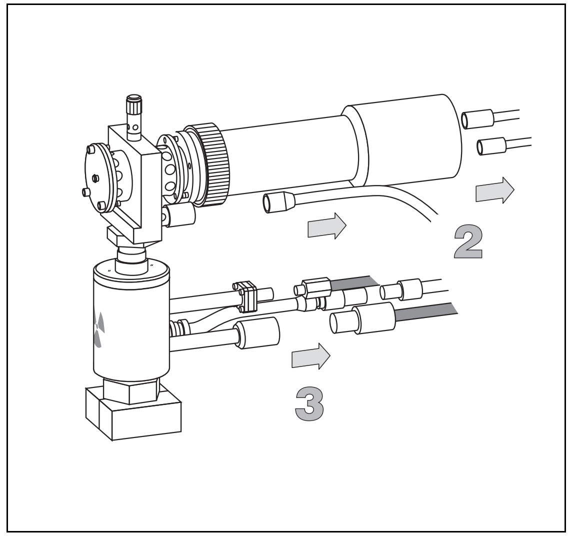
WARNING! **This operation must be carried out at low temperature to avoid burns. Therefore, before beginning the sequence, the detectors must be cooled to room temperature.**

PRECAUTIONS

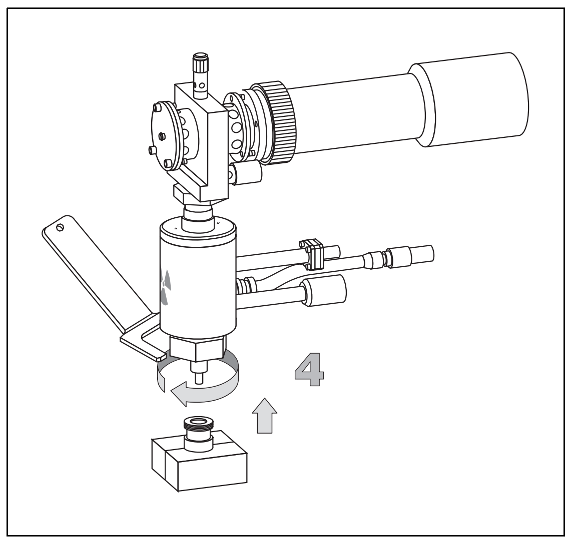
WARNING! **Before disconnecting the cables or disassembling an FPD detector, switching off the GC power supply is imperative to avoid electrical shocks and damages to the instrument.**

1. Switch off the GC power supply and wait until the detectors have cooled to room temperature.

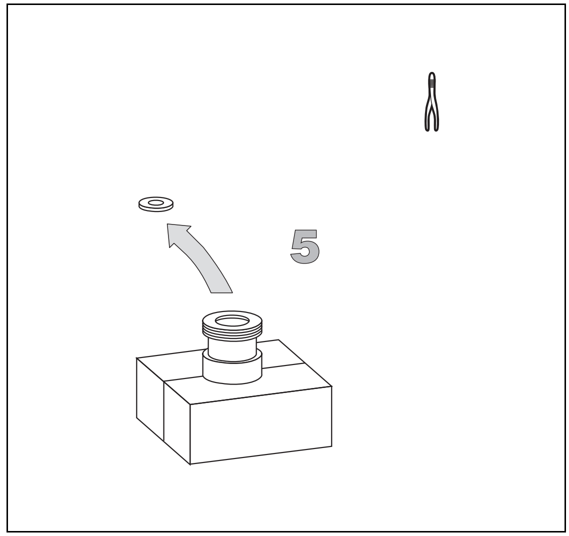
2. Disconnect the signal, excitation voltage and ignition/heating cables from the FPD detector.
3. Disconnect the signal, excitation and heater cables from the ECD detector.



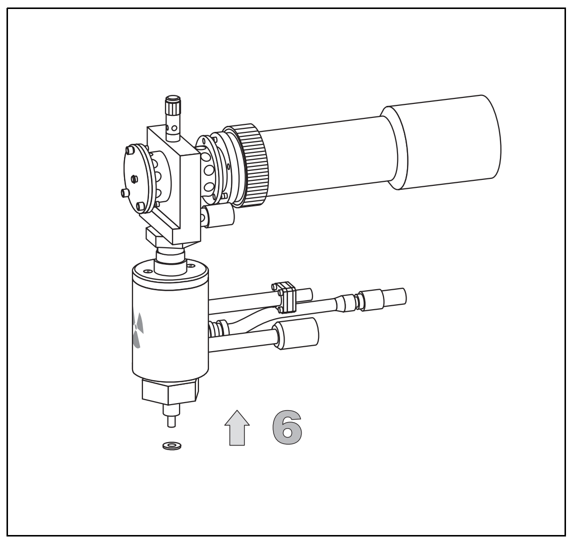
4. Loosen the nut that fixes the ECD detector to its base body with the proper tool contained in the standard outfit and remove the detectors from the ECD base body.



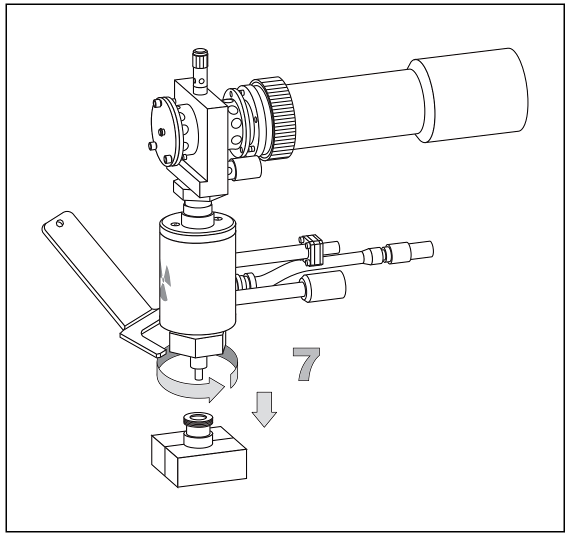
5. Using tweezers, remove the worn silver seal from its housing in the base body.



6. Insert a new silver seal on the lower part of the ECD detector.

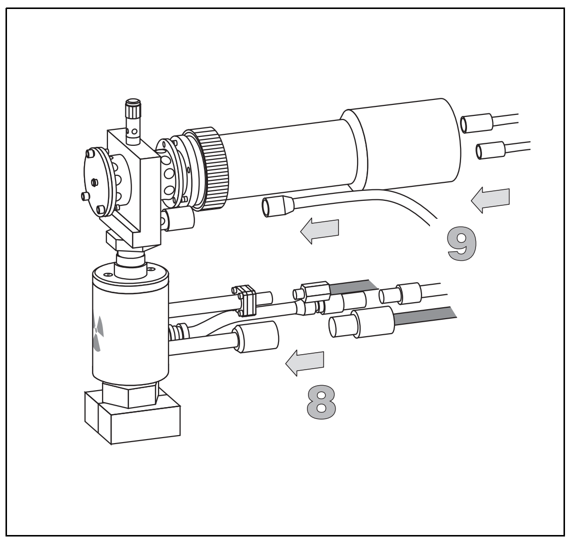


7. Remount the detectors on the ECD base body, tightening the fixing nut with the proper tool.



8. Reconnect the signal, excitation and heater cables to the ECD detector.

9. Reconnect the signal and ignition polarization cables to the FPD.



10. Switch on the GC power supply.

11. Press **LEFT DETECTOR** or **RIGHT DETECTOR**, depending on the location of the ECD, and set the ECD to the required operating conditions.
12. Press **AUX**.
13. Scroll to `Detector` and press **ENTER**.
14. In the `AUX DETECTORS` table, set the FPD detector to the required operating conditions.

Troubleshooting Guidelines

Troubleshooting detectors in stack configuration does not differ from what you would do if they would be installed separately. So, in case of troubles, you will find the necessary information in the relevant chapters of this manual.

Incorrect installation of the column

In case the ECD features an irregular response or no response at all, while the auxiliary detector auxiliary gives a normal response, this can be due to incorrect installation of the column inside the ECD.

To correct the symptom, reinstall the column and check it for leaks.To ensure a right insertion of the column inside the detector base body, the distance between the ferrule and the end of the column must be 108-110 mm.

CAUTION **When reinstalling the column, do not overtighten the connection to avoid damaging the ferrule and the column.**

18

Regenerating Carrier and Split Lines Filters

This chapter provides instruction to regenerate the active carbon filters mounting on the carrier gas line and on the split line when S/SL and PTV injectors are present.

Operating Sequences

OPERATING SEQUENCE

To Regenerate Carrier Gas and Split Lines Filters

The carrier gas and split lines active carbon filters must be regenerated annually or when ghost peaks are detected during blank analysis.

ATTENTION This operation requires the use of an external nitrogen line and an external oven to keep the filter at the regeneration temperature.
If an external oven is not present in your lab, the column oven of the GC should be use.

Before starting, the following preliminary operations must be carried out:

- Cool the oven and detector base body to room temperature.
- Close the gas supplies.
- Turn off the main power on the rear panel of the GC.
- Disconnect the main power cable from the rear of the GC.

Remove The GC Top Cover

1. Lift the detector cover off the GC top cover.
2. Open the oven door and unscrew the two top cover fastening screws.
3. Push the cover back about 1 cm and lift it up and off the GC.

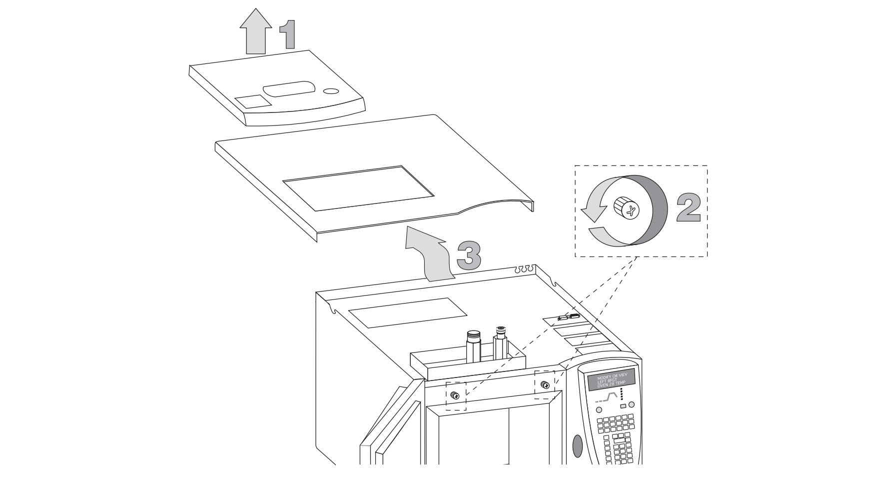
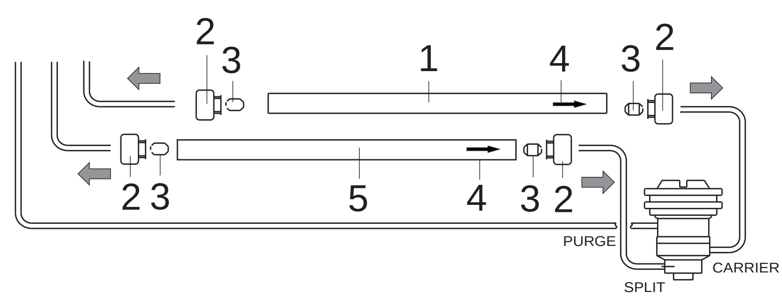


Figure 1-1. TRACE GC Ultra Top Cover

Removing Active Carbon Filter

1. Use the appropriate wrench the loosen each retaining nut.



2.1. Carrier Gas Line Filter

3.2. M6x0.75 mm Retaining Nut

4.3. 1.6 mm OD Aluminium Ferrule

5.4. Arrow indicating the Flow Direction

6.5. Split Line Filter

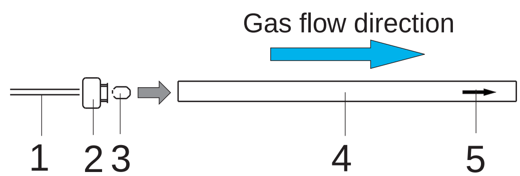
7.

8. Finger-loosen the retaining nuts of the filter to regenerate.

9. Remove the filter from its line.

Regenerating the Active Carbon Filter

10. Connect the inlet of the filter to the external nitrogen line by using the proper nut and ferrule. Make sure that the arrow printed on the filter indicates the outlet direction of the gas as shown in the next figure.



11.1. External Nitrogen Line	12.2. M6x0.75 mm Retaining Nut
13.3. 1.6 mm OD Aluminium Ferrule	14.4. Active Carbon Filter
15.5. Arrow indicating the Flow Direction	16.

17. Set a supply gas pressure of 0.5 bar to obtain a flow of at least 100 mL/min.

18. Place the filter into an external oven (or into the column oven of the GC). Keep the filter at a temperature of 300 °C for two hours.

19. At the end of the regeneration, cool the oven (or column oven of the GC) and remove the filter.

20. Disconnect external nitrogen line from the filter and remount it on the GC.

21. Remount the GC cover

22. Restart the GC.

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Analytical Troubleshooting

Chapter at a Glance…

SYMPTOMS SOURCE

Baseline

Behavior	Characteristics	Cause	Remedy
Drifting		Accumulation of stationary phase	Replace the end section of the column.
		Carrier gas cylinder pressure too low to allow control	Replace the carrier gas cylinder or increase the pressure.
		Drifting carrier gas or combustion gas flows	Check the gas controllers.
		Accumulation of impurities in column	Check impurity levels in the gas source. Use correct gas purity.
Drifting	Falling	Carrier gas leak in the system	Perform a leak test and ensure the tightness of the connections on the carrier gas line.
		Column is baking out	Allow enough time for the column to stabilize.
	Rising	Accumulation of impurities in column	Check impurity levels in the gas source. Use correct gas purity.
		Contaminated detector	Check the detector and clean it.
	Rising under temperature program operation	Column contaminated	Recondition the column.
High-standing current		Carrier gas flow rate too high	Reduce the carrier gas flow.
		Contaminated column	Recondition the column.
		Contaminated gases	Replace gas cylinders or gas filters.

Behavior	Characteristics	Cause	Remedy
High-standing current		Excessive column stationary phase bleeding	Check the oven temperature, ensuring it doesn't exceed the column upper limit. Recondition the column. Replace the column.
		Loose connections	Ensure all interconnections and screw connections are tight.
Irregular shape	Dip after solvent peak	Detector contaminated	Bake out or clean the detector.
	S-shaped	Excessive column bleed during column temperature programming	Reduce the upper column temperature. Bake out the column. Install a high temperature column.
		Oxygen contamination is decomposing the stationary phase	Install oxygen filters in carrier gas line.Check pneumatic and inlet systems for leaks. Use correct gas purity with low oxygen content.
	Square waves	Large AC power fluctuations; heavy equipment on same line	Use a dedicated clean AC line of sufficient amperage
Noise	High-frequency noise	Contaminated detector	Isolate the detector from electronics. If noise disappears, clean the collector.
		Combustion-gas flow too low or too high	Check the detector-gas flows
		Contaminated column	Condition the column.
		Contaminated detector gas supply	Check the gas purity and install appropriate filters
		Defective electrometer	Replace electrometer.

Behavior	Characteristics	Cause	Remedy
Noise	High-frequency noise	Detector temperature higher than column maximum temperature	Reduce the detector temperature to the column temperature upper limit.
		External electrical interference	Attach an AC line monitor and check purity of the AC supply
		Loose column fittings	Tighten fittings accordingly.
		Loose detector electrical connections	Make sure the leads are properly connected.
Spiking		Defective electrometer or amplifier	Replace the electrometer or amplifier
		F.s. column too close to flame (FID)	Lower the column to the correct position (2-3 mm below the tip of the jet).
		Dirty jet or detector	Isolate the detector from the electronics. If noise disappears, clean the jet and collector.
		External electrical interference	Attach an AC line monitor and check the purity of the AC supply
		FID temperature too low	Increase the temperature to at least 150°C

SYMPTOMS SOURCE

Peaks

Behavior	Characteristics	Cause	Remedy
Broadening		Column flow too high	Reduce the flow to slightly above optimum.
		Column flow too low	Increase the flow to slightly above optimum.
		Split flow too low in split injection	Increase the flow to 40-50 ml/min.
		Column performances degraded	Test the column at the optimum flow rate.
		Dirty injector	Clean or replace the liner.
		Stationary phase accumulated in the outlet	Remove the last two coils from the column.
		Detector base body temperature too low.	Increase the temperature to 5°C below the column maximum.
Clipping	at bottom	Detector or integrator zero set too low	Set the zero correctly.
	at top	Data system zoomed in too close	Zoom out to view the entire chromatogram.
		Detector or integrator attenuation set too low	Set the attenuation higher.
		Detector range too sensitive	Set a less-sensitive detector range.
		Incorrect input to recording unit	Correct and check the recording unit.
Double peaks		Injection speed too low	Inject more rapidly in a smooth motion.
		Wrong autosampler injection speed or mode	Use a higher speed.

Behavior	Characteristics	Cause	Remedy
Fronting		Column or detector overloaded	Decrease the injected amount and/or analyte concentrations. Increase the split ratio.
		Column temperature too low	Increase the temperature.
		Stationary phase too thin	Use a thicker-film column.
Ghost peaks		Contaminated carrier gas	Replace the cylinder or replace the filter.
		Contamination from laboratory glassware.	Ensure the glassware is clean and contamination-free.
		Decomposition of injected sample	Decrease the injection port temperature. Use the on-column injection technique.
		Dirty injection solution	Carry out adequate clean-up of sample prior to injection.
Ghost peaks	Broad ghost peaks	Contaminated inlet or pneumatics	Remove the column and bake out the inlet. Use a high-quality septum. Replace the split vent filter. Install an in-line filter between the pneumatics and the inlet.
		Incomplete elution of previous sample	Increase the final oven program temperature or total run time. Increase the column flow rate.
Irregular shape	Chair-shaped	Solvent flooding of column	Increase the initial oven temperature. Reduce the injection volume (OC). Install a retention gap (OC).
Negative peaks	All	Integrator wires reversed	Correct the connections.

Behavior	Characteristics	Cause	Remedy
Negative peaks	Some	Symptom can be normal	
No peaks	after solvent peak	Carrier gas flow too high	Reduce the carrier gas flow rate.
		Combustion gas flow incorrect	Check the combustion gas flow.
		Detector contaminated	Bake out or clean the detector.
		FID flame extinguished by solvent peak	Check the detector temperature.
		Too much sample injected	Inject less sample.
		Incorrect column position in S/SL injector (too high)	Check the column position.
No peaks	at all	Clogged syringe needle	Replace or repair the syringe.
		Column broken or disconnected	Check the column and connections.
		Defective electrometer or amplifier	Replace the electrometer or amplifier.
		Defective recording device	Replace the recording device.
		FID flame is out	Light the flame.
		Poor or missing electrical connection	Check the cable connections.
		Incorrect column position in S/SL injector (too high)	Check the column position.
Tailing	Sample peaks	Column degradation causing activity	Inject a test mixture and evaluate the column.
		Column/oven temperature too low	Increase the column/oven temperature. Do not exceed the recommended maximum temperature for the stationary phase.
		Dirty liner	Clean or replace the liner.

Behavior	Characteristics	Cause	Remedy
Tailing	Sample peaks	Glass wool or inlet liner causing activity	Replace with fresh silanized wool and a clean inlet liner.
		Inlet temperature too low	Increase the inlet temperature
		Poor or obstructed column connections	Remake the column inlet connection.
		Wrong stationary phase	Replace the column according to the column manufacturer literature.
Tailing	Solvent peak	Incorrect column position in inlet	Reinstall the column.
		Initial oven temperature too high (On Column)	Reduce the initial oven temperature.
		Septum purge flow too low and/or split/splitless vent flow too low	Check and adjust the septum purge and vent flows.
		Too large injection size	Reduce the injection size.
Unresolved peaks		Carrier gas flow rate too high	Reduce the carrier gas flow rate
		Column deteriorated	Replace the column
		Column temperature too high	Lower the column oven temperature
		Column too short	Use a longer column
		Incorrect column choice	Install a suitable column
		Injection technique is not adequate	Choose a correct injection technique

SYMPTOMS SOURCE

Results

Behavior	Characteristics	Cause	Remedy
Low reproducibility of peaks area		Concentration not compatible with the dynamic range of the detection system	Ensure that the sample concentration is suitable for the detection system.
		Inappropriate injection technique	Try a different injection technique.
		Injection parameters inappropriate	Check the injection temperature and flow rates.
		Non reproducible sample injection technique	Evaluate the sample preparation sequences. Compare the results with a series of standard injections.
		Leaking syringe or septum	Check and replace the syringe and/or septum at regular intervals.
		Leaks at the injection	Check the column connections. Run a leak check.
		Poor injection technique	Carefully meter the injected amount. Use a clean, good-quality syringe.
		Poor split flow or ratio control	Monitor the flow. Replace the in-line filter.
Poor sensitivity	with increased retention time	Carrier gas flow rate too low	Increase the carrier gas flow rate. Locate and remove possible obstructions in the carrier gas line. Check the injector/column ferrules.

Behavior	Characteristics	Cause	Remedy
Poor sensitivity	with normal retention time	Leaks in the GC carrier gas line.	Run a leak test and correct leaks.
		Syringe leaks during injection	Replace syringe or piston seals, if applicable.
		Split injection temperature too low	Increase the temperature of the injector.
Retention times	Decreasing	Stationary phase deteriorated by oxygen and/ or water	Use a carrier gas free of oxygen and water.
		Stationary phase loss due to column bleeding	Reduce the column temperature.
	Increasing	Increasing carrier leakage	Check the septum and column connections.
		Carrier gas supply running out	Replace the bottle.
	Low reproducibility	Drifting or unstable pneumatic controller	Monitor the column pressure or flow. Check and replace the controller if necessary.
		Poor injection technique	Start the run at consistent time after injection.
		Sample size is too large	Reduce the injected amount and/ or volume.
		Unstable column temperature	Check the main oven door and cooling flap. Monitor the column temperature.

A

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
Manual: Maintenance and Troubleshooting Manual
Part No.: 31709180

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:
Thermo Fisher Scientific S.p.A
Strada Rivoltana km 4
20090 Rodano (MI)
ITALY
Fax: +39 0295059388

B

Reagents Safety Information

Material Safety Data Sheets for the chemicals mentioned in the sequences in this manual should be requested from respective manufacturers. You should comply with the relevant legislation in your area.

Chemical safety information is also available from the Internet. For example, International Chemical Safety Cards can be found at http://www.cdc.gov/niosh/ipcs/ipcsname.html

The following are the chemical references of the solvents mentioned in this manual.

ACETONE

ACETONE
2-Propanone
Dimethyl ketone
C_3H_6O/CH_3-CO-CH_3

Molecular mass: 58.1

CAS # 67-64-1
RTECS # AL3150000
ICSC # 0087
UN # 1090
EC # 606-001-00-8

METHANOL

METHANOL
Methyl alcohol
Carbinol Wood alcohol
CH_4O/CH_3OH

Molecular mass: 32.0

CAS # 67-56-1
RTECS # PC1400000
ICSC # 0057
UN # 1230
EC # 603-001-00-X

TOLUENE

TOLUENE
Methyl benzene
Toluol
$C_6H_5CH_3/C_7H_8$

Molecular mass: 92.1

CAS # 108-88-3
RTECS # XS5250000
ICSC # 0078
UN # 1294
EC # 601-021-00-3

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
DS	data system

E

ECD	Electron Capture Detector
EMC	electromagnetic compatibility
ESD	electrostatic discharge

F

ºF	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
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
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:
°C min^{-1} instead of °C/min
g L^{-1} instead of g/L

Operating Sequences