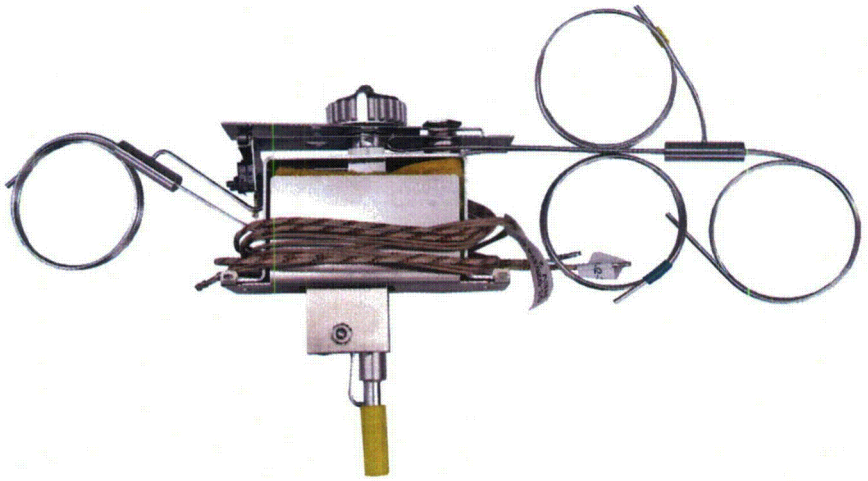


The S/SL is a dedicated Split/Splitless Isothermal Capillary Injector which operates in 2 modes:

- Split
- Splitless

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

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

Features of the S/SL Injector

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

Automatic Start Switch

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

WARNING:
BURN HAZARD

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

S/SL Injector Inserts

Refer to S/SL Injector Inserts on page 324.
Note that all S/SL Injector inserts are deactivated for maximum inertness.

S/SL Modes of Injection

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

Split Mode

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

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

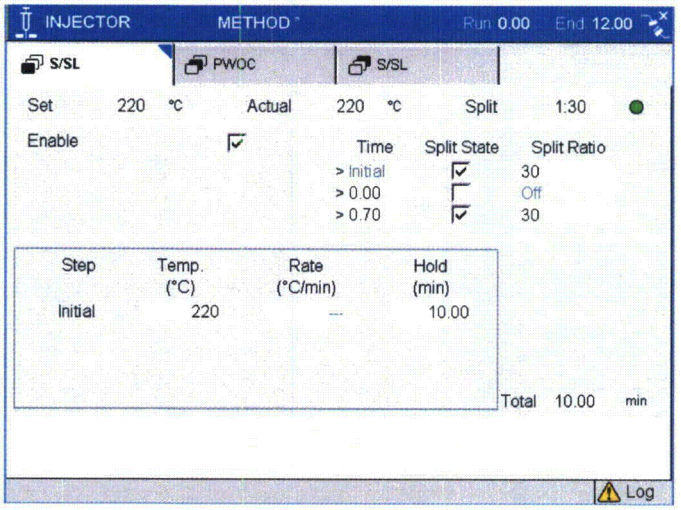
Splitless Mode

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

S/SL Injector Screen

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

In the example below is a splitless injection. The Injector is held in the split state for the initial period with split ratio of 30. When the run is started the split state will be switched Off. After 0.9 minute the split is switched on again.



S/SL Electronic Flow Control (EFC)

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

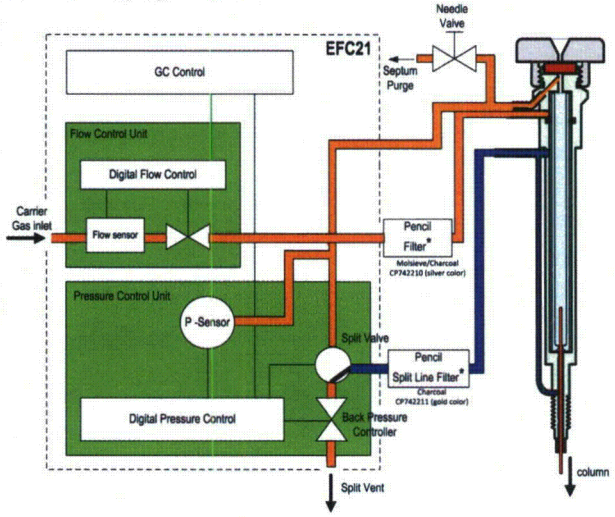


Figure 7: EFC21 and S/SL in Split mode Flow Diagram

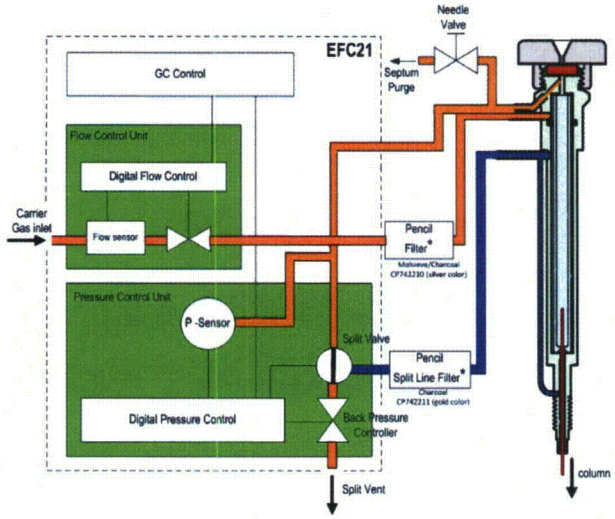


Figure 8: EFC21 and S/SL in Splitless mode Flow Diagram

* See Pencil Filters on page 315

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

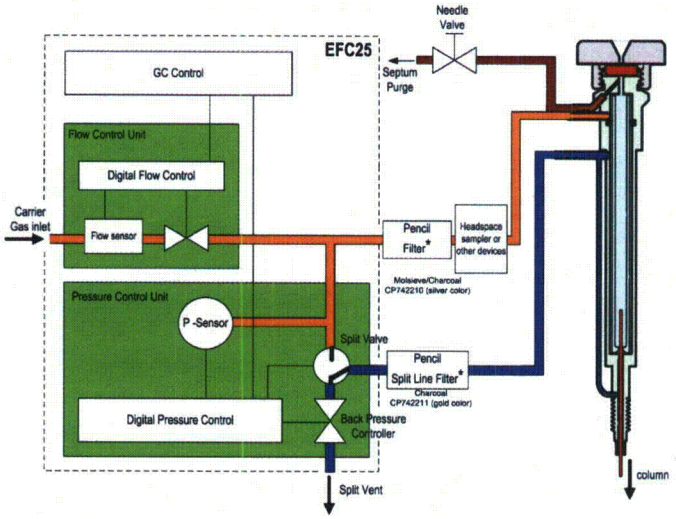


Figure 9: EFC25 and S/SL Flow Diagram

* See Pencil Filters on page 315

S/SL Manual Pneumatics

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

In the splitless mode, gas does not flow through the split line from the Injector to the 3-way solenoid valve (see Table 5). Rather, gas from the flow controller by-passes the Injector to pass through the 3-way solenoid valve. The back pressure regulator uses this flow to control column head pressure, which in turn controls carrier gas flow through the column.

Operation of the S/SL Injector

The following section describes how to operate the S/SLSplit/Splitless Capillary Injector with the Bruker 436-GC/456-GC. It is separated into a series of procedures, starting with installation of a column and basic programming of the Injector from the display, followed by detailed information on the various modes of injection.

Column Installation

The "Connect Capillary Column to Injector" procedure (available in the Installation Manual) describes the installation of a capillary column in a Bruker 436-GC/456-GC equipped with an S/SL Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see "Column Installation and Conditioning" procedure (available in the Installation Manual).

Column Installation in Detector

For connecting a capillary column into a detector, see "Connect Capillary Column to Detector" procedure (available in the Installation Manual).

Setting S/SL Gas Flow Rates

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

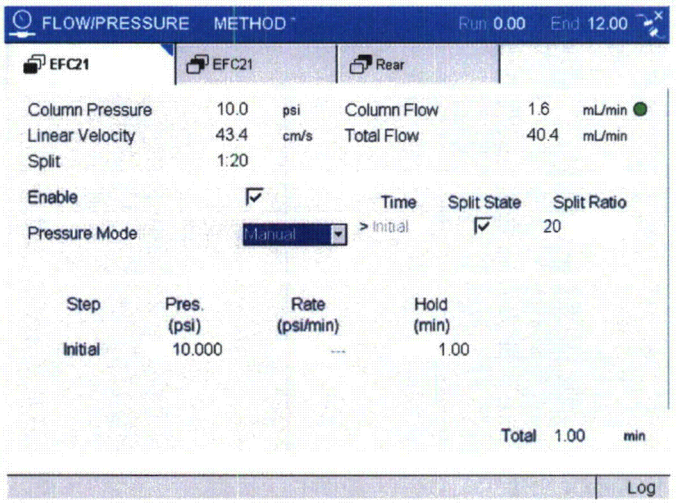
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged

EFC Pneumatics

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

The S/SL is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessible via the FLOW/PRESSURE key on the 436-GC/456-GC display.

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



Pressure Mode

For the Pressure Mode 3 desired pressure modes are available: Manual, Constant Flow and Constant linear velocity.

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

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

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

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

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

The EFC21, EFC24 and EFC25 support **Constant Linear Velocity** mode. This mode improves the RT (Retention Time) stability when the ambient pressure is fluctuating.

Pressure Pulse

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

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

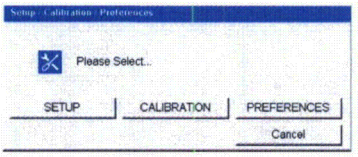
If Pressure Pulse is used, perform a septum purge calibration using the higher pressure value of the pressure pulse.

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

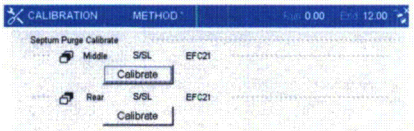
Septum Purge Calibration

The 436-GC/456-GC is equipped with a septum purge for the Model S/SL split/splitless Injector and is controlled by a manual needle valve. This is located behind the front cover of the GC. The manual needle valve can offer advantages over fixed restriction types as it can be adjusted to suit the more demanding applications. For the accurate display of total column flow and velocity it does require calibration when columns of different length and diameter are installed. With type EFC21, the septum purge calibration routine should be carried out when the instrument is first set up or when a new column is installed or when a significantly different pressure point is chosen.

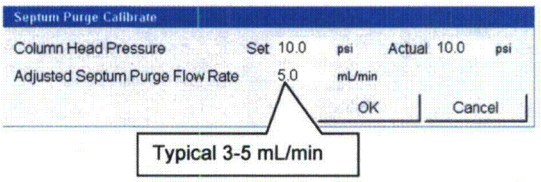
1. Press the SETUP key and click on Calibration.



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



3. Enter the desired Column Head pressure.



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

5. Press OK.

Manual Pneumatics

Tools and equipment needed

Bubble or electronic flowmeter.

1. Turn the S/SL Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.
2. Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge).

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

3. Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.
4. Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3 - 5 mL/min. Readjust the split vent flow rate to 50 mL/min.
5. Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

With manual pneumatics the split valve should be configured in the sample delivery/valve table in set- up of the 436-GC/456-GC. Split/splitless mode can be programmed through the valve table in the sample delivery page of the GC method.

S/SL Modes of Operation

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

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

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

In many cases switching from one S/SL mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of this manual, on page 110. In most injection modes the insert is installed with an O-ring.

Split Injection

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

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

Table 13: Split Injection Typical Conditions

Splitless Injection

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

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

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

Table 14: Splitless Injection Method Parameters

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

Testing the S/SL Injector Performance

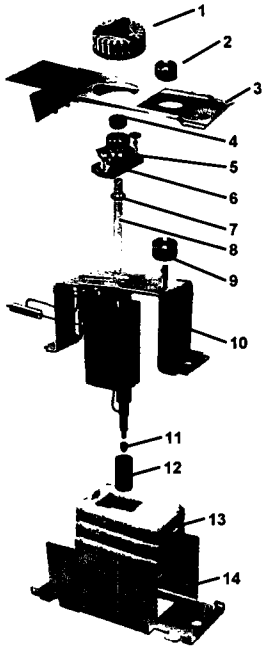
The following procedure describes how to test the performance of the S/SL Capillary Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for 436-GC/456-GC.

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	8200504801	3.00 µg/µL of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	8200504802	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	8200504803	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
NPD	8200504804	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C_{17} in iso-octane.
FID	8200504807	30 ng/µL of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 15: Detector Test Samples

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and NPD, 0.05 for TCD, 10 for PFPD and 1 for ECD. The resultant chromatogram on page 302 should approximate that shown in the detector section of this manual.

S/SL Injector Assembly



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

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

6. Purge head screw (2x 391866308)
7. O-ring, Liner
   Graphite, 6.5 mm for Splitless (392611930)
   Viton, 6.3-6.5 mm (8850103100)
8. Glass Insert (default 392611936[1] More liners)
9. Spacer (39258101)
10. Injector Body
    Stainless Steel (392599401)
    Inert Steel (392599411)
    Manual (392599501)
11. Column Ferrule (see table below)
12. Column Nut 0.9 mm brass for capillary connections (394955100)
    Column Nut 1.6 mm brass for 1/16" connections (CP742351)
    Column Stainless Steel Nut 0.9 mm for High temperature applications (CP743117)
13. Insulation
14. Cover

Figure 10: S/SL Injector Assembly[5]

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

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

Maintenance

Before maintaining the S/SL Injector please read the common Injector/septum maintenance information in the section Maintenance,on page 312.

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

Tools Required

- Tweezers or septum pick (P/N:7200008400).
- Injector nut wrench (P/N: 0390842300).
- Philips screwdriver (long handle).
- Clean laboratory tissue.

WARNING: BURN HAZARD

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

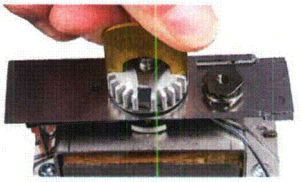
CAUTION

Before proceeding any maintenance procedure; extinguish the detector flame (if any) and cool down detector and oven temperature to <50 °C.

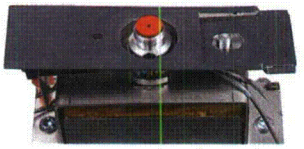
Remove the Glass Insert

Follow these steps to remove the glass insert from the S/SL Injector.

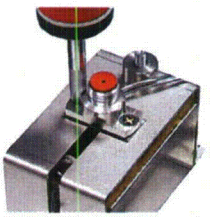
1. Use the Injector nut wrench to remove the Injector nut. Place the nut on a clean surface (e.g., clean tissue).



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

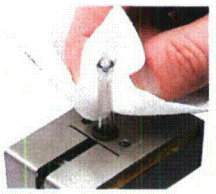


3. Unscrew the 2 captive screws holding the top of the Injector to the base. Carefully move this assembly upwards and to one side. The Septum purge line and carrier gas supply lines may hinder movement of the top piece somewhat.



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

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



Replace the Glass Insert

Follow these steps to replace the glass insert in the S/SL Injector.

1. Using tweezers or clean lab tissue, carefully slide the new insert into the Injector body. Place a new o-ring over the insert.
2. Carefully place the top of the Injector over the insert. Tighten the 2 captive screws alternately for uniform sealing. Note that placing the top piece onto the body may take some care since the gas supply line and septum purge lines may have minor resistance to movement.
3. Install a new septum, place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/4 turn using the Injector nut wrench.

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

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

Injector body cleaning

To clean the Injector body, proceed as follows:

1. Cool down the all temperature zones using the procedure on page 110. Wait for the zones to reach their set temperatures and then turn the column oven off by unchecking the checkbox "Power" located in the Oven screen .
2. Use the 5/16" open-end wrench to loosen the capillary column nut.
3. By hand, carefully withdraw the fused silica column and nut from the Injector assembly. Place the column nut and column end on the floor of the GC oven.

WARNING:
CHEMICAL HAZARD

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

WARNING:
FIRE HAZARD

4. Remove the Injector nut, septum and top of Injector.
5. Remove the Insert.
6. Moisten a cotton swab and a stick with methanol and gently swab center of the Injector body.

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

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

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

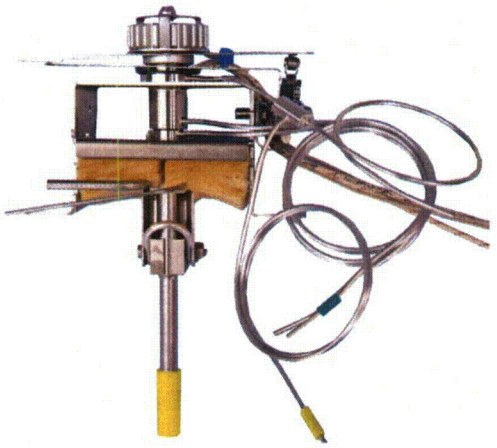
10. Gently twist the column nut in the opposite direction used to tighten the nut. Place the column nut on the end of the Injector and tighten the nut. This procedure minimizes twisting the column.

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

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

13. Turn the column oven back on by checking the checkbox "Power" in the oven screen  .

THE PTV INJECTOR



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

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

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

Features of the PTV Injector

- The PTV can be operated isothermally or temperature programmed. The temperature range of the Injector is from -160 to 450 °C. Sub-ambient temperatures are achieved using cryogenic cooling.

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

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

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

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

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

- The PTV combined with the ChromatoProbe on page 131 is an effective tool for chromatographic analysis of dirty samples (liquids, solids and slurries, blood, urine or milk), without cleanup or extraction. The ChromatoProbe is based on intra-Injector thermal extraction of the semi volatile compounds in a sample microvial, while non-volatile residue is retained in the microvial, which is disposed of after the analysis.

Automatic Start Switch

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

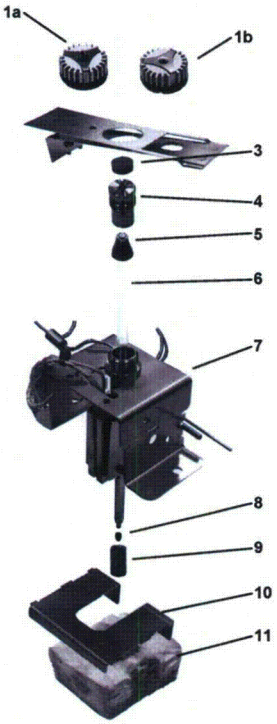
WARNING:
BURN HAZARD

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

PTV Injector Inserts

Click here to see the PTV Injector Inserts, on page 327.
Note that all PTV Injector inserts are deactivated for maximum inertness.

PTV Injector Assembly



1. a Injector Nut (392595401) default (CP8400/8410)
   b Injector Nut (394966601)
   Injector Nut Wrench, (390842300)
2. Automatic Start Switch (390820601)
3. Septum, 11.5 mm
   BTO (lowest bleed, CR298777)
   Marathon (Autosampler, CR239787)
   Advanced Green 3 (general purpose, CR246725)
   Septum pick, 7200008400
4. Septum Support (391867600)
5. Insert Ferrule (392534201)
6. Glass Insert, default 392611945 More liners
7. Injector Body

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

8. Column Ferrule, see table below
9. Column Nut 0.9 mm brass for capillary connections (394955100)
   Column Nut 1.6 mm brass for 1/16" connections (CP742351)
   Column Stainless Steel Nut (0.9mm) for High temperature applications (CP743117)
10. Cover
11. Insulation

Figure 11: PTV Injector Assembly

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

Table 17: PTV Column Ferrules 1/16"

PTV Modes of Injection

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

Temperature Ramp Splitless Mode

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

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

Large Volume

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

Split Mode

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

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

Isothermal Splitless Mode

The 3.4 mm insert packed with glass wool is typically used for isothermal splitless injection. The small capillary section of the glass insert fits snugly around the syringe needle and restricts backflush of the sample vapor during injection. In the splitless injection mode, the sample enters the column during a variable sampling time at the beginning of the analysis. This period is typically 30 - 90 seconds during which there is no flow from the Injector to the split vent. After the sampling time, the Injector is vented to clean any residual sample out of the Injector. The S/SL Injector on page 97 is optimized for Splitless application.

On-Column Modes

In the on-column modes the column is sealed to the glass insert (0.18 - 0.53 mm columns). This ensures that there is maximum transfer of sample to the column. In the on-column modes the

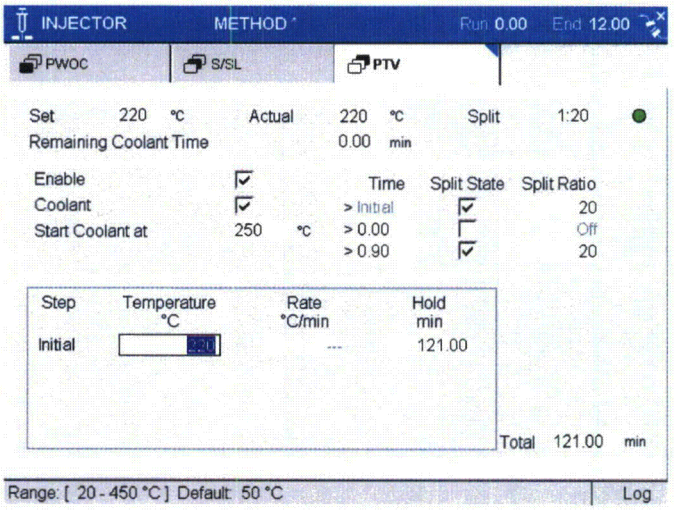
sample is normally injected at or below the solvent boiling point and then the Injector is ramped to transfer the sample onto the column. The COC OC Injector, on page 157 is optimized for On-Column applications. Especially for applications operating at an oven temperature range that requires short wide-bore columns.

PTV Injector Screen

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

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

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



PTV Electronic Flow Control (EFC)

The Electronic Flow Control module used on a PTV Injector is identified as a type EFC21 or EFC25. In simple terms it duplicates the behavior of the PTV manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure.

As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC21 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

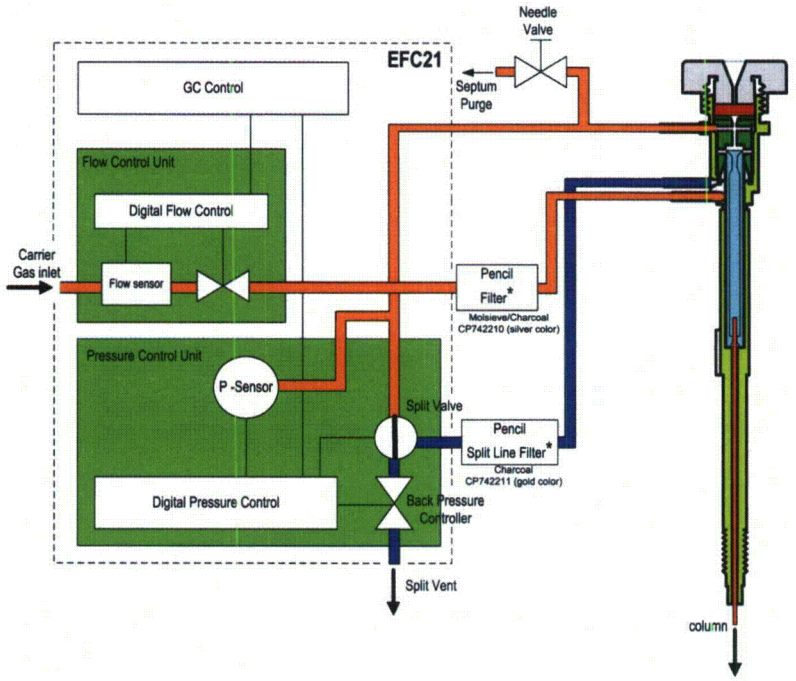


Figure 12: EFC21 and PTV in Split mode Flow Diagram

*Pencil Filters on page 315

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

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

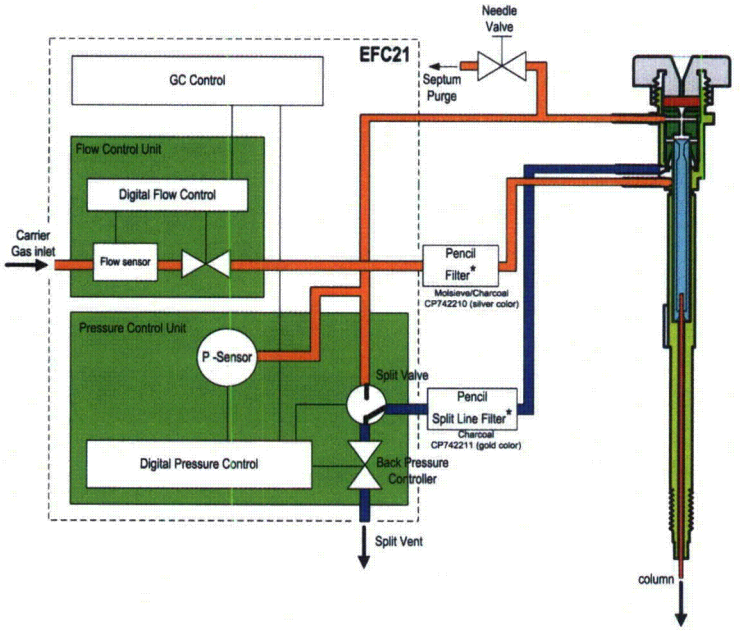


Figure 13: EFC25 and PTV Flow Diagram

* See Pencil Filters on page 315

PTV Manual Pneumatics

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

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

Operation of the PTV Injector

The following section describes how to operate the PTV Injector with the Bruker 436-GC/456-GC. It is separated into a series of procedures, starting with installation of a column and basic programming of the Injector from the display, followed by detailed information on the various modes of injection.

Column Installation

The Connect Capillary Column to Injector procedure (available in the Installation Manual) describes the installation of a capillary column in a Bruker 436-GC/456-GC equipped with a PTV Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning (available in the Installation Manual).

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector (available in the Installation Manual).

Setting PTV Gas Flow Rates

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

CAUTION

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

EFC Pneumatics

Type EFC21 used with the PTV Injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow.

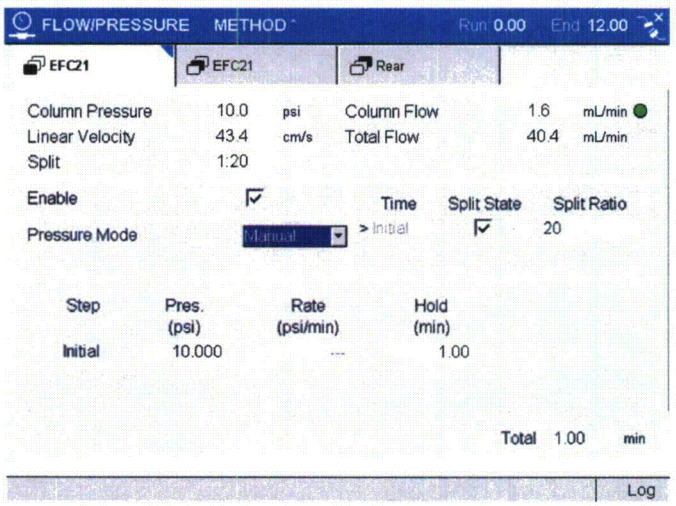
In addition, a split ratio can be set or time programmed. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven.

When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

The PTV is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 436-GC/456-GC display.

The Type EFC21 status field displays the actual column head pressure (in the units chosen in Setup), calculated column flow rate, calculated column linear velocity and the split ratio. The split ratio status is either off if the split state is set to OFF, or a whole number.

The lower part allows you to view/edit constant flow mode.
The following screen is an example of a type EFC21 method.



Pressure Mode

For the Pressure Mode 3 desired pressure modes are available: Manual, Constant Flow and Constant linear velocity.

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

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

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

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

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

The EFC21, EFC24 and EFC25 support **Constant Linear Velocity** mode. This mode improves the RT (Retention Time) stability when the ambient pressure is fluctuating.

Pressure Pulse

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

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

If you use Pressure Pulse you will need to perform septum purge calibration using the higher pressure pulse pressure.

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

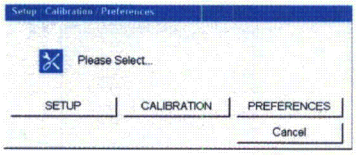
Septum Purge Calibration

The 436-GC/456-GC is equipped with a septum purge for the Model PTV Injector and is controlled by a manual needle valve. This is located behind the front cover of the GC.

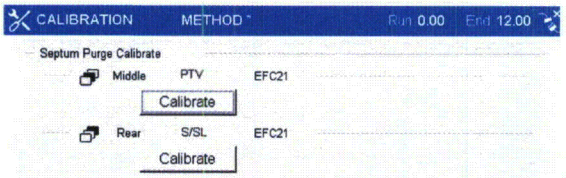
The manual needle valve can offer advantages over fixed restriction types as it can be adjusted to suit the more demanding applications. For the accurate display of total column flow and velocity it does require calibration when columns of different length and diameter are installed.

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

1. Press the SETUP key and click on Calibration.

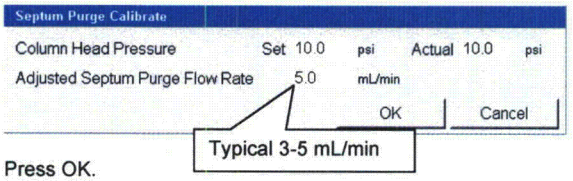


2. The Calibration page will appear, choose Calibrate for the correct Injector (front, middle or rear).



3. Enter the desired Column Head pressure.

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



5. Press OK.

Manual Pneumatics

Tools and equipment needed

- Bubble or electronic flowmeter, e.g., Intelligent Digital Flowmeter.

1. Turn the PTV Split Flow Controller (on the GC pneumatics panel) counterclockwise to open the split flow controller.
2. Adjust the Back Pressure Regulator (on the GC pneumatics panel) to establish a positive column head pressure (monitored on the pressure gauge).

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

3. Connect the flowmeter to the split vent on the left side of the GC and measure the split vent flow rate. Turn the Split Flow Controller valve to adjust the split vent flow rate to 50 mL/min.
4. Adjust the Septum Purge Needle valve to adjust the septum purge flow rate to 3-5 mL/min. Readjust the split vent flow rate to 50 mL/min.
5. Before heating the column, purge the system with carrier gas for 10 - 15 minutes.

PTV Modes of Operation

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

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

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

In many cases switching from one PTV mode to another involves changing the glass insert. A detailed stepwise procedure to carry out this task is given in the Maintenance section of this manual, on page 129. In most injection modes the insert is installed with a graphite ferrule. Use the special fixture supplied with the PTV accessory kit to install the ferrule correctly on the insert.

Isothermal Split Injection

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

Injector Insert	Install the fritted split insert (0392611946) or unpacked split insert (0392611945). See instructions in the Maintenance section on page 129 for changing the PTV insert.
Column Installation	7.5 cm from the bottom of the column nut at the base of the Injector
Injector Temperature	Isothermal 250 °C.
Column Temperature	50 °C initial for 0.1 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier Gas Control	Set the split mode to ON for the duration of the run in the PTV method section and set the split ratio to 100. The sample will be split upon injection and a representative portion representing 1/100th of the amount injected will enter the column. Note that the split ratio is a method specific parameter and should be set appropriately for individual analyses. In addition the Injector pressure or pressure ramp should be set to achieve the desired column flow rate.

Table 18: Split Injection Typical Conditions

Isothermal Splitless Injection

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

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

Injector Insert	Install the standard 3.4 mm ID splitless insert (P/N: 0392611945).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature isothermal 250 °C.
Column Temperature	50 °C initial for 1 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 0.75 minutes. In this case the splitless sampling time is 0.75 minutes. The split ratio during the split ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow rate.

Table 19: Isothermal Splitless Injection Method Parameters

Temperature Ramped Splitless Injection

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

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

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

Injector Insert	Install either the narrow bore 0.5 mm ID insert (0392611949) or the glass wool packed 2 mm ID insert (0392611953) or the open 3.4 mm ID splitless insert (0392611945).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature 65 °C, hold for 0.1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Set the initial split mode to OFF and time program it to ON after 2.00 minutes. In this case the splitless sampling time is 2.00 minutes. The split ratio during the ON period should be set to 50. Set the appropriate pressure or pressure ramp to achieve the desired column flow.

Table 20: Temperature Ramped Splitless Injection

On-column Injection

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

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

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

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

Injector Insert	Install the on-column insert (P/N: 0190010907) for wide bore (0.53 mm ID) columns.
Seal/Position Column	Seal the column within the tapered insert.
Injector Temperature	Initial temperature 50 °C, hold for 0.1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 2 min, ramp to 250 °C at 20 °C/min, hold 5 minutes.
Carrier gas control	Split does not apply in on-column mode. The Splitless vent flow must be set to 20 mL/min or greater in Setup. Delete any splitter program and set initial state to off in the Injector screen.

Table 21: On-Column Injection

Large Volume Injection

The large volume injection technique is used where the absolute lowest level of detection is required. Up to 250 µL of sample may be introduced into the PTV Injector. The sample is injected at a very slow rate while the Injector temperature is set a few degrees below the solvent boiling point. In the example below, hexane is used as the solvent which has a boiling point of 68 °C.

Using the large volume injection technique the Injector is maintained in the split ON state at the beginning of the run to vent most of the solvent. The sample components are trapped in the Injector insert so the same type of insert is used as for temperature ramped splitless injection. The split state is then programmed to OFF and the Injector temperature ramped to transfer the sample components to the head of the column. The following are typical conditions for a large volume injection.

Injector Insert	Install either the narrow bore 0.5 mm ID insert (0392611949) or the glass wool packed 2 mm ID insert (03926119-53).
Column Position	7.5 cm from the bottom of the column nut at the base of the Injector.
Injector Temperature	Initial temperature 66 °C, hold for 1 minute, ramp to 250 °C at 150 °C/minute, hold 10 minutes.
Column Temperature	50 °C initial for 3.00 min, ramp to 250 °C at 20 °C/min, hold 5 min.
Carrier gas control	Set the initial split mode to ON, time program it to OFF after 1.00 minutes and then back to ON after 3.00 minutes. The split ratio should be set to 50 during the split ON periods.

Table 22: Large Volume Injection

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

Testing the PTV Injector Performance

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

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	8200504801	3.00 µg/µl of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	8200504802	33.0 pg/µl of lindane and aldrin in iso-octane.
PFPD	8200504803	20.0 ng/µl of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µl of n-pentadecane in iso-octane.
NPD	8200504804	2.00 ng/µl of azobenzene, methyl parathion; 4.00 ng/µl malathion and 4.00 µg/µl C_{17} in iso-octane.
FID	82005048-07	30 ng/µl of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 23: PTV Test Samples

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

The PTV Injector can be operated in several modes. These modes include split, splitless, on-column and large volume injection. Typically, to change from one mode of operation to another involves changing the Injector insert. The insert should be replaced on a routine basis. This is especially important when dirty samples are analyzed.

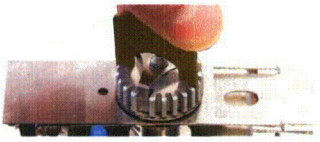
After prolonged use, the PTV Injector glass insert may need to be replaced with a new insert.

Tools Required

- Tweezers or septum pick (P/N:7200008400)
- Injector nut wrench (P/N: 0390842300)
- Flat-blade screwdriver (short handle)
- Clean laboratory tissue
- Graphite ferrules (P/N: 0392534201)
- Insert/ferrule positioning tool (P/N: 0392538500)

Remove the Glass Insert

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



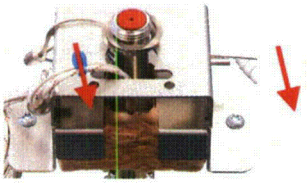
WARNING: BURN HAZARD

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

CAUTION

Before proceeding any maintenance procedure; extinguish the detector flame (if any) and cool down detector and oven temperature to <50 °C.

2. Unscrew the 2 T-20 Torx screws holding the top of the Injector to the base. Carefully move this assembly to one side. The Septum purge line and carrier gas supply lines may hinder movement of the top piece somewhat.



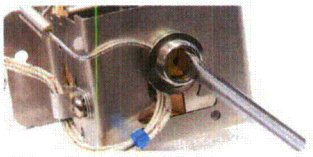
If CP-8400 AutoSampler is installed, remove the Injector access plate by removing the 2 retaining screws and firmly lifting the plate. It may be hot if another Injector is also installed and powered. The plate may be a little tight, do not force it off. Lift straight up, a slight back and forth rocking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

3. With tweezers or septum pick, remove the septum.

Replace the Injector septum each time the glass insert is replaced.

4. Use a clean flat-blade screwdriver to unscrew the septum support nut until it is loose.

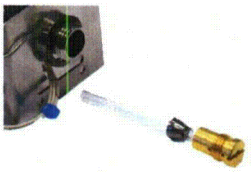
5. Remove the septum support with the tweezers or septum pick.



Typically, when the septum support is removed, the insert and ferrule remain in the septum support. If the ferrule and insert are in the Injector body after the septum support nut is removed, use the tweezers to grasp the top of the insert and lift it from the Injector body.

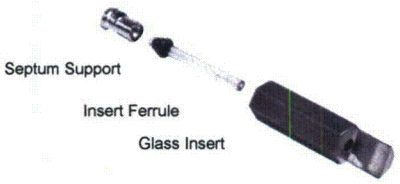
6. Use a laboratory tissue to grasp the glass insert and remove it from the septum support nut.

7. To remove the graphite ferrule from the glass insert, use clean lab tissues to hold the graphite ferrule and the glass insert. Gently turn the glass insert while you pull off the graphite ferrule.

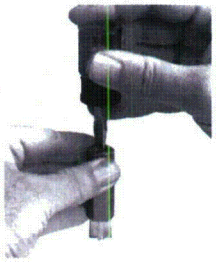


Replace the Glass Insert

1. Use the insert/ferrule positioning tool supplied in the PTV accessory kit to set the 5 mm graphite ferrule on the insert and in the septum support. See the pictures for an exploded view of the tool with septum support, insert ferrule, glass insert and tool as well as the correct position of the tool when setting the ferrule. The objective is to have the ferrule set with the bottom of the insert, flush with the bottom of the tool.



Insert/ferrule positioning tool (392538500)



2. Position the tool as shown on a flat, clean surface. Use clean laboratory tissue on the surface. Tighten the septum support finger-tight. Holding the tool with a 5/8" wrench, give the septum support an extra 1/3 to 1/2 turn past finger-tight. Now unscrew the septum support which now has the ferrule and insert seated in it. If there is any graphite extruded past bottom of the septum support, cut it off with a blade or sharp knife. Carefully wipe off any graphite flakes which may adhere to the insert or septum support. Gripping the septum support unit with a piece of laboratory tissue, carefully put this unit in the PTV Injector and tighten the septum support 1/6-turn past finger-tight.

3. Use tweezers to place a new septum over the septum support.

If the septum has a Teflon face, place the Teflon face toward (down) the column.

4. Place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/4 turn using the Injector nut wrench.

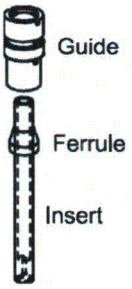
5. Condition the insert by setting the PTV Injector to the split mode and purging with carrier gas for 30 min at 300 °C.

How to Assemble the ChromatoProbe

Refer to the exploded view on the next page.
The ChromatoProbe assembly consists of the adapter, guide, ChromatoProbe, ChromatoProbe cap and the small and large O-rings. One small O-ring is placed on the ChromatoProbe shaft; the other is placed on the ChromatoProbe cap shaft. The large O-ring is placed in the groove in the bottom of the adapter which connects to the PTV Injector body.
The storage stand may be used to hold and protect the ChromatoProbe or ChromatoProbe cap. The user may find the small holes in the top of the storage stand useful for holding microvials during filling or just prior to running an analysis.

How to Install the ChromatoProbe

The ChromatoProbe is designed to be installed into the PTV Injector body. Several parts of the Injector must be removed and replaced with the appropriate parts from the ChromatoProbe kit before the ChromatoProbe can be connected. Remove the Injector nut, septum, septum support (gold piece) and the Injector insert. The Injector nut, septum support and the Injector insert should be set aside for use when you use the Injector in the normal manner. Install the glass insert included in the ChromatoProbe kit into the Injector (3.4 mm split insert). This insert must be installed with the small orifice at the bottom, as shown in the picture. This position will allow the ChromatoProbe to slip into the insert during operation.
Use the following procedure to set the insert, ferrule and the guide.

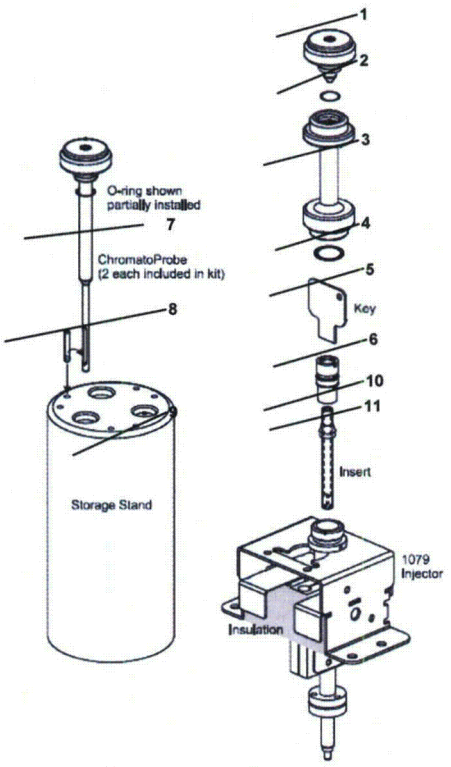


1. Place the 1/4 inch graphite ferrule over the open end of the insert so that the tapered end of the ferrule is up.

2. Place the insert and the ferrule into the guide so that the ferrule can fit snugly inside the guide.

3. Using the ferrule positioning tool (P/N: 0392538500) included with the PTV Injector, place the insert and guide into the tool and screw the guide clockwise with the key provided to tighten the ferrule around the insert. The insert is positioned correctly if the bottom of the insert is aligned with the bottom of the ferrule positioning tool. Refer to the maintenance section of the PTV for removal of the glass insert on page 129.

Once the ferrule is positioned and tightened, the guide and insert can be placed into the PTV Injector body. Screw the guide into the Injector body with the key and tighten snugly.
At this point the ChromatoProbe is ready to be attached onto the Injector. The adapter is attached to the Injector such that the portion containing the large O-ring is screwed onto the Injector. Tighten this fingertight to prevent any leaks. **DO NOT use a tool to tighten this connection**. Insert the ChromatoProbe cap into the top of the adapter and hand tighten to cap off the Injector when not using the ChromatoProbe.



ChromatoProbe Kit (0392567791)

1. ChromatoProbe Cap (0392559201)
2. Small O-ring, 0.239 ID (0393010911)
3. Adapter (0392558901)
4. Large O-ring, 0.426 ID (0393010912)
5. Key (0392569001)
6. Guide (0392558201)
7. ChromatoProbe (0392559101)
8. Microvials (100/bottle) (0392567111)
9. Stand (0392569401)
10. Glass insert 0391846400
11. Graphite ferrule (5 mm) (2/pack) 0392534202 or 0392534201 (10/pack)

- Passivation Lacquer (0392569901)
- Tweezers (2989951000)
- Ferrule (0.4 mm) (10/pack) (2869458001)
- Ferrule, 2-hole (0.4 mm) (2869455901)
- Column nut (0394955100)

Figure 14: ChromatoProbe Assembly

NOTE: If it appears that there is an alignment problem while inserting the ChromatoProbe, follow this alternative method for installing the adapter:

1. Connect adapter to the Injector **without tightening it**.
2. Insert the ChromatoProbe through the adapter with a loose fit and then tighten the adapter to the Injector while the ChromatoProbe is inserted.
3. Finger tighten to prevent any leaks. **DO NOT use a tool to tighten this connection**.

A 2m x 0.10 mm fused silica column with 0.1 µm methyl silicone coating is provided for use with the ChromatoProbe. For a gas chromatograph containing 2 Injectors, it is possible to have the ChromatoProbe and a traditional column connected simultaneously. In this case, please refer to here on page 134. If only one Injector exists or the ChromatoProbe is installed alone, please continue as follows:

1. Remove any column that you have installed in the GC/MS and replace it with this 2 meter column.
2. Use the 0.4 mm single hole ferrule and the column nut provided in the kit.
3. The column should be inserted a distance of 7.5 cm into the Injector (standard for PTV Injectors). The mass spectrometer side is set so that the column end is 1 mm past the end of the transfer line.
4. Set the head pressure to 10 psi and measure a flow out of the split vent of 50 mL/min. This will provide a flow rate of about 1 mL/min into the mass spectrometer and a split ratio of about 50:1.

5. Condition the Injector and the column at 300 °C for 30 minutes. If an EFC is included, provide 1 mL/min column flow rate with split vent of 50 mL/min.

Using the ChromatoProbe

How to Perform Analysis with the ChromatoProbe

Liquids, solids and slurries, or even samples such as blood, urine, or milk can be analyzed using the ChromatoProbe.
Place the sample into a microvial provided with the kit. Solid or powder samples are easier to introduce to the microvial as a liquid solution with a standard syringe. This way is also cleaner and has smaller memory effects.
Alternatively, powders can be introduced into the microvial with the help of a Pasteur pipette.
The microvial containing the sample is placed into the microvial holder position at the bottom of the ChromatoProbe shaft. Refer to the exploded view if this is unclear.
Remove the ChromatoProbe cap from the adapter which should be installed on top of the Injector and position the ChromatoProbe through the adapter, into the Injector.
Finger tighten the ChromatoProbe to seal the Injector. The sample is volatilized by raising the Injector port temperature.
The column can be hot and isothermal or temperature programmed to match the Injector. Since most analyses of solid samples do not require high sensitivity, the split vent should be opened during the entire analysis to only allow low nanogram amounts into the mass spectrometer. Adjust the split flow to achieve a fast response, reduce thermal decomposition and prevent the mass spectrometer from being overloaded and contaminated. A good starting value is about 50:1. If the split ratio is too high for a particular sample, rerun a fresh sample with a lower split ratio to get the desired sensitivity. In order to prevent overloading of the system, either load the smallest amount of solid possible or preferably dissolve the solid and load 1 µL of about 0.1% solution. Note Injector comment below.
Samples can be analyzed in any of the mass spectrometer modes of operation (EI, CI, MS/MS, CI-MS/MS). In general, use AGC target values of 5,000-10,000 to ensure a high spectral quality. A suggested temperature program for the PTV Injector and column oven is as follows.

INJECTOR: 120 °C (or 20 °C above the boiling point of the solvent if a solution is introduced) for 1 minute, then heat at 40 °C/min to 300 °C and hold for 4.5 min.

COLUMN OVEN: 140 °C for 1 minute, then heat at 40 °C/min to 300 °C and hold for 5 min.

If liquids are to be analyzed, the user must empirically determine certain parameters to prevent sample from being inadvertently expelled out of the microvial during analysis, caused by sudden boiling ("bumping") or spraying. These parameters include maximum fill level in the microvials, maximum initial temperature and heating rates of the Injector.

Analysis of Thermally Labile Samples

Thermally labile samples may decompose in the injection port. The hot metal surface of the ChromatoProbe can contribute to thermal decomposition resulting in improper spectra.
In order to eliminate this, Silcosteel® coated ChromatoProbes have been introduced.

Using the ChromatoProbe Along with Traditional GC/MS

For gas chromatographs containing 2 Injectors it is possible to have both the ChromatoProbe column and a traditional column connected simultaneously. This can be accomplished by employing a 2-hole ferrule and connecting both columns into the mass spectrometer. When the ChromatoProbe is not in use, replace the probe with the ChromatoProbe cap.
Heat the ChromatoProbe Injector to about 300 °C with the split vent open to remove any contamination from previous samples. Now, reduce the ChromatoProbe Injector head pressure to 3-5 psi to minimize the helium flow into the mass spectrometer. The total flow of helium should remain between 1 and 1.5 mL/min to insure the best sensitivity and spectral quality. Other options include a "Y" connector for the 2 columns going into a single transfer line into the mass spectrometer. Be aware of the possible problems of surface activity when using this type of connection.

Using the ChromatoProbe as a Dirty Sample Inlet

The ChromatoProbe can be used as an effective tool for chromatographic analysis of dirty samples, without cleanup or extraction. This use of the ChromatoProbe is based on intra-Injector thermal extraction of the semivolatile compounds in the sample microvial, while non-volatile residue is retained in the microvial, which is disposed of after the analysis. For effective use of the ChromatoProbe with dirty samples, the following advice should be considered:

1 The sample microvial should be handled with *tweezers only*. Similarly, the microvial holder should not be touched with the hands to avoid fingerprints and dirt being included in the analysis.

2 Introduce into the microvial the smallest sample size that is easy to quantitatively transfer with a syringe. This is typically 1-3 microliter liquid samples.

3 Solid samples might be blended or dissolved for a more quantitative transfer, as well as for more efficient thermal extraction from the microvial.

4 The sample should be introduced at an Injector temperature about 20 °C above the solvent boiling temperature, to enable fast but gentle solvent vaporization without sample splashing from the microvial. This temperature can be 120 °C for water (urine) or 90 °C for acetone blended fruit and vegetables.

5 The initial column temperature should be low enough to trap the extracted semivolatile compounds. For example, 50-80 °C initial column temperature is desirable for pesticide analyses.

6 Solvent vaporization takes about one minute for each 3-4 microliter sample. The split vent can be opened during that time, with a split flow rate above 20 mL/min to speed up the solvent vaporization.

7 After approximately one minute solvent vaporization time, the Injector temperature should be fast programmed to 250 °C (for pesticides) or the appropriate temperature required for the thermal extraction of the semivolatile compounds. The final temperature chosen is a compromise between higher temperature for more effective and faster thermal extraction and a lower temperature value that might be required to prevent thermal degradation of delicate compounds.

8 During the Injector heatup and thermal desorption time, the split valve must be closed and the carrier gas flow rate must be at least 4-5 mL/min for effective thermal extraction. At lower carrier gas flow rates a higher Injector temperature will be required.

9 The thermal desorption stage takes between 0.5-2 minutes. After that time, the Injector is cooled down, the carrier gas flow rate is programmed to the chromatographic optimal value and the GC oven program can begin in the usual way. Note that the final GC oven upper temperature and time can be significantly reduced since the less volatile compounds are retained in the microvial.

10 At the end of the analysis, dispose of the sample microvial. Do not re-use microvials. The microvial holder should only be removed from the adapter after both the GC oven and Injector are cool and a helium purge flow from the ChromatoProbe protects the column.

11 In addition to passivation of the microvial holder, analysis of thermally labile sample compounds is facilitated with a shorter microvial (up to 6 mm long). The standard 15 mm long microvials can be cut (like a column) to the desirable length. Handle the microvial carefully while cutting to avoid contamination.

12 The microvial volume is 30 microliters. Up to 20 microliters can be loaded and the solvent evaporated outside the GC prior to sample introduction.

13 Test your method for thermal vaporization efficiency, reproducibility and long term stability before beginning routine analysis.

Cleaning

Should the ChromatoProbe become contaminated with time, it can be cleaned by placing it in a solvent such as acetone and placed in a sonic bath. If it is
severely contaminated a mild abrasive can be used to clean it.

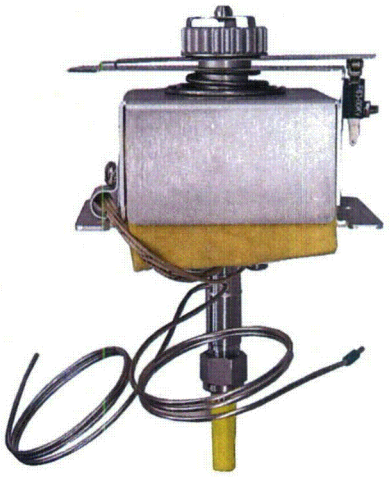
Recommended Reading

1. Aviv Amirav and Shai Dagan, "A Direct Sample Introduction Device for Mass Spectrometry Studies and GC/MS Analysis". European Mass Spectrometry, 3, 105-111 (1997).

2. Hongwu Jing and Aviv Amirav, "Pesticide Analysis with the Pulsed Flame Photometric Detector and a direct sample Introduction Device". Analytical Chemistry, 69, 1426-1435 (1997).

3. Samuel B. Wainhaus, Shai Dagan, Mark L. Miller and Aviv Amirav, "Fast Drug Analysis in a Single Hair". Submitted for publication.

THE PWOC ON-COLUMN INJECTOR



The PWOC On-Column Injector is designed for on-column sample injection onto 530 micron I.D. fused silica columns, 1/4 inch and 1/8 inch O.D. packed columns (glass or metal).

On-column injection provides complete sample transfer from Injector to column results in good quantitation.
The fused silica column or glass column extends all the way from the Injector septum to the detector, providing a clean inert system without cross-over's.

Quick and easy to switch from megabore to packed column or vice versa by changing an adaptor. The PWOC is supplied with capillary mounting hardware as standard. If you wish to operate the Injector in the packed column mode then a packed column adapter kit must be ordered (03925588-91). This kit contains Injector and detector hardware to facilitate use of the Injector with 1/8" metal packed columns. In addition, adapter kits are available for 1/4" columns (03925586-91 for glass and 03925586-93 for S.S.).

The PWOC On-Column Injector is available in manual pneumatics and electronic flow control (EFC) for different laboratory requirements.

Choose from EFC23 (constant flow control) and EFC24 (constant pressure control and constant total flow control) for different application requirements.

Automatic Start Switch

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

WARNING:
BURN HAZARD

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

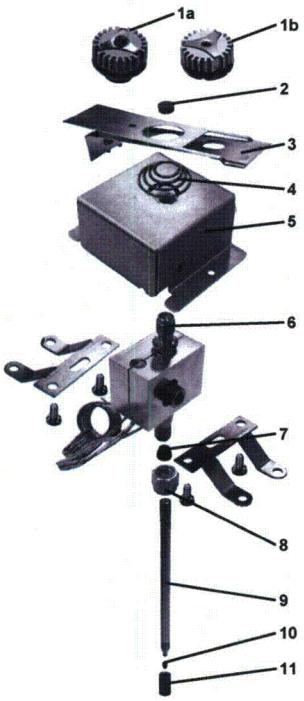
PWOC Injector Insert

Click here to see the PWOC Injector Insert, on page 328.

Note that all PWOC Injector inserts are deactivated for maximum inertness.

Injector Assembly and Insert

A cross-sectional view of the PWOC On-Column Injector with insert and 530µm ID fused silica capillary column is shown below.



1. a Injector Nut (392595501) Default (CP-8400/CP-8410)
   b Injector Nut (390812700)
   Injector Nut Wrench (390842300)
2. Septum, 9.5 mm
   BTO (lowest bleed, CR298705)
   Marathon (Autosampler, CR239188)
   Advanced Green 3 (general purpose, CR246124)
   Septum pick (7200008400)
3. Automatic Start Switch (390820601)
4. Spring
5. Injector cover and insulation
6. Injector Body
   EFC type (392548201)
7. Injector Body Ferrule
   40% Graphite/60% Vespel (up to 400°C) (CR213400)
   Graphite (up to 450 °C) (CR211400)
8. Injector Body Nut (Stainless Steel, SWSS4021)
9. Injector Insert (Stainless Steel, 392543101)
10. Column Ferrule, see table below
11. Column Nut 0.9 mm brass for capillary connections (394955100)
    Column Nut 1.6 mm brass for 1/16" connections (CP742351)
    Column Stainless Steel Nut 0.9 mm for High temperature applications (CP743117)

Figure 15: PWOC On-Column Injector with Insert

Column ID	*Teflon* *Max 250 °C*	*Vespel* *Max 350 °C*	*40% Graphite 60% Vespel* *Max 400 °C*	*Graphite* *Max 450 °C*	*SilTite* *Metal, GC/MS*
0.53 mm ID	CR214108	CR212108	CR213108	CR211108	SG073302

Table 24: PWOC Column Ferrules 1/16" x 0.8 mm

PWOC Electronic Flow Control (EFC)

The Electronic Flow Control module used on a PWOC Injector can be type EFC23 or EFC24.

The EFC23 is used to control the PWOC Injector under flow control, in order to set the constant column flow. EFC23 sets a required carrier flow into the Injector/Column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

The EFC24 is used to control the PWOC Injector in combination with Headspace sampler and other devices that have a long carrier gas line.

Both EFC types are designed specifically for the PWOC Injector to support its various modes of operation. In simple terms it duplicates the behavior of the PWOC manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection.

The type EFC23 flow diagram shown on the next page is an indication of the control mechanism of this type of EFC module. The EFC23 is a constant flow system.

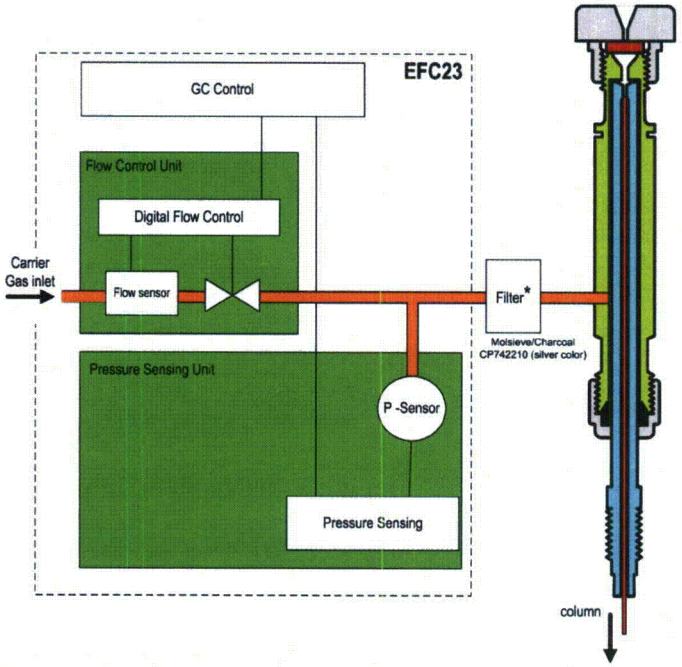


Figure 16: EFC23 and PWOC Flow Diagram

*See Pencil Filters on page 315

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module. The EFC24 is a (constant) pressure system.

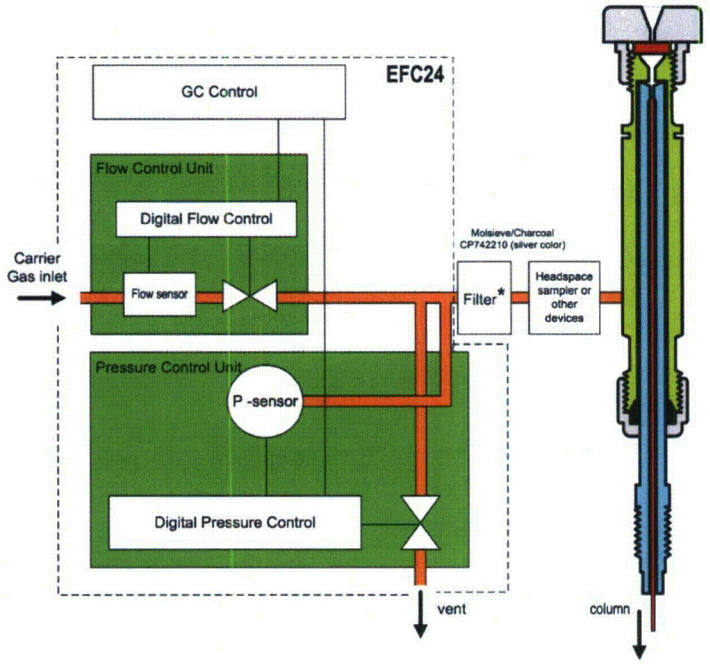


Figure 17: EFC24 and PWOC Flow Diagram

* See Pencil Filters on page 315

Column Installation

The Connect Capillary Column to Injector procedure (available in the Installation Manual) describes the installation (or reinstallation) of a capillary column in a Bruker 436-GC/456-GC equipped with a PWOC Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning (available in the Installation Manual).

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector (available in the Installation Manual).

Setting PWOC Gas Flow Rates

The gas flow rates for the PWOC On-Column Injector can be set using manual pneumatics or Electronic Flow Control (EFC). Note that a positive flow through the column must be set before heating the column.

CAUTION

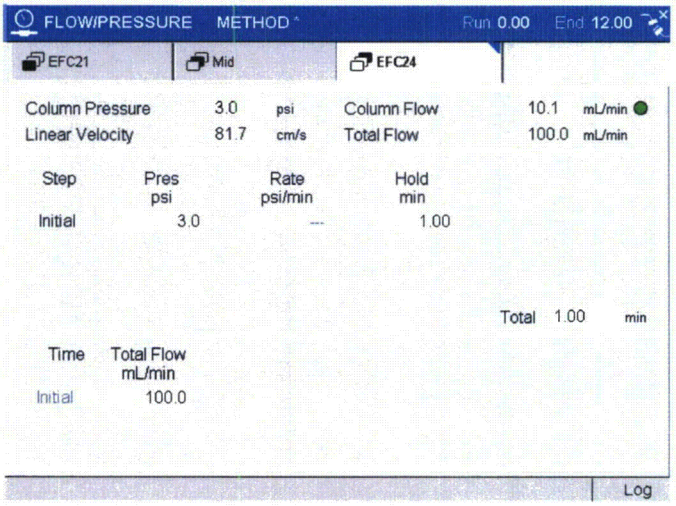
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type EFC24 used with the PWOC or FLASH Injectors allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. With EFC23 equipped Injectors a flow range of 0 – 100 mL/min may be set from the 436-GC/456-GC display. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded. The only user settable parameter with the PWOC type of EFC is the desired flow rate.

The PWOC is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 436-GC/456-GC display.

The next screen is an example if a type EFC24 is used.



Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph here-below for your operational mode.

Capillary Mode

When operating the Injector in the capillary mode, the following carrier gas flow rates are recommended:

Carrier Gas	*Column Flow Rate (Optimum)*	*Column Flow Rate (Typical)*
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Table 25: Carrier Gas Flow Rates for PWOC

Packed Mode

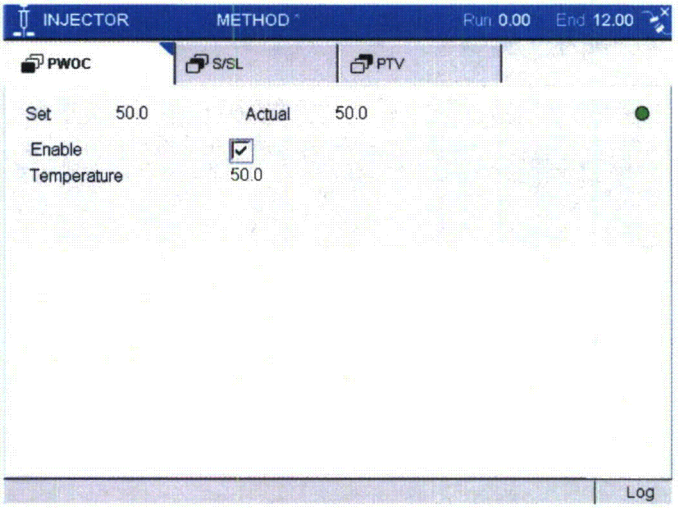
When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min), depending on carrier gas type.

PWOC Operation

This screen contains the specific operating information and procedures required for optimum performance of the PWOC on-column Injector. All installations for both the instrument and the Injector must be completed before continuing further.

PWOC Injector Method

The PWOC Injector method component on the 436-GC/456-GC display contains one screen for setting the required Injector temperature and viewing the status information. The PWOC is an isothermal Injector and cannot be temperature programmed.



Testing the PWOC Injector

The most effective method of testing Injector performance is by running a test sample. The following procedure describes how to test the performance of the PWOC on-column Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The Table 46, on page 302 lists the series of test samples available for Bruker GC detectors.

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range.

Example chromatograms can be found in the detector section on page 302 of this manual.

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C / minute and hold for 5 minutes
Injection Volume	1 µL

Table 26: PWOC Injector Chromatographic testing conditions

Observe the eluting peaks for symmetry, separation and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.

Hardware Replacement and Cleaning Procedures

Routine cleaning and maintenance of the PWOC On-Column Injector system includes septum replacement and column conditioning. Pressure testing and leak checking procedures are included to assure proper functioning of the system.

Septum Replacement

Septum replacement represents the major part of routine chromatographic maintenance. Septum damage from the needle penetrations can be avoided by injecting into the same hole and not using syringes with needles having burrs or bends at the tip which cut the septum.

WARNING: BURN HAZARD

The Injector nut and automatic start switch assembly may be very hot during instrument operation and should not be touched with unprotected hands. Allow sufficient time for the Injector nut and surrounding assemblies to cool before continuing with this procedure.

CAUTION

Handling a septum with bare fingers may result in column contamination. Use tweezers, finger cots, or gloves when installing a new septum.

CAUTION

Before proceeding any maintenance procedure; extinguish the detector flame (if any) and cool down detector and oven temperature to <50 °C.

1. Be sure the Injector nut is cool. Unscrew the Injector nut (use the Injector Nut wrench) and place on a clean, uncontaminated surface.
2. Using tweezers, remove the septum, taking care not to scratch the internal surfaces of the Injector.
3. Using tweezers, place a new high temperature septum in the Injector. If the septum is TFE or FEP coated put that side down.
4. Replace the Injector nut and tighten until resistance is felt, then tighten an extra 1/2 to full turn.

Needle/ Syringe Cleaning

For normal use, rinse the needle/syringe by slowly drawing up and quickly expelling solvent or the next sample to be used. Repeat the process several times.

When rinsing with the next sample, DO NOT expel sample back into the sample container

Leak Checking

Leak checking methods used with the PWOC On-Column Injector are presented in order of overall sensitivity and performance. When leak checking the Injector only, be sure to use the appropriate procedure.

1 Remove the test column and plug the Injector outlet with a 1/16" no-hole ferrule (P/N:28-69459001). Remove the Injector nut, install a new septum, then replace Injector nut and turn nut clockwise until it comes to a stop.

2 Pressurize the Injector to 400 kPa (60 psig) with carrier gas. Turn off the carrier gas flow at the supply.

3 A pressure drop less than or equal to 3 kPa (0.5 psig) in 15 minutes is acceptable. Locate leaks with an appropriate leak detector.

Any changes in temperature while performing this test may result in false readings due to expansion/contraction of the gas with temperature.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

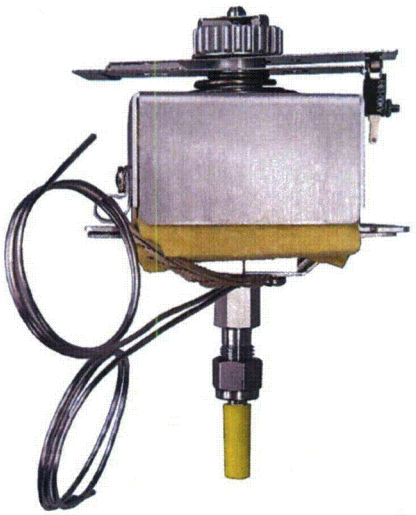
4 An alternate way to leak check the Injector only is to have the column installed in the Injector end and the opposite column end sealed with a flame or appropriate fitting.

Syringe Leak Checking

In some instances, non-reproducible chromatographic responses can be attributed to a worn and/or leaky syringe. The syringe must be leak tight. To check the syringe, insert the needle in an Injector operating at 150-200 kPa (20 to 30 psig) with a no-hole ferrule. Place a drop of solvent at suspect leak locations and look for bubbles.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

THE FLASH VAPORIZATION INJECTOR



The FLASH vaporization Injector is designed for use with wide-bore columns of 0.53 mm ID.
Used with packed glass liner with vaporization volume for dirty samples.
Glass liner is removable from the top. No need to disconnect the column.

The FLASH Injector can operate in 2 modes: the capillary column mode or the packed column mode. Switching from one mode to another involves changing the Injector and detector column mounting hardware.
The FLASH is supplied with capillary mounting hardware as standard.

If you wish to operate the Injector in the packed column mode then a packed column adapter kit must be ordered (0392558892). This kit contains Injector and detector hardware to facilitate use of the Injector with 1/8″ metal packed columns. For ¼" columns, the adapter kit P/N: 392558691 for glass columns and 392558693 for stainless steel columns.

Automatic Start Switch

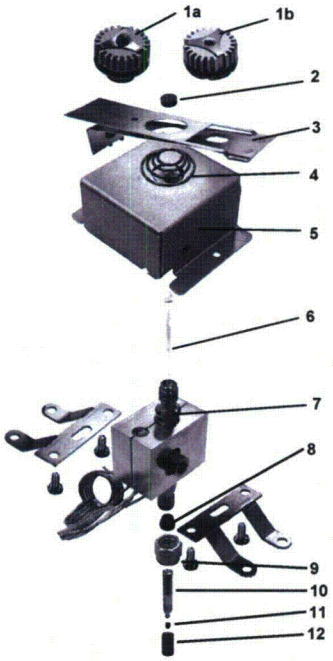
The automatic start switch is a spring loaded actuator that fits over and is aligned with the injection port of the FLASH Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only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

WARNING: BURN HAZARD

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

Injector Assembly and Insert

The FLASH Injector with insert and 530µm ID capillary column



1. a Injector Nut (392595501) Default (CP-8400/CP-8410)
   b Injector Nut (390812700)
   Injector Nut Wrench (390842300)
2. Septum, 9.5 mm
   BTO (lowest bleed, CR298705)
   Marathon (Autosampler, CR239188)
   Advanced Green 3 (general purpose, CR246124)
   Septum pick (7200008400)
3. Automatic Start Switch (390820601)
4. Spring
5. Cover and insulation
6. Glass Insert, default 392611943 More liners
7. Injector Body, EFC23 (392548301)
8. Ferrule,
   40% Graphite/60% Vespel (up to 400°C) (CR213400)
   Graphite (up to 450 °C) (CR211400)
9. Injector body Nut (SWSS4021)
10. Column Guide (392558301)
11. Column Ferrule, see table below
12. Column Nut 0.9 mm brass for capillary connections (394955100)
    Column Nut 1.6 mm brass for 1/16" connections (CP742351)
    Column Stainless Steel Nut (0.9mm) for High temperature applications (CP743117)

Figure 18: FLASH Vaporization Injector with Insert

Column ID	*Teflon* *Max 250 °C*	*Vespel* *Max 350 °C*	*40% Graphite 60% Vespel* *Max 400 °C*	*Graphite* *Max 450 °C*	*SilTite* *Metal, GC/MS*
0.53 mm ID	CR214108	CR212108	CR213108	CR211108	SG073302

Table 27: FLASH Column Ferrules 1/16" x 0.8 mm

FLASH Electronic Flow Control (EFC)

The Electronic Flow Control module used on a FLASH Injector can be type EFC23 or EFC24. The EFC23 is used to control the FLASH Injector under flow control, in order to set the constant column flow. A leak in the system would be indicated to the user by a drop in inlet pressure. The EFC24 is used to control the FLASH Injector in combination with Headspace Sampler or other devices that have a large carrier gas line.

Both EFC types are designed specifically for the FLASH Injector to support its various modes of operation. In simple terms it duplicates the behavior of the FLASH manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC23 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

The EFC23 is a constant flow system.

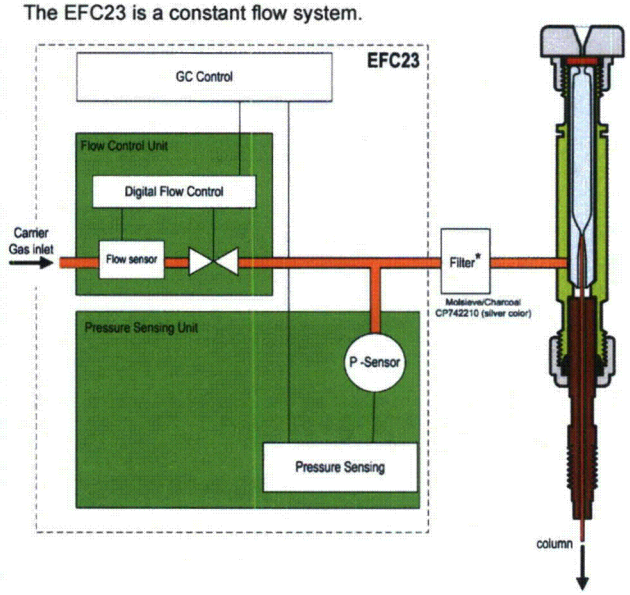


Figure 19: EFC23 and FLASH Flow Diagram

* See Pencil Filters on page 315

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module.
The EFC24 is a (constant) pressure system.

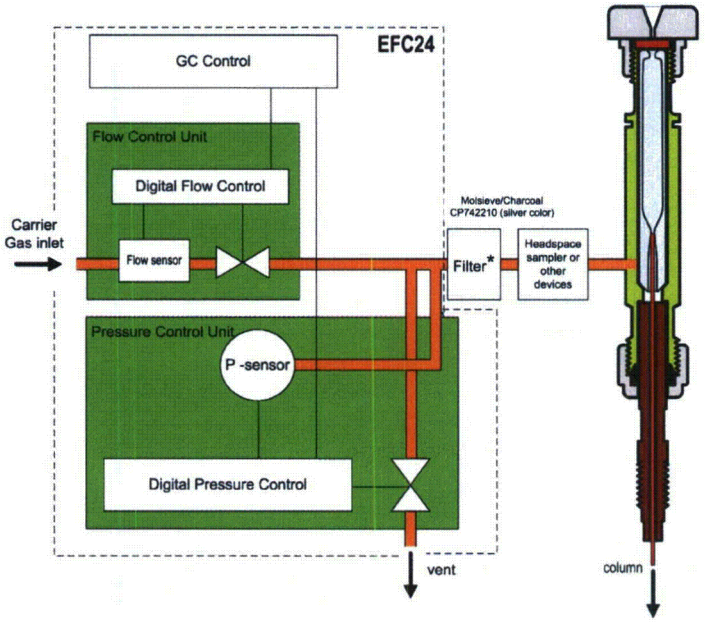


Figure 20: EFC24 and FLASH Flow Diagram

* See Pencil Filters on page 315

FLASH Injector Insert

Click here to see the FLASH Injector Insert, on page 328.
All FLASH Injector inserts are deactivated for maximum inertness.

The FLASH Injector contains a glass insert with a tapered section on the bottom. This allows a wide bore capillary column seal with the insert. The sample is vaporized in the insert and then swept onto the column. The use of glass wool in the insert allows the analysis of samples containing non-volatile components. The non-volatile material is trapped on the glass wool while the volatile components vaporize onto the column. The packed column kit for the FLASH also contains a glass insert, in this case the packed column buts up against the bottom of the insert.

Column Installation

The following instructions apply to installing a 0.53 mm ID capillary column in the FLASH Injector. Note that the thin polymeric coating on fused silica columns will give some protection against breakage; however, fused silica columns are somewhat fragile and must be handled with care. Alternative is to use Inert Steel 0.53 mm wide-bore columns.

Column Installation

The Connect Capillary Column to Injector procedure (available in the Installation Manual) describes the installation of a capillary column in a Bruker 436-GC/456-GC equipped with a FLASH Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning (available in the Installation Manual).

Column Installation In Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector (available in the Installation Manual).

Setting FLASH Gas Flow Rates

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

CAUTION

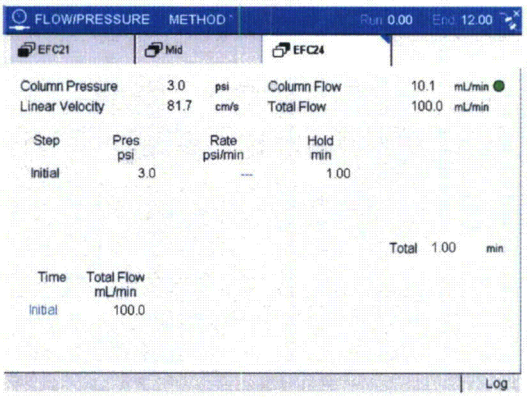
Do not heat the column oven above 50 °C without carrier gas flowing through the column. The column phase can be irreversibly damaged by exposure to oxygen at elevated temperatures. Allow 10 – 15 minutes to purge the column before heating.

EFC Pneumatics

Type EFC24 used with the FLASH Injector allows the user to set a constant column head pressure, build a pressure program, or emulate a constant column flow when the column is temperature programmed. With EFC23 equipped Injectors a flow range of 0 – 100 mL/min may be set from the 436-GC/456-GC display. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow value is desired, the pressure program needed to maintain constant flow is created on the EFC24 screen and derived whenever the method is loaded.

The FLASH is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessed via the FLOW/PRESSURE key on the 436-GC/456-GC display.

The next screen is an example if a type EFC24 method.



Flow Rates for Operation

Recommended flow rates differ, depending on the operating mode used. Refer to the appropriate paragraph on the next page for your operational mode.

Capillary Mode

When operating the Injector in the capillary mode, the following carrier gas flow rates are recommended:

Carrier Gas	*Column Flow Rate (Optimum)*	*Column Flow Rate (Typical)*
Nitrogen	1.5 mL/min	3 - 15 mL/min
Helium	2.4-4.0 mL/min	4 - 15 mL/min
Hydrogen	4.0-8.0 mL/min	8 - 20 mL/min

Table 28: Carrier Gas Flow Rates for FLASH Injector

Adjust the make-up gas flow to give a total of 30 mL/min.

Packed Mode

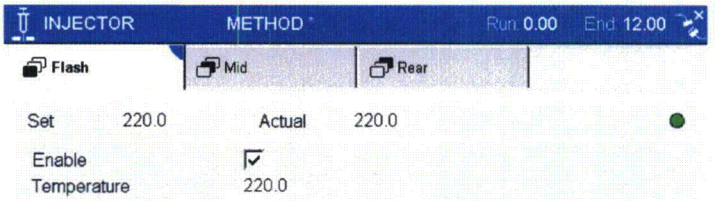
When operating in the packed column mode, adjust the carrier gas flow to applicable packed column flow rates (20 to 50 mL/min).

FLASH Injector Operation

This section contains the specific operating information and procedures required for optimum performance of the FLASH Injector. All installations for both the instrument and the Injector must be completed before continuing further.

FLASH Injector Method

The FLASH Injector method component on the 436-GC/456-GC display contains one screen for setting the required temperature for the Injector and viewing the status information. The FLASH Injector is an isothermal Injector. The temperature of the FLASH Injector must be optimized in order to have good sample evaporation in the liner.



Testing the FLASH Injector

The most effective method of testing Injector performance is by running a test sample. The following procedure describes how to test the performance of the FLASH vaporization Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The Table 46, on page 302 lists the series of test samples available for Bruker GC detectors.

To run one of these test samples, use the chromatographic conditions listed below. The detector should be operated at its most sensitive range.

Example chromatograms can be found in the detector section on page 302 of this manual.

Injector temperature	250 °C
Column temperature	50 °C initial, ramp to 250 °C at 20 °C/minute and hold for 5 minutes.
Injection Volume	1 µL

Table 29: FLASH Injector Chromatographic Testing Conditions

Observe the eluting peaks for symmetry, separation and elution time. Abnormally wide or skewed peaks, excessive elution times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.

The FLASH is mechanically similar to the PWOC except that it uses a glass insert for flash vaporization injection. Follow the same maintenance procedures for the FLASH that are outlined above for the PWOC, except in the case of changing or cleaning glass inserts.

Replacing the FLASH Glass Insert

After prolonged use, the FLASH glass insert may need to be removed for replacement with a new insert. Note that there are 2 different glass inserts, one for use with wide bore (0.53 mm ID) capillary columns and one for use with packed columns. These inserts are not interchangeable.

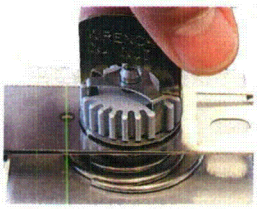
1. Wide bore glass insert, Part Number: 0392611943.
2. Packed column glass insert, Part Number: 0392611944.

See replacement parts on page 328.

Remove the Glass Insert

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

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



WARNING: BURN HAZARD

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

CAUTION

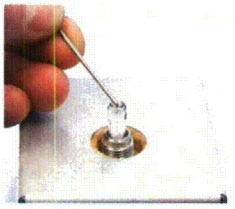
Before proceeding any maintenance procedure; extinguish the detector flame (if any) and cool down detector and oven temperature to <50 °C.

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

2. With tweezers or a septum pick, lift the edge of the septum. Remove the septum.

Replace the Injector septum each time the glass insert is replace.

3. Use tweezers or a septum pick to grasp the glass insert and remove it from the Injector.



Replace the Glass Insert

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

1. Pick up the new insert with tweezers and place it in the Injector carefully.
2. Use tweezers to place a new septum in the Injector.

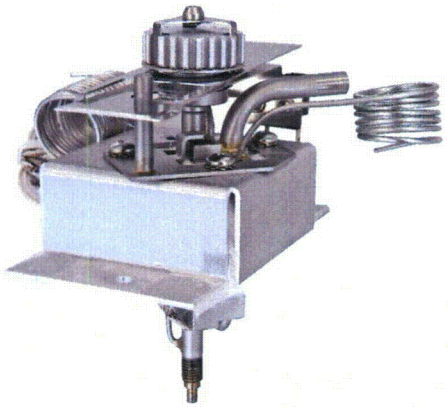
If the septum has a Teflon face, place the Teflon face toward (down) the column.

3. Place the Injector nut on the Injector and tighten by hand until you feel some resistance, then tighten an extra 1/2 to full turn using the Injector nut wrench.

After the Injector nut has been replaced check that the head pressure (EFC status on display or manual pressure gauge on pneumatics panel) increases to its normal value. If this does not happen there is an indication of a leak.

Condition the insert by setting the FLASH Injector temperature to 300 °C and allowing the system to condition for 30 minutes.

COC Septum-Equipped Programmable Injector (SPI)



The Bruker COC Injector gives optimum performance for non-vaporizing (cold) injection into conventional fused silica capillary columns (0.32-0.53 mm ID columns).
It incorporates several unique design features to produce improved peak resolution, quantitation and versatility over other cold on-column Injectors.

The result is not only a capillary on-column injection system ideally suited for analyses that cannot be done with conventional split/splitless injection, but one that gives highly accurate and precise results with a wide variety of trace to semi-trace level samples.

The COC Injector can replace manually operated vaporizing capillary Injectors in many cases and may provide improved quantitation of labile and high molecular weight samples.

Temperature-programmed, cold injection: The Injector is held at a temperature 20 °C to 30 °C below the solvent boiling point during injection and then is rapidly temperature programmed up to the final column temperature.

The oven is started at the same temperature as the Injector. Conventional temperature programmed analysis is carried out in the GC column.

Frequently, good results may be obtained and the run time may be reduced by injecting into a cold Injector but with the column at 5°C above the solvent boiling point. In this case, the column is held at the initial temperature until the Injector is at its final temperature.

Cold injection reduces or eliminates many of the problems of conventional vaporizing split/splitless injection, such as molecular weight discrimination (loss of high and low boiling substances), thermal decomposition of the sample and sample adsorption onto hot, active Injector surfaces. These problems are largely eliminated in cold injection because the sample is directly introduced into the column as a liquid. In the split/splitless Injector, the sample is first vaporized and then allowed to pass into the column, where it may be condensed back into its liquid state.

The COC Injector may also be used as an isothermal, vaporizing Injector. However, best performance is achieved using the temperature-programmed mode. The system consists of three major components: a glass insert, the heated Injector body and a standard syringe. Maximum syringe needle diameter is 0.019" (0.49 mm) or 26 gauge.

All heated parts and all parts in direct contact with the carrier gas stream are constructed of stainless steel, borosilicate glass, fused silica, silicone rubber (septum) and Inconel X750.

The Injector body is mounted on an insulated enclosure. The Injector heating rate is adjustable to allow optimization for thermally sensitive samples. A purge exit is provided for bake-out and septum purge. The internal dead volume of the Injector body has been minimized.

In operation, the fused silica column is inserted up into the Injector body from the column oven and seated in the glass insert. The column is sealed to the Injector with conventional polyimide/graphite or graphite ferrules. The syringe needle enters the glass insert from the top. After the syringe is withdrawn, the entire injection zone is rapidly heated, driving the sample into the column where the peaks are sharpened by the solvent effect and cold trapping. Significant peak broadening occurs (up to 2X) in Injectors without this rapid heating step, especially when larger samples and high-boiling samples are used.

Of the 2 internal parts in the Injector body, the septum support and glass insert require periodic removal for cleaning. A biweekly check is recommended during regular use. The need for cleaning will vary with different samples and the glass insert is easily replaced in case of non-removable deposits or breakage.

The COC Septum-Equipped Programmable Injector (SPI) is factory-installed or available as a field upgrade (must be installed by trained personnel).

Automatic Start Switch

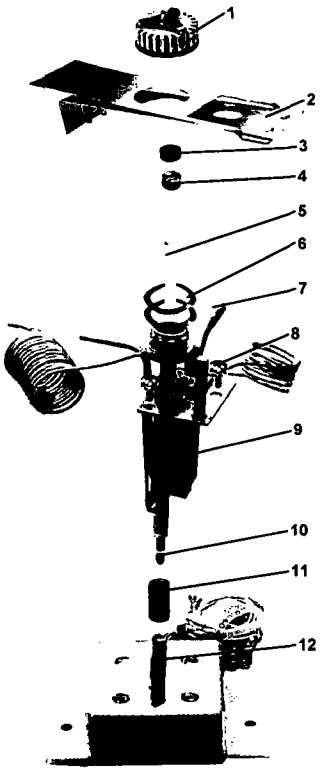
The automatic start switch is a spring loaded actuator that fits over and is aligned with the injection port of the FLASH Injector nut. The GC run is started when the actuator is depressed by the syringe barrel, or manually pressed at the moment of sample injection. The GC run can also be manually (only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

WARNING:
BURN HAZARD

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

Injector Assembly and Insert

The COC Septum-Equipped Programmable Injector (SPI).



1. Injector Nut, (3949666601)
   Injector Nut Wrench, (390842300)
2. Automatic Start Switch (390820601)
3. Septum, 7/16"
   BTO CenterGuide™, (CR298777)
   Marathon (Autosampler, CR239787)
   Advanced Green 3 CenterGuide™ , CR246725)
   Septum pick (7200008400)
4. Septum Support, (391821100)
5. Glass Insert, default SG092034 More liners
   High performance (190010906)
   On-Column (190010907)
   Packed (392611950)
6. Wave spring washer 2x, (1492000500)
7. Coolant exit, Frit Restrictor (3600004601)
   Plug, 1/8" tube (2824707101)
8. Screws 3x, (391866306)
9. Injector Body
10. See table below
11. Column Nut 0.9 mm brass for capillary connections (394955100)
    Column Nut 1.6 mm brass for 1/16" connections (CP742351)
    Column Stainless Steel Nut 0.9 mm for High temperature applications (CP743117)
12. Heater/probe 120 Vac, (391833400)
    230 Vac, (391833401)

Figure 21: COC Injector Assembly

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

Table 30: Column Ferrules 1/16"

COC Injector Parts

Click here to see the COC Injector parts on page 329.

COC Electronic Flow Control (EFC)

The Electronic Flow Control module used on a COC Injector can be type EFC23 or EFC24. The EFC23 is used to control the COC Injector under flow control, in order to set the column flow. A leak in the system would be indicated to the user by a drop in inlet pressure. The EFC24 is used to control the COC Injector in combination with Headspace Sampler or other devices that have a large carrier gas line.

Both EFC types are designed specifically for the COC Injector to support its various modes of operation. In simple terms it duplicates the behavior of the COC manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection. The type EFC23 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

EFC23 is a constant flow system.

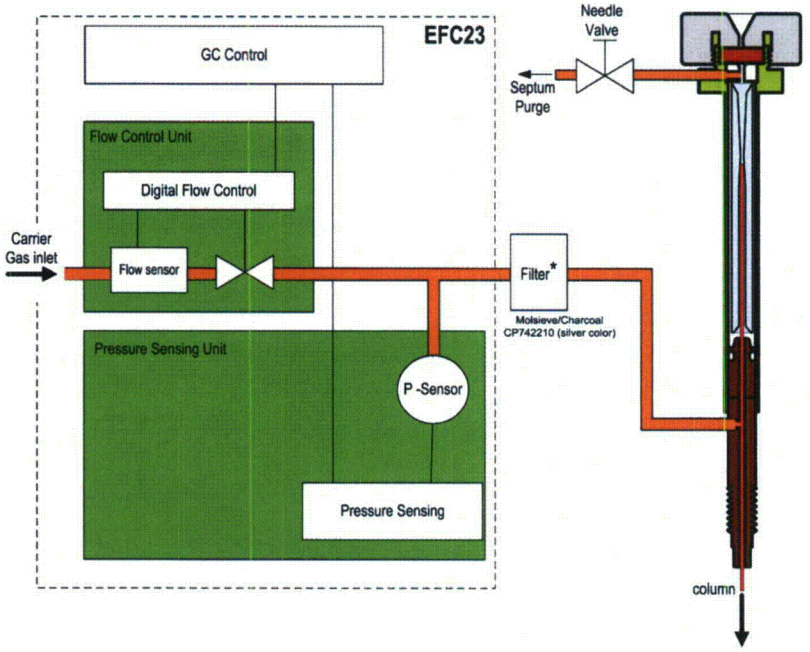


Figure 22: EFC23 and COC Flow Diagram

* See Pencil Filters on page 315

The type EFC24 flow diagram shown below is an indication of the control mechanism of this type of EFC module.

EFC24 is a (constant) pressure system.

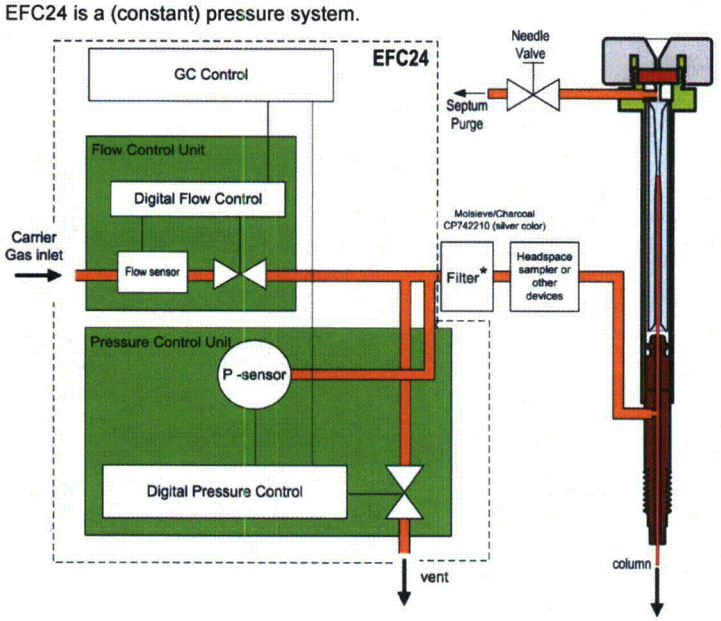
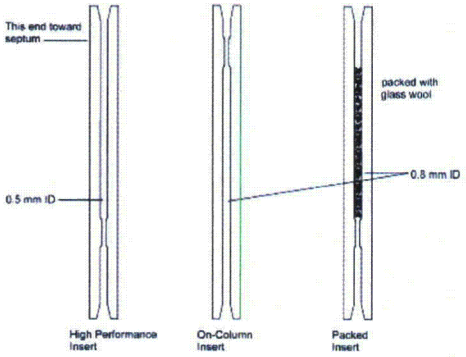


Figure 23: EFC24 and COC Flow Diagram

* See Pencil Filters on page 315

Glass Insert

Different glass inserts are available for use in the COC Injector.
Click here to see all inserts of the COC Injector parts on page 329
The standard insert is the high performance (P/N: 190010906, only columns up to ID=320um).



The high-performance insert (P/N: 190010906) is used when the presence of glass wool is unacceptable. The small internal diameter of this insert provides efficient transfer of the liquid sample from the syringe. If inserts with larger internal diameters are used without any packing, then sample transfer from the syringe may not be quantitative.

The on-column insert (P/N: 190010907) is used for on-column injection into large bore (0.53 mm ID) columns.
The glass wool packed insert (P/N: 392611950) provides efficient transfer of the liquid sample from the syringe and traps any contaminants present in the sample.

Syringe

The required syringe needle length is 2.00" (excluding the hub) for both manual injections and used in the CP-8400 and CP-8410 AutoSamplers.

Mounting the Column

During operation, the fused silica capillary column hangs on the capillary column holder in the column oven.
Hang the coiled column on the capillary column holder, then prepare the column end and complete column connections to both the Injector and detector fittings, as detailed in the following paragraphs.

Column Installation

The Connect Capillary Column to Injector procedure (available in the Installation Manual) describes the installation (or reinstallation) of a capillary column in a Bruker 436-GC/456-GC equipped with a Bruker COC Injector. Each step of the procedure is described in detail.

Condition the Column

For conditioning the capillary column see Column Installation and Conditioning (available in the Installation Manual).

Column Installation in Detector

For connecting a capillary column into a detector, see Connect Capillary Column to Detector (available in the Installation Manual).

Connecting CO_2 to the COC Injector

The recommended tank for use with the COC Injector is a size 1A cylinder containing approximately 60 pounds of "bone dry" liquid CO_2. The tank should be equipped with a liquid eductor tube. Special fittings provided in the Start-Up Kit (P/N: 0391833590) for the Injector are required for connection to the CO_2 tank. Refer to figure below.

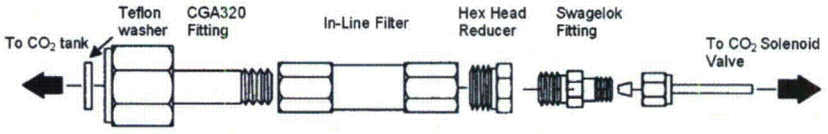


Figure 24: Connection CO_2 tank to GC

Approximately 100 chromatographic runs can be made per tank of CO_2 when the Injector is cooled to -50 °C for each run. Cooling to higher temperatures will consume less coolant per run. The rate of use of CO_2 is dependent on the operational conditions, thus your actual use may vary from this estimate.

1. Use 1/8" copper or stainless steel tubing for the connection between the CO_2 solenoid valve and the CO_2 tank. **NOTE: If stainless steel tubing is used, change all fittings to stainless steel.**

2. Connect one end of the 1/8" Swagelok fitting to the GC and the other end of the tubing to the in-line filter and CGA 320 adapter. All hardware is supplied in the Start-Up Kit.

3. The CGA 320 fitting will be connected to the CO_2 tank. A Teflon washer is fitted in the nut that will connect to the tank and it must be inspected. Check that the washer is in good condition. If it is not, replace that washer and then connect the CGA 320 fitting to the tank. **NOTE: Do not over tighten the CGA 320 fitting. A high torque is not required, as the Teflon washer is the seal.**

Connecting LN2 to the COC Injector

When connecting LN_2 to the COC Injector, Neoprene insulation tubing (P/N:2400046700) must be installed over the entire length of tubing from the solenoid valve to the tank, to prevent ice build up and condensation. Consumption of LN_2 is approximately 0.5 to 1.0 pounds per run when the Injector is cooled to -50 °C for each run. Cooling to higher temperatures will consume less coolant per run. The rate of use of LN_2 is dependent on the operational conditions, thus your actual use may vary from this estimate.

Full tank pressure is directed to the GC and the tank regulator should be less than 50 psi for stable operation.

WARNING

LN2 tanks that are designed for pressures higher than 50 psi must not be used with in-line regulators, shut-offs, or restrictors due to high pressure build-up. In all cases, never limit the tank's built-in venting system.

Compressed air at 40 psig may be used to cool the Injector using the LN_2 option; however, it is not recommended due to the low efficiency of cooling with air. If operation above 50 °C is acceptable, then air may give satisfactory cooling rates.

Setting COC Gas Flow Rates

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

CAUTION

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

EFC Pneumatics

The Electronic Flow Control module used on a COC Injector can be type EFC23 or EFC24.

The EFC23 is used to control the COC Injector under flow control, in order to set the column flow. EFC23 sets a required carrier flow into the Injector/Column system. A leak in the system would be indicated to the user by a drop in inlet pressure.

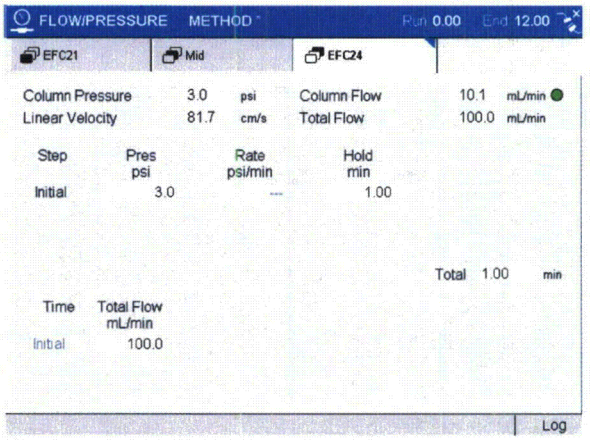
The EFC24 is used to control the COC Injector in combination with Headspace sampler and other devices that have a long carrier gas line.

Both EFC types are designed specifically for the COC Injector to support its various modes of operation. In simple terms it duplicates the behavior of the COC manual pneumatics system in that there is an inlet mass flow controller supplying carrier gas to the Injector and a pressure control valve downstream from the Injector which sets the Injector pressure. As Injector pressure determines the rate of carrier gas flow through the column, this pressure is monitored close to the point of injection.

Type EFC23 or EFC24 used with the COC Injector allows the user to set a constant column head pressure, build a pressure program, or set a constant column flow. A pressure program is typically used to maintain the column flow at a constant value while temperature programming the column oven. When Constant Flow Programming is enabled, the pressure program needed to maintain constant flow is derived whenever the method is loaded.

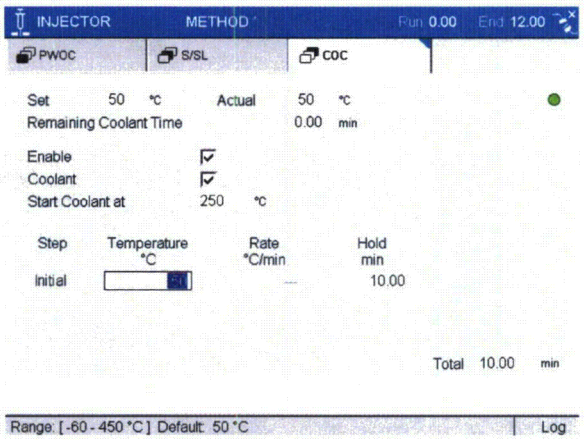
The COC is a pressure-controlled Injector; thus the column flow decreases with increasing column temperature if the pressure remains constant. EFC method parameters and status are accessible via the FLOW/PRESSURE key on the 436-GC/456-GC display.

The screen is an example if a type EFC24 used.



COC Injector Operation

This screen contains the specific operating information and procedures required for optimum performance of the COC Injector. All installations for both the instrument and the Injector must be completed before continuing further.
The COC Injector method component on the 436-GC/456-GC display contains one screen for setting the required temperature, Injector temperature rate and hold time can be time programmed for the Injector.



Testing the COC Injector Performance

The following procedure describes how to test the performance of the COC Injector. This is best done with a test sample containing an appropriate set of components for the installed detector. The following table lists the series of test samples available for 436-GC/456-GC.

Test Sample	*Part Number*	*Concentrations of Test Compounds*
TCD	8200504801	3.00 µg/µL of C_{14}, C_{15} and C_{16} in iso-octane.
ECD	8200504802	33.0 pg/µL of lindane and aldrin in iso-octane.
PFPD	8200504803	20.0 ng/µL of n-dodecanethiol, tributylphosphate, methyl parathion; 4000 ng/µL of n-pentadecane in iso-octane.
NPD	8200504804	2.00 ng/µL of azobenzene, methyl parathion; 4.00 ng/µL malathion and 4.00 µg/µL C_{17} in iso-octane.
FID	82005048-07	30 ng/µL of C_{14}, C_{15} and C_{16} in iso-octane.
Note: *If the FID test sample is not available, the TCD test sample can be used if first diluted 100:1.*		

Table 31: Detector Test Samples

To run one of these test samples, use the chromatographic conditions listed previously for the injection technique you are currently using. The detector should be operated at the most sensitive range, e.g., 12 for FID and NPD, 0.05 for TCD, 10 for PFPD and 1 for ECD. The resultant chromatogram on page 302 should approximate that shown in the detector section of this manual.

Observe the eluting peaks for symmetry, separation and retention time. Abnormally wide or skewed peaks, excessive retention times, abnormally small peaks and noisy or drifting baselines indicate faulty performance.
When the instrument is installed a definitive indication of instrument performance can be obtained by running a test chromatogram for the detector(s) that will be used with the Injector. This initial test chromatogram should be retained as a standard of comparison for later checking the instrument if change in detector sensitivity is suspected.

Maintenance

Before maintaining the COC Injector please read the common Injector/septum maintenance information in the section Maintenance,on page 312.

Septum Replacement

Septum replacement represents the major part of routine chromatographic maintenance. Septum damage from the needle penetrations can be avoided by injecting into the same hole and not using syringes with needles having burrs or bends at the tip which cut the septum.

Handling a septum with bare fingers may result in column contamination. Use tweezers when installing a new septum.

Follow steps 1 through 7 below for replacing the septum in the Injector. Be sure to cool all heated zones to less than 80 °C before opening the septum nut and decrease the column head pressure to less than 2 psig. If pressure is too high, the septum support and glass insert may pop up, rather than remain in their correct positions.

Removal of Glass Insert

Use care when removing or replacing the glass inserts for the COC Injector.

WARNING:
BURN HAZARD

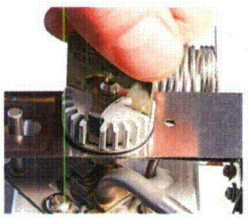
The Injector nut may be hot. Lower the Injector temperature to 50 °C and permit the Injector nut to cool before proceeding.

CAUTION

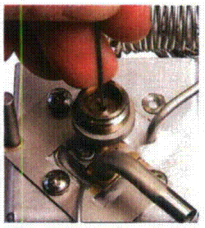
Before proceeding any maintenance procedure; extinguish the detector flame (if any) and cool down detector and oven temperature to <50 °C.

Removal

To avoid contaminating the capillary system, ALWAYS use tweezers or the extraction tool to handle the Injector internal parts.



1. Use the Injector nut wrench to remove the Injector nut and place on a clean, uncontaminated surface, such as a fresh cleaning tissue.
2. Remove the Injector switch and the 2 wave spring washers.
3. Using tweezers or the extraction tool (P/N: 7200008400) supplied in the Accessory Kit, remove the septum, septum support and glass insert. Place these parts on a clean, uncontaminated surface.

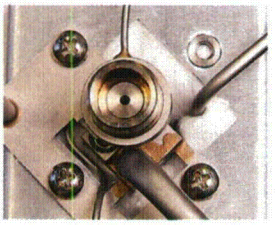


4. If the glass insert does not pop up when the septum support is removed, it may be necessary to push up on the column from the column oven. The polyimide on polymer-coated fused silica columns often forms a very tight seal to the glass insert. This seal must be broken before the insert can be removed.

Re-assemble

Install a new glass insert by following steps 5 through 8.

To avoid contaminating the capillary system, ALWAYS use tweezers or the extraction tool to handle the Injector internal parts.



5. Place the insert into the Injector body.
6. Install the septum support over the insert. The line on the septum support **must not be visible!**
7. Using tweezers, place a new high temperature septum over the septum support. If a Teflon-coated septum is used, the Teflon side must face down.
8. Place the Injector switch and the 2 wave spring washers and nut on the Injector and tighten until resistance is felt, then tighten an extra 1/4-turn.

Leak Checking

Leak checking methods used to leak check the COC Injector are presented in order of overall sensitivity and preference.

Leak Checking the Injector Only

1. Remove the (test) column and plug the Injector outlet with a 1/16" no-hole ferrule (P/N: 2864950300). Turn Injector nut clockwise until it comes to a stop. Turn off the septum purge.
2. Pressurize the Injector to 30 psig with carrier gas. Turn off the carrier gas flow at the ON-OFF valve.
3. The pressure should hold for 30 minutes, as shown on the status screen. A pressure drop less than or equal to 1 psig per minute at 30 psig is acceptable. Locate leaks with an appropriate leak detector.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

4. An alternate way to leak check the Injector only is to have the column installed in the Injector end and the opposite column end sealed.

Gas Leak Checking

A convenient and non-contaminating way to leak test fittings and connections after installation or hardware replacement procedures is to direct a small jet of gas (butane from a disposable lighter is recommended) at the point to be tested, then use the detector, at maximum sensitivity, to detect leakage of gas into the system. Use normal column flow, a cool oven and an operational detector. If a peak is detected in excess of the magnitude given in table below, repair the leak.

Detector	*Gas*	*Attenuation, Range*	*Maximum Peak Size*
FID	Butane	16×10^{-12}	30%
ECD	Dichloromethane	2 x 1	50%
PFPD, Sulfur Mode	Natural gas containing Methanethiol	2×10^{-10}	50%
NPD	Dichloromethane	2×10^{-12}	2%

Table 32: Detectable Gas Leak Checking

Visual Leak Checking

The least preferred method of leak checking is to place a drop of pure solvent (isopropyl alcohol is recommended) on the suspected fitting or connection and look for bubbles.

Commercial soap type leak detection fluids should not be used at any point in a capillary system, since, if a leak is present, the fluid will penetrate and contaminate the system. Column performance will be degraded and a substantial period of time may be required to achieve a clean system.

Syringe Leak Checking

In some instances, non-reproducible chromatographic responses can be attributed to a worn and/or leaky syringe. The syringe must be leak tight. To check the syringe, insert the needle in an Injector operating at 20 to 30 psi (150-200 kPa) with a no-hole ferrule. Place a drop of solvent at suspect leak locations and look for bubbles while moving the syringe plunger up and down.

Injector and Column Conditioning

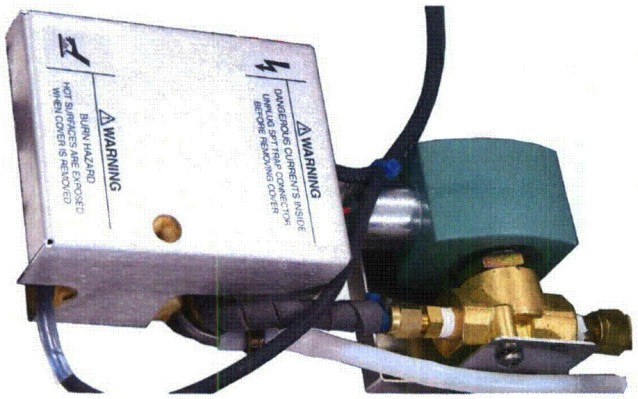
Initial bake out of a factory-installed Injector or test column is not necessary. If bake out becomes necessary due to contamination from handling, etc., follow the procedures or recommendations in the following paragraphs.

Baking Out the Injector

It may be necessary to bake out the Injector if it has become contaminated during the re-assembly process, i.e. if the internal parts were placed on dirty surfaces or touched with bare hands. Then, both the Injector and column should be conditioned.

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

1. Disconnect column from Injector.
2. Fully open the septum purge.
3. Install a no-hole ferrule in the capillary column nut. Thread the nut up onto the assembly and tighten.
4. Set flow between 100 and 200 mL/min. Measure flow at the purge line exit, then remove the no-hole ferrule from the column nut.
5. Set the Injector temperature to 450 °C.
6. Bake out the Injector for not less than 4 hours, to a maximum of 16 hours at a time.
7. Cool the Injector to 50 °C and then install the capillary column.
8. Replace the septum.
9. Readjust the septum purge to the desired flow rate.



Description

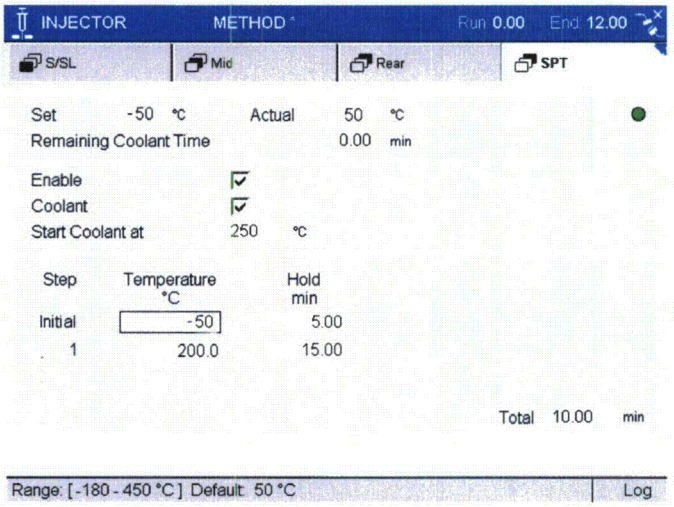
The SPT is a concentrating system for the determination of trace level volatile organic components in air and gaseous matrices. Integrated into the top of the GC, its design provides the following features:

- Fast heating rates.
- Very low cryogenic consumption.
- Short and long trap length options.
- Liquid carbon dioxide (LCO_2), liquid nitrogen (LN_2) cryogenic cooling.

Trap heating rates of up to 40 °C/sec provide fast injection from the trap to the capillary column. This helps to ensure good chromatographic performance without the need of an isolation valve, even for low boiling analytes.
Fast injection also allows higher initial column oven temperatures, thereby reducing or eliminating the need for column oven cryogenics. Cryogenic usage for trap cooling is minimized because of the compact design of the SPT and the specially designed algorithm that controls the cooldown and temperature of the SPT.

SPT temperature zone

The SPT temperature zone is independently controlled by a heated zone of the 436-GC/456-GC for setting the trapping temperature and up to 2 desorption programs. Because of the trap's fast heating rates, there is no rate parameter setting for the desorption programs.



Each SPT option includes a SPT valve oven, installed either for operation with 2 gas switching valves or with one valve and one Injector. The SPT valve oven is controlled by the 436-GC/456-GC heated zone and provides a constant temperature environment for the trap ends and tubing connecting them to an SPT valve.
The SPT is assigned to heated zone 3 only.

The multi-valve oven may be installed on the 436-GC/456-GC for SPT systems which require more than 2 valves.
See building an SPT method on page 182 for more details.

Two trap sizes available

Two trap sizes and various packing materials are available:

1. The short trap is generally used for cryogenic trapping on glass beads using LN_2 or for highly adsorbing trap materials.
2. The long trap length is suitable for trapping with adsorbents such as Tenax or charcoal-based materials at ambient or near ambient trapping temperatures where LCO_2 cryogen may be used.

The following are nominal measurements for the 2 trap sizes:

Trap Size	*Active Bed Length*	*Total Length*
Short	6.8 cm	17.02 cm
Long	29.2 cm	39.47 cm

Using the SPT

Trapping Methodology

There are fundamentally 2 methods of preconcentrating volatile organics: cryogenic trapping and adsorbent trapping. Cryogenic trapping takes place on an inert material, such as glass beads, at a very low temperature. For example, hazardous air pollutants with boiling points down to -30°C can be preconcentrated on glass beads at -160°C using LN_2.

The other trapping method uses adsorbents, such as Tenax, charcoal or other carbon-based materials, porous polymers, or coated column packings, alone or in various combinations at near ambient trapping temperatures. For example, the same pollutants mentioned above can be trapped on a Tenax/Charcoal, Carbopack B, or Vocarb 4000 (Supelco) packed trap at ambient temperatures.

Trapping Media

Several trapping media were mentioned above. In general, the least active medium that will still provide adequate capacity (retention volume) for the components should be used. This will keep the desorption temperature at a minimum and allow faster release of the analytes from the trap to the column.

Trap Dimensions

Once the trapping material is chosen, the trap size (long or short) should be selected based on the volume of the sample to be passed through it and the trapping temperature (all SPT traps are made from 1/8" o.d., type 316 stainless steel tubing). Most traps should be able to accommodate samples of approximately 400 milliliters at flow rates of about 40 mL/min in order to obtain the detection levels required.

Trap Temperature

The temperatures required for cryogenic trapping on glass beads are easy to determine. As noted above, a trap temperature of -160 °C has been used for air toxics. When preconcentrating even lower boiling analytes, such as the C_2-hydrocarbons, trap temperatures as low as -180 °C are necessary. When using adsorbents, the trapping temperature is determined by the breakthrough volume of the sample. Lower activity adsorbents require lower temperatures.

Trapping and Desorption

The flow rate during trapping is generally from 10 to 40 mL/min. Trapping is always more efficient at lower flow rates. Flow rate during desorption depends on the analytical system to which the analytes are passed. Desorption flow rates of 5 mL/min or greater are compatible with 0.53 mm i.d. columns and usually produce narrow peaks for early eluting components. When the capillary column i.d. does not allow this high flow, fast desorption flow rates in combination with inlet splitters may be used.

To improve peak shape and quantitation in cases where narrow bore column systems are in use but detectivity requirements prohibit the use of split inlet systems, column oven cryogenics will be required. This allows refocussing of early eluting components.

Electrical System

The SPT option provides independent temperature control of 2 parts of the trap assembly:

1. Heating and cooling of the trap.
2. Heating of the trap ends.

Trap temperature programming is accomplished in 436-GC/456-GC heated zone 3, controlling heating and cryogen cooling of the trap. The SPT trap is mounted on the SPT valve oven, which maintains the SPT trap ends at a constant temperature. The oven is powered by one of the other remaining heated zones.

Extremely fast heating of the trap is made possible by utilizing the wall of the trap tubing as an electrical heating element. This eliminates the extraneous thermal mass associated with conventional heater cartridges. To match the electrical resistance characteristics of the trap tubing, a specific electrical voltage and current are provided by the SPT transformer (mounted in the rear of the GC). To provide the required voltages, the SPT trap harness connectors for the short and long trap are different. Although low voltages (less than 8 volts AC) are used to heat the trap, the SPT transformer is designed to deliver very high currents.

The trap ends carry dangerous currents. Do not put any conducting material, such as wrenches, screwdrivers, or metal tubing, across the trap ends. Do not wear metallic jewelry while working near the trap.
Variations in line voltage supplied to the GC will have an effect on the heating rate while the trap is desorbing.

Circuit breaker

The circuit breaker reset button is located above the SPT transformer on the rear panel of the GC. This pop-out button protects the SPT from abnormal power usage by interrupting the electrical connection to the trap. Wait at least 5 minutes after the circuit breaker is tripped before resetting it.

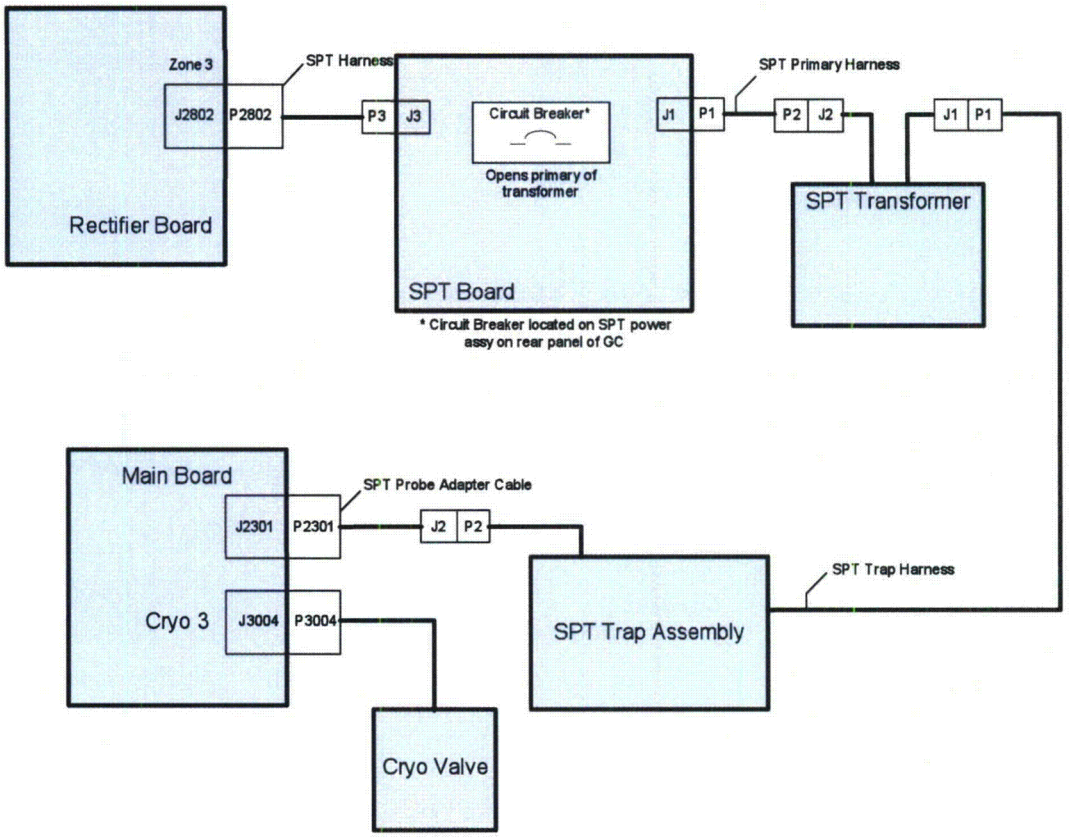


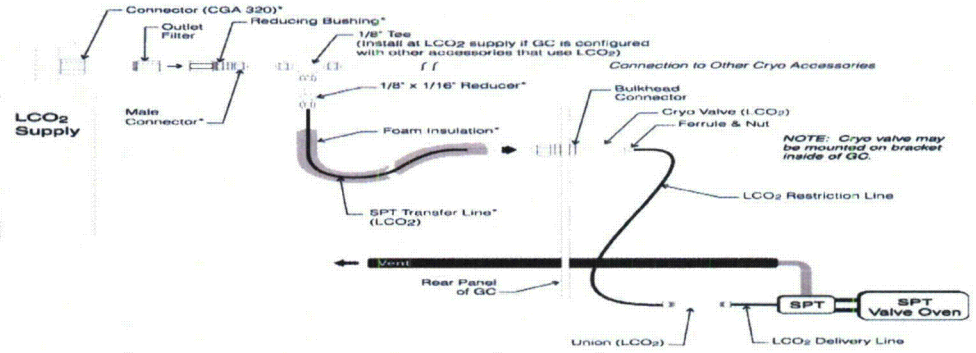
Figure 25: SPT Electrical Diagram

LCO_2 Option

System Description

The SPT-LCO_2 option allows trapping temperatures as low as -60 °C. This system is very efficient and consumes a minimum amount of LCO_2.

Because of low coolant consumption, conditions that affect thermal efficiency of the coolant transfer system are very important to consider.



*Included with SPT Accessory Kit (LCO_2 Option)

Figure 26: LCO_2 Coolant Flow Diagram

Theory of Operation

As LCO_2 expands into the gas phase in the SPT cryo Enclosure, the latent heat of vaporization provides cooling power. The coolant flow rate is regulated by varying the pulse rate of the cryo solenoid valve.

The latent heat of vaporization drops rapidly as the temperature increases to the CO_2 critical point of 31°C. In order for the SPT to have adequate cooling power, LCO_2 should be delivered to the cryo valve at 27°C or cooler.

Characteristics of LCO_2 as a Coolant

The coolant for this option is liquid phase carbon dioxide (LCO_2) supplied from a pressurized cylinder[6]. At pressure, this cylinder contains both liquid and gas phases coexisting at equilibrium. The cylinder pressure is then equal to the saturation pressure and is dependent on the supply temperature. As the cylinder temperature rises, the cylinder pressure rises. Thus if a constant temperature is provided a constant pressure is maintained. This is true until the liquid phase CO_2 is depleted. For this reason it is advisable to keep the cylinder at a fairly "full" condition. Do not let it drop more than 60% of its weight.

Inadequate Cooling

To insure reliable cooling power and trapping temperature stability, the CO_2 must be delivered as liquid phase CO_2. When cooling power is lost, the CO_2 is often being delivered as gas phase. This may be caused by 2 different conditions.

1. CO_2 supply is too low. There is insufficient CO_2 remaining in the cylinder to build supply pressure to the saturation pressure.

2. The CO_2 has vaporized in the transfer line because the temperature of the transfer line is higher than that of the supply. The supply temperature must be equal to or warmer than the transfer line.

Locating the LCO_2 supply cylinder near the GC in a temperature controlled room is ideal.

Transfer Line

The SPT option comes with a 3 meter long, small diameter, low volume transfer line. The supply cylinder must be located then, less than 3 meter from the GC.

The low volume feature of the transfer line helps this option tolerate situations where the GC and transfer line are slightly warmer than the supply cylinder. Keep the transfer line cool and stable by insulating it with foam insulation provided in the Accessory Kit.

Recommended Supply Conditions

Supply	High pressure cylinder with siphon tube.
Supply Conditions	Temperature 15°C to 27°C (740 psia to 975 psia)
Outlet Filter	15 micron particulate filter (accessory kit) installed at the cylinder connection.
Transfer Line (from LCO_2 cylinder to the GC)	A 3 meter long, 1/16" dia. Stainless steel tube is included with this option. Install as a dedicated line from supply to SPT. Insulate with foam pipe insulation.

[6] The critical point of CO_2 is 31 °C

LN_2 Option

Description

The SPT - LN_2 option allows trapping temperatures as low as -190°C. For best utilization of the high performance features of the SPT, it is recommended that the transfer line from the LN_2 source to the GC be very efficient. The LN_2 transfer line should have the following characteristics:

- Minimal length.
- Should not have excessive restriction to flow.
- Low thermal mass.
- Well insulated.

In addition, the transfer line should not be routed near heat sources that could reduce the cooling efficiency, such as the column oven exhaust fan.

If the SPT system is at ambient temperature (e.g., 23° C) the time required for initial cooling of the SPT from 50 to -190° C might be longer (approximately 5-10 minutes) than subsequent cooling cycles.

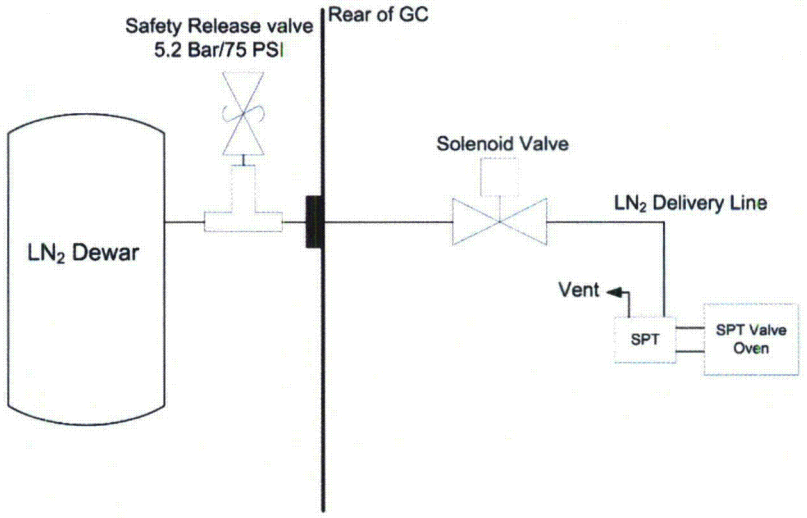


Figure 27: LN_2 Coolant Flow Diagram

Recommended Supply Conditions

Due to high pressure build-up, LN2 tanks that are designed for pressures higher than 50 psi must not be used with in-line regulators, shut-offs, or restrictors. In all cases, never limit the tank's built-in venting system.

Supply	LN_2 tank configured for liquid delivery.
Supply Pressure	19 to 25 psig
Transfer Line (from LN_2 tank to GC).	1 to 3 meters length of (0.190" i.d.) ¼" o.d. copper tubing, insulated with foam insulation tubing.

Recommended temperature ranges

Parameter	*436-GC/456-GC Range*	*Recommended*
Trapping Temperature	-60 to 450 °C -180 to 450 °C	LCO2 - 60 to 50 °C LN2 - 180 to – 40 °C
Desorption Temperature Program 1	-180 or -60 to 450 °C[7]	50 to 420 °C
Desorption Temperature Program 2	-180 or -60 to 450 °C[8]	50 to 420 °C
SPT Temperature Limit	-60 to 450 °C -180 to 450 °C	--
SPT Valve Oven	--	150 to 250 °C

Table 33: SPT recommended operating ranges

No cryogen is used for trap temperature settings ≥ 50 °C; no heat is applied to the trap at trapping temperatures < 0 C.

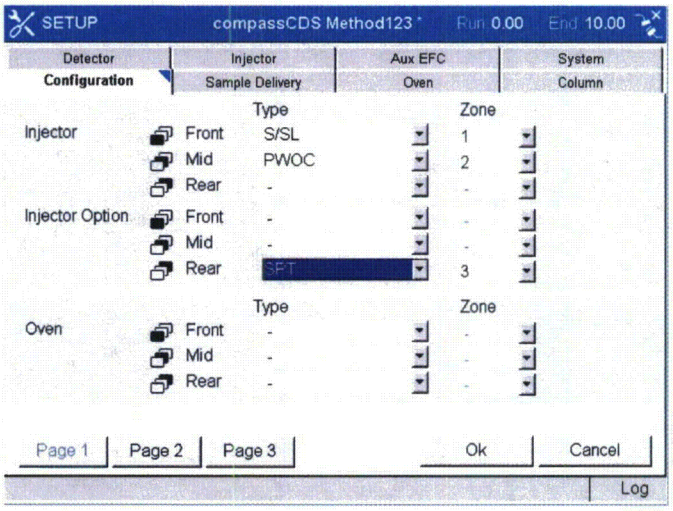
It is advisable to purge adsorbent-containing traps by using an inert gas (N_2 or He). This procedure prevents inadvertent oxidation of the adsorbent by accidental heating of the trap.

[7] Trapping temperature range is determined by cooling type

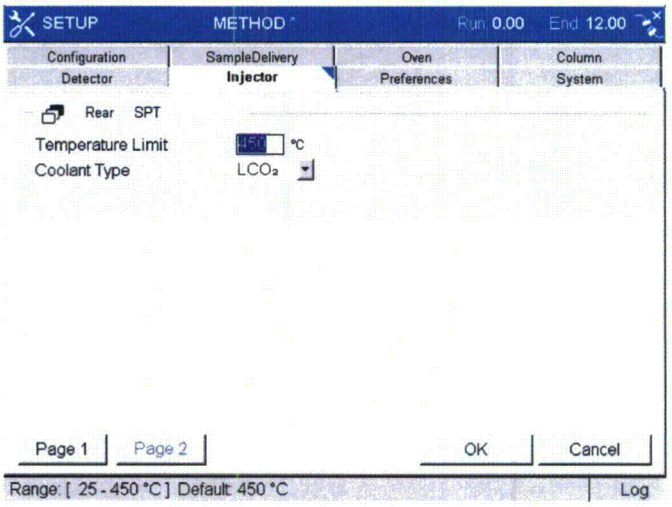
[8] In an automated sequence the 60 seconds delay helps to recover from FID flame faults and avoids having the fault again in the next run. It also prevents a workstation starting a run while FID ignition is still going on.

Configuring the SPT

The SPT is typically factory installed and should be already configured when the 436-GC/456-GC is delivered. To check the SPT configuration press the setup button. The SPT **must** be installed in **zone 3**.



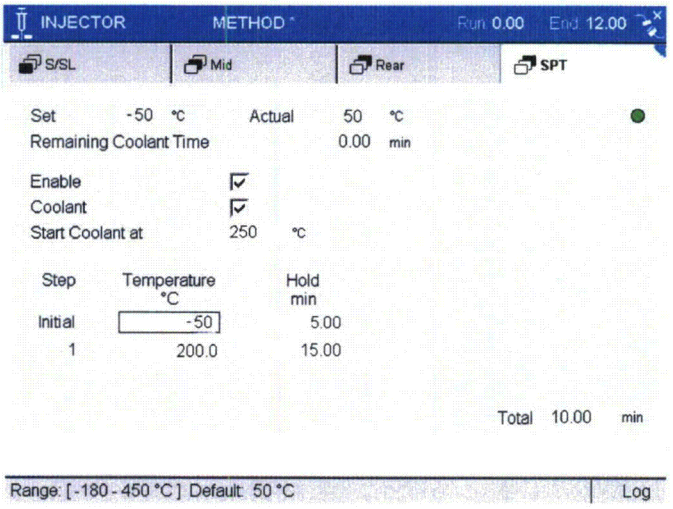
Press on the Injector tab.
Press Page X button to view the SPT Injector setup, verify that the coolant choice of LCO_2 or LN_2 matches the 436-GC/456-GC hardware.



Building an SPT method

Follow the steps to set the SPT parameters:

1. Press the Injector button and press the SPT tab.



2. Verify that the SPT Enable checkbox is checked.
3. Verify that the Coolant checkbox is checked.
4. The "Start Coolant at" parameter is the temperature at which the coolant valve turns on.

When the cooling starts the "Remaining Coolant Time" will count down. If the cooling reaches the set temperature and the 436-GC/456-GC does not go into run, the cryo-valve will be turned off it the coolant time-out is reached, and no further coolant will be applied. An event error will then be generated.

5. The SPT temperature program can now be entered, see above for a typical SPT program.

Replacing the Trap Assembly

WARNING:
BURN HAZARD

The trap can suddenly become extremely hot. Both the trap and the SPT valve oven can remain hot for an extended period of time, although the SPT cover, cryo enclosure, and SPT valve oven cover might become cool. Always turn power off to the GC and disconnect the SPT trap harness connector from the SPT transformer prior to removing the SPT cover or working on the SPT trap.

Tools required

- 3/16" back-up wrench (supplied in kit)
- 7/16" open-end wrench
- Needle nose pliers
- #2 Phillips screwdriver
- Large flat blade screwdriver (LN_2 Option only)
- 1/4" open-end wrench (2)

CAUTION

Ultra-clean reducing ferrules (supplied in kit) are required for connecting the 1/16" stainless steel oven/trap lines to the trap ends. These ferrules provide leak-tight seals, while electrically isolating the trap end fittings from the tubing, and have been specially treated to ensure that they are free of contamination.

To maintain the ultra-clean condition of the reducing ferrules, the use of clean cotton gloves or clean forceps is recommended.

CAUTION

The replacement trap assembly is provided as a single unit. It should not be disassembled. The following description of its basic components is for reference only during installation of a replacement trap assembly.

VTAT traps cannot be used in the SPT.

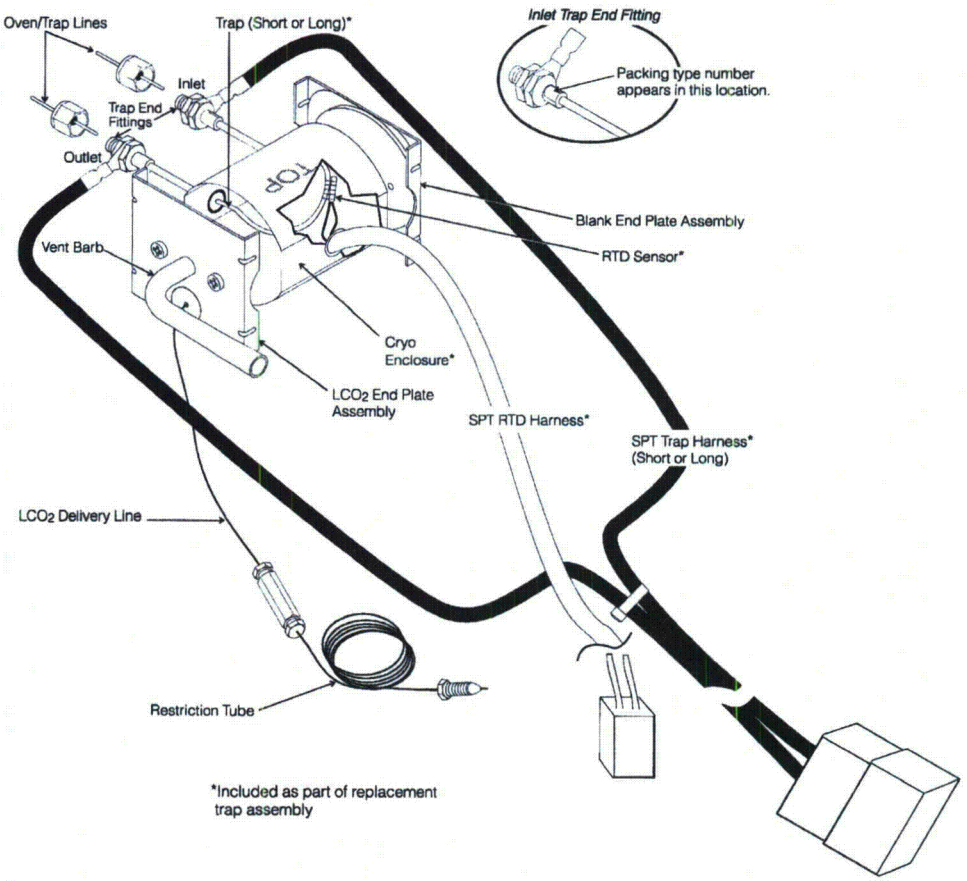
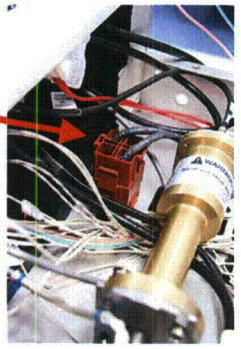


Figure 28: SPT Components (Short Trap Assembly, LCO_2 Option Shown

Remove the SPT Cover

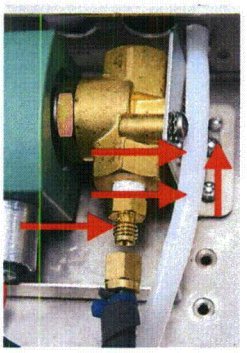
Follow these steps to remove the SPT cover and valve oven.

1. Turn the 436-GC/456-GC power off (using the powerswitch) and unplug power cord.
2. Shut of coolant supply.
3. Remove GC top cover.
4. Unplug SPT trap harness connector from SPT transformer harness (on page 176).

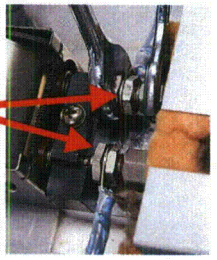


5. Remove SPT cover, by loosening the 2 screws.

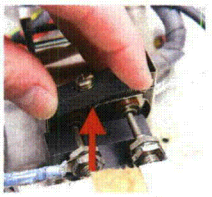
6. Disconnect the **LCO_2 delivery line** (part of **LCO_2 end plate assembly**) from the **union** that connects it to the **restriction tube** or Disconnect **LN_2 delivery line** (part of **LN_2 end plate assembly**) from the **LN_2 solenoid valve** by loosening and disengaging the Swagelok nut from the fitting on the **LN_2 solenoid valve**. The **valve mounting bracket** might need to be loosened slightly so fitting can be moved towards the rear.



7. Disconnect oven/trap lines from **trap end fittings.** Use the 3/16" backup wrench to prevent rotation of the **trap end fittings** (the flats on **trap end fittings** should remain vertical). With the 7/16" open-end wrench, loosen the two **Swagelok nuts** to release any strain on the **reducing ferrules**.



8. Locate the screw holding the trap clamping bar. Notice the Belleville washer under the screw head. Unscrew and remove the bar.



9. Lift the trap/end plate assemblies to disengage trap ends from slots in trap mounting block.

10. Remove the trap/end plate assemblies (if necessary, lift up the SPT valve oven cover).

Detach Trap Assembly

1. Unhook the four **SPT extension springs** from the slots along the edge of the **LCO_2** or **LN_2 end plate**. The springs should remain attached to the **blank end plate assembly**. Use needle nose pliers to gently grip the open hook end of the extension springs. Do not overextend the **springs**. Overextending them might result in their permanent distortion.

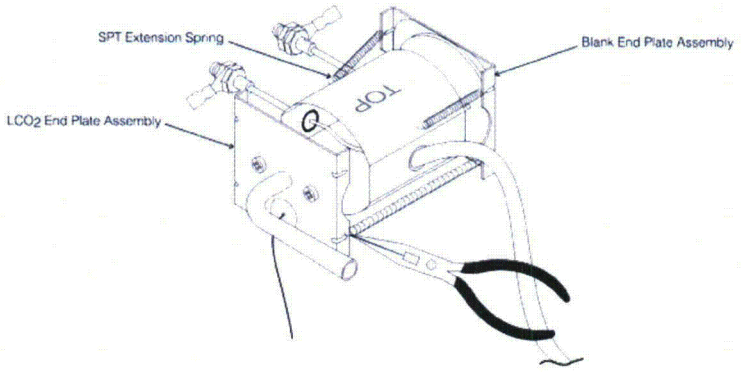
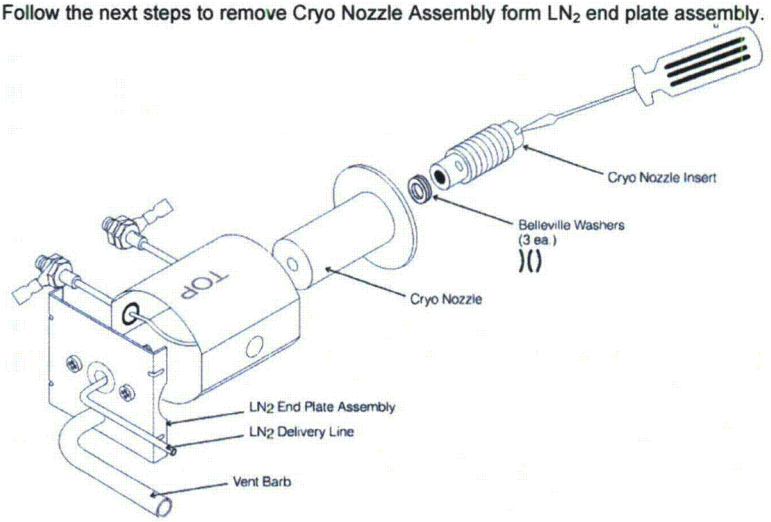


Figure 29: Removing Springs from Trap assembly

Remove Cryo Nozzle Assembly

Follow the next steps to remove Cryo Nozzle Assembly form LN_2 end plate assembly.



1. Unscrew the **cryo nozzle insert** using a large flat bladed screwdriver.
   Do not remove the **cryo nozzle insert** from within the **cryo nozzle assembly**.
2. Remove entire **cryo nozzle assembly:** hold both **cryo nozzle** and **cryo nozzle insert** together while removing them from within the trap coil. This prevents loss of the three **belleville washers** that are stacked inside the **cryo nozzle assembly**.

CAUTION

If the used trap assembly is still functional, its ends should be capped off to isolate it from atmospheric contamination during storage.

Installing the Trap Assembly

Follow the steps below to prepare the replacement trap:

1. Confirm the part number of the intended replacement **trap assembly**. Check that the size of the trap is correct (the long trap has three coils).
2. Uncap the replacement trap.
3. Hold the **trap assembly** *with the trap ends pointed towards you, and the word* "TOP" *on the* **cryo enclosure** *facing upwards.*

In the following procedures, the terms "right" and "left" refer to this orientation.

4. Preassemble the **LN_2** cryo nozzle components ((Figure 33, on page 201):
   a. Using a narrow pointed tool (such as a small screwdriver), align the stack of three **belleville washers** in alternating orientations, placing the first washer with its convex side facing the pointed tool.
   b. Holding the pointed tool pointing up so the washer stack doesn't fall off, lower the **cryo nozzle** over the washer stack so that the washers are inserted fully into the large cavity of the **cryo nozzle.**

c. Tip the **cryo nozzle** so that its large cavity is facing upwards, then remove the pointed tool so as to not disturb the orientation of the washer stack.

d. Insert the **cryo nozzle insert** (slotted end facing out) into the cryo **nozzle cavity**.
e. Keep the **cryo nozzle insert** closely engaged within the **cryo nozzle.**

5. Align the **LN_2** end plate assembly with the right open end of the cryo enclosure and engage the cryo nozzle assembly (Figure 33, on page 201):

   a. Orient the **LN_2 end plate assembly** so that its **delivery tube** is pointing away from you. **Vent barb** and **vent tube** should be at the bottom with **vent tube** exiting to the rear.

   b. The **LN_2 end pilot assembly**, which is attached to the inside of the **LN_2 end plate assembly**, should fit into the right open end of the **cryo enclosure**. Take care not to catch the edge of the **gasket** against the rim of the **cryo enclosure**; the rim should bear evenly against the **gasket** facing in the area surrounding the **end pilot.**

   c. From the left side of the trap coil, engage the **cryo nozzle assembly** through the coil. Screw the **cryo nozzle insert** onto the threaded **stud** of the **LN_2 end pilot assembly** using a large flat blade screwdriver. Take care not to cross-thread the **cryo nozzle insert**. Tighten gently, then back off approximately $1/12^{th}$ of a turn.

6. To align the **LCO_2** end plate assembly, with the right open end of the cryo enclosure:

   a. Orient the **LCO_2 end plate assembly** so that its **delivery tube** is near the bottom of the right face and pointing away from you. The **Vent barb** and **vent hose** should exiting to the rear.

   b. The **LCO_2 end pilot assembly**, which is attached to the inside of the **CO_2 end plate assembly**, should fit into the right open end of the **cryo enclosure**. Take care not to catch the edge of the **gasket** against the rim of the **cryo enclosure**; the rim should bear evenly against the **gasket** facing.

7. Align the **blank end plate assembly** (see Figure 29, on page 186) with the left open end of the **cryo enclosure**. The four **SPT extension springs** attached to this **assembly** will have to be held out of the way of the **enclosure**. The **blank end pilot** should engage into the left rim of the cryo enclosure. Take care not to catch the edge of the **gasket** against the rim of the enclosure; the rim should bear evenly against the **gasket** facing.

8. Use needle nose pliers to gently grip the open hook end of the **extension springs**, and extend the **springs** to engage their respective slots that should be aligned on the right end of the **cryo enclosure** in the **LCO_2** or **LN_2 end plate assembly**. Do not overextend the **springs**. Overextending them might result in their permanent distortion.

9. If both **end plate assemblies** are properly engaged into the **cryo enclosure**, they should be parallel to each other and perpendicular to the sides of the cryo enclosure. The **SPT extension springs** should firmly hold the **end plate assemblies** against the rims of the **cryo enclosure**. The **SPT extension springs** should not touch the trap ends.

10. The oven/trap lines that connect to the **trap end fittings** should be preformed for proper alignment with the **trap end fittings**. The **nut** and the **reducing ferrules** should be preplaced onto the line ends so that when the trap is installed, there is minimal bending of the lines.

To maintain the ultra-clean condition of the reducing ferrules during installation, the use of clean cotton gloves or clean forceps is recommended.

11. Confirm that the **trap assembly** with the trap ends pointing towards the front of the GC, with the word "TOP" on the **cryo enclosure** facing upwards.

12. Engage the oven/trap lines into trap ends.

13. Align both trap ends with the slots in the **trap mounting block**. Lower trap ends to engage into the slots.

14. Place the **trap clamping bar** over the trap ends.

15. Make sure the belleville washer on the trap clamping bar screw has its concave side facing the trap clamping bar. Tighten **trap clamping bar screw** to anchor the **trap assembly** into the **SPT valve oven**. Do not overtighten.

16. Reconnect the valve oven/trap lines to **trap end fittings**. The packing type number is stamped on the inlet **trap end fitting** (seeFigure 28, on page 184). Carefully engage each **nut** onto its respective **fitting** to prevent cross-threading. Using the faces of the hexagonal nut as a guide, tighten each nut fingertight, then tighten an additional:
    - Using new ferrules, 4/6 - 5/6 of a turn.
    - Reusing ferrules, 2/6 of a turn.

17. Do not over tighten. Use 3/16" backup wrench to prevent rotation of the **trap end fittings** (the flats on **fittings** should remain vertical).

To avoid the possibility of shorting the SPT electrical circuit, do not insert the valve oven/trap lines more than 8 mm into the trap ends. Inserting the lines too far into the trap ends will cause them to come in contact with the trap.

Do not plumb the valve in such a way that metal tubing shorts across trap tube ends.

18. Connect the **LCO_2** delivery line:

    a. Connect the **LCO_2 delivery line** (part of **LCO_2 end plate assembly**) to the union that connects to the **restriction tube**.

    b. Check that **vent barb** and **vent hose** are exiting to the rear. Feed the **vent hose** through the back panel of the GC.

19. Connect the **LN_2 delivery line** (part of **LN_2 end plate assembly**) to the **LN_2 solenoid valve** by engaging the Swagelok nut to the connector on the valve (see on page 185). Do not over tighten Swagelok fitting. The **Valve mounting bracket** might need to be anchored to the GC. Do not induce strain onto the **delivery line**: carefully align the **LN_2 solenoid valve** and its bracket during tightening of the **valve mounting bracket.**

20. Check that the **drip pan** is located under the **LN_2 solenoid valve**. The **drip pan** should not excessively block vent holes in the column oven top.

21. Tighten down the SPT valve oven cover.

22. Install the SPT cover.

23. Connect the SPT trap harness connector to the SPT transformer harness.

24. Replace GC top cover.

25. Turn on coolant supply.

26. Power up GC.

Trap Conditioning

With carrier or auxiliary gas passing through the trap, condition the trap according to the following table:

Packing Type	*Mesh Size*	*Conditioning Procedure*

Silanized Glass Bead	60-80	1 to 16 hours @ 250 to 400 °C
Tenax/GR Charcoal	60-80	4 to 16 hours @ 250 °C
HayeSep D	60-80	4 to 16 hours @ 250 °C
Carbopack B, Carbopack C, Carboxen 1000, Carboxen 10001	60-80	4 to 16 hours @ 270 °C
5% OV-101 on Chromosorb G/HP Haysep D	60-80	4 to 16 hours @ 270 °C
Tenax Gr, Carbopack B, Carbosieve S-111	60-80	4 hours @ 225 °C
Tenax TA,	60-80	4 hours @ 225 °C
Tenax TA, Charcoal	60-80	4 hours @ 200 °C
Carbopack C, Carbosieve S-111	60-80	4 hours @ 200 °C
Carbopack C, Carbopack B	60-80	4 hours @ 270 °C
Carbopack B, Carbosieve S-111	60-80	4 hours @ 200 °C
Tenax GR	60-80	4 hours @ 200 °C

Table 34: SPT Trap conditioning

Replacement SPT traps

The table below is a partial list of available trap assemblies. Consult your local sales representative or Parts and Supplies Marketing for additional versions.

Trap Description	*Partnumber*
Short Traps	
Short Trap Assembly Kit (8 cm), silanized glass beads	0392571392
Short Trap Assembly Kit (8 cm), blank	0392571391
Blank Trap	0392571301
Packed, Glass Beads	0392571302
Long Traps	
Long Trap Assembly Kit (30 cm), blank	0392571491
Blank Trap	0392571401
Tenax GR/Charcoal	0392571402
Tenax GR	0392571403
Carbopack B, Carbosieve S-III	0392571404
5% OV-101 on Chromosorb G/HP, HayeSep D	0392571405
Carbopack C, Carbopack B, Carboxen 1000, Carboxen 1001	0392571406
Carbopack C, Carbosieve S-III	0392571407
HayeSep D	0392571408
Tenax TA, Charcoal	0392571409
Tenax TA	0392571410
Tenax GR, Carbopack B, Carbosieve S-III	0392571411
Kit, Blank Trap	0392571491
Kit, Tenax GR/Charcoal	0392571492
Kit, Tenax GR	0392571493
Kit, Carbopack B, Carbosieve S-III	0392571494
Kit, 5% OV-101 on Chromosorb G/HP, HayeSep D	0392571495
Kit, Carbopack C, Carbopack B, Carboxen 1000, Carboxen 1001	0392571496
Kit, Carbopack C, Carbosieve S-III	0392571497
Kit, HayeSep D	0392571498
Kit, Tenax TA, Charcoal	0392571499
Kit, Tenax TA	0392571480
Kit, Tenax GR, Carbopack B, Carbosieve S-III	0392571481

Table 35: SPT replacement traps

SPT replacement parts

Part Description	Partnumber
Cryo Valve: LCO_2 LN_2	0392555501 0392555701
Ferrule for Restriction Tube (LCO_2)	2869450200
Nuts for Restriction Tube (LCO_2)	2869450100
Insulation Tubing (LN_2)	2400046700
Outlet Filter (LCO_2)	2759082600
Reducing Ferrule (Ultra-clean, for Trap Assembly)	0391885000
Restriction Tube, 22" Long (LCO_2)	0391885301
SPT PCB	0391875800
SPT Transformer	0391878800
Transfer Line from LN_2 Supply to GC (1/4" Copper, 10' Length)	3700014601
Trap Clamping Bar	0391876500
Trap Mounting Block	0391876400
Union (LCO_2)	2821145700
Transfer Line from LCO_2 supply to GC (1/16" stainless steel, 10')	0391885200
Ferrules for stainless steel Transfer Line (Front)	2869399600
Ferrules for stainless steel Transfer Line (Back)	2869399700
Nuts for stainless steel Transfer Line	2869399800
Ferrules for stainless steel Transfer Line (1/8") (Front)	2869402700
Ferrules for stainless steel Transfer Line (1/8") (Back)	2869402800
Nuts for stainless steel Transfer Line (1/8")	2869402900
Ferrules, Vespel, Reducing 1/8" To 1/16"	0391885000
Installation Kit, LCO_2	0391880101
Installation Kit, LN_2	0391880102

Table 36: SPT Replacement Parts

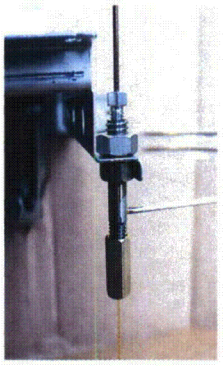
Troubleshooting

This section provides troubleshooting information to aid the operator in locating and correcting apparent problems when working with an SPT system. The following table is geared specifically to SPT troubleshooting. If you have questions about any of the corrective actions described below, contact your Bruker Customer Support Representative.

Symptom	*Possible Cause*	*Remedy*
SPT overheating.	Faulty SPT RTD sensor.	Replace trap assembly.
	SPT RTD harness not properly connected to SPT electronic board.	Check and reconnect harness.
SPT heating too slowly.	*Short in electrical system:*	
	Oven/trap line making contact with trap.	Adjust oven/trap line and trap end fitting.
	Insulating surface (gray) of the SPT trap mounting block damaged.	Replace mounting block.
	Bushings not isolating trap from the cryo enclosure.	Replace trap assembly.
	Cryo valve stuck open.	Replace cryo valve.
SPT not heating.	SPT transformer circuit breaker tripped.	Correct problem; reset.
	SPT trap harness not properly connected to the SPT transformer.	Reconnect harness.
	SPT RTD harness open or shorted.	Replace trap assembly.
	SPT trapping temperature set to <0° C.	Set temperature to equal or greater than 0°C.
No coolant flow.	SPT performing desorption temperature program.	Perform trapping temperature program.
	Coolant supply exhausted or low.	Replace coolant supply.
	Coolant restriction tube plugged (LCO_2).	Clean or replace restriction tube. Replace in-line filter.
	Coolant supply not connected.	Connect and turn on coolant supply.
	Coolant is not enabled in GC Configure table.	Enable coolant.
	Cryo valve is not connected to Temperature Control electronic board.	Connect cryo valve.
	Cryo valve stuck closed.	Replace cryo valve.
SPT overcooling.	Cryo valve stuck open.	Replace cryo valve.

Symptom	*Possible Cause*	*Remedy*
SPT undercooling or cooling too slowly.	Supply cylinder too warm or to cold.	Relocate coolant supply to a location within recommended temperature limits. Make sure that GC and transfer line are cooler than cryo supply cylinder.
	Coolant supply has been depleted of liquid phase CO_2.	Replace supply with a full dewar or cylinder.
Trapping temperature is oscillating out of ready.	Supply too warm (≥27°C)	Relocate coolant supply to a location within recommended temperature limits. Make sure that GC and transfer line are cooler than cryo supply cylinder.
	GC and transfer line are too warm relative to the supply.	Provide a cooler environment for the GC (should be cooler than cryo supply).
	Transfer line is too long.	Shorten transfer line. Insulate transfer line.

MINIGAS SPLITTER



The MiniGas Splitter is used as replacement for an S/SL/PTV Injector. The MiniGas Splitter is installed onto the column bracket in the GC/Large-Valve/Dual-valve oven.

When a capillary column is used in combination with the MiniGas Splitter, alignment of the column is critical. Use the following drawing to precisely align the capillary column.

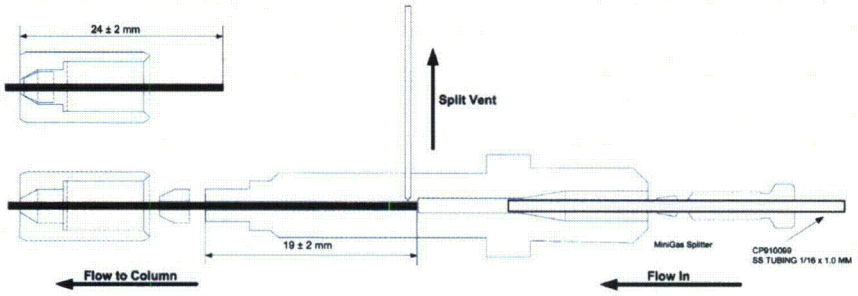


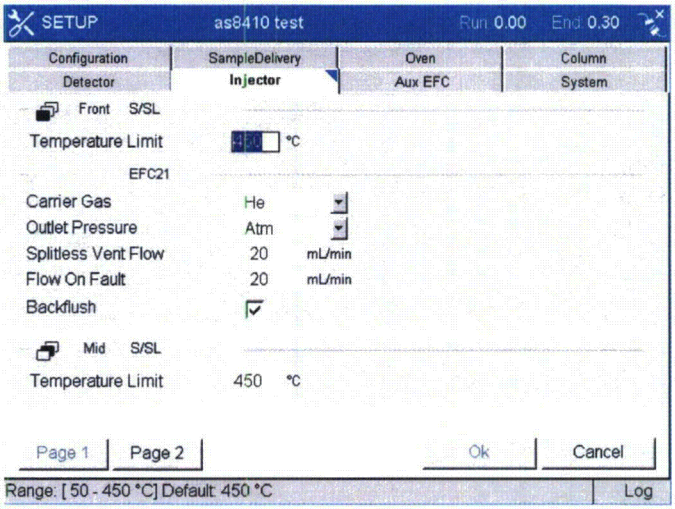
Figure 30: MiniGas Splitter Column Depth

BACKFLUSH OPTION

The 400-GC series Gas Chromatographs can optionally be equipped with a backflush option. Backflush is the method that enables you to:

1. Shorten the overall analysis time by focusing on the part of interest only.
2. Prevent high boiling sample constituents to reach the detector.
3. Enable MS users to change the injector septum without the need to take the vacuum from the MS system.
4. The backflush system, serially coupled columns can be used for selectivity tuning; by carefully selecting the pressure drop over the respective columns having different polarity creating an overall polarity between the two column polarities.

Back flush needs to be activated in the Setup and is coupled to an injector S/SL or PTV. After selecting the backflush option an extra method parameter becomes active in the flow/pressure page of the EFC corresponding to the injector!



The backflush option is built around a combination of EFC21/EFC25, controlling an S/SL or PTV injector, and EFC24 to provide a mid-point pressure. Two columns, a pre-column and an analytical column, are serially connected via a T-junction to which also the mid-pressure has been connected.

Backflush of the first (or pre-) column is realized by giving a low pressure to the EFC21/EFC25, e.g. lower than the EFC24 pressure. The components that originally entered the first column are flushed out of the gas chromatographic system via the split vent line.

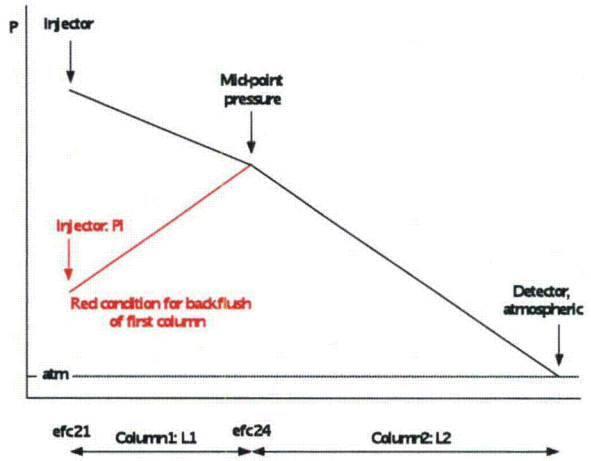
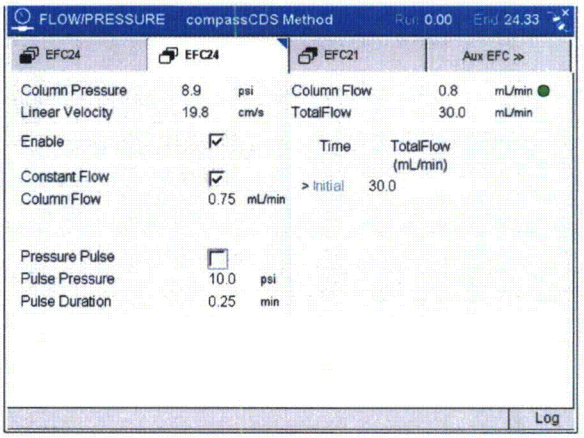


Figure 31: Backflush column pressure overview

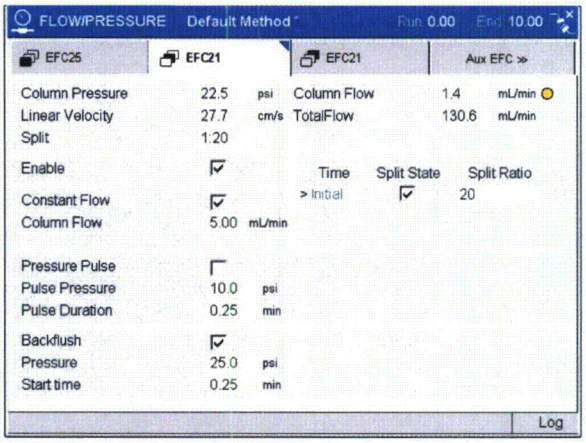
The above representation is valid under isothermal conditions or under temperature programmed conditions with constant pressure control. In modern gas chromatography, temperature programmed analysis is often performed using constant flow operation in order to reduce the analysis time and reduce the elution temperature.

In the 436-GC and 456-GC-GC constant flow operation is added to the EFC24 control. Additionally, the flow may be entered and read in two decimals.



In case of serially coupled columns, as is the case with the backflush option, it is necessary to have both controllers, EFC21/EFC25 and EFC24, under constant flow (or programmed pressure) control while temperature programming.

In the EFC21/EFC25 control a new control parameter has been added to allow constant flow, or pressure programming, for a specific, user defined period, followed by a backflush condition. Backflush is achieved via programming the EFC21/EFC25 from the current pressure to a method (user) defined low pressure at a rate of-30 psi/min. (The programming rate to enter the backflush state is fixed.)
The backflush option must be selected in set-up on page 49!


FLOW/PRESSURE
Default Method *
Run 0.00
End 10.00
EFC25
EFC21
EFC21
Aux EFC »
Column Pressure 22.5 psi
Linear Velocity 27.7 cm/s
Split 1:20
Enable
Constant Flow
Column Flow 5.00 mL/min
Pressure Pulse
Pulse Pressure 10.0 psi
Pulse Duration 0.25 min
Backflush
Pressure 25.0 psi
Start time 0.25 min
Column Flow 1.4 mL/min
TotalFlow 130.6 mL/min
Time
Split State
Split Ratio
> Initial
20
Log

Optimizing

Serially coupled columns operate optimal when the flow through the first column is less than the flow through the second column. In this case all eluting through the first column is transferred to the second column plus a small contribution from the EFC24 for the pressure control over the second column.

In case the pre-column and analytical column have the same inner diameter, in SetUp the actual column length can be programmed, and all is calculated well. (The set-up has not yet been modified for this!)

In case of different inner diameters for pre-column and analytical column EFC24 gets the actual column dimensions; EFC21/EFC25 requires an estimate for proper control. The estimate should represent the flow resistance of the total length (pre-column plus analytical) related to the analytical column inner diameter.

For deviating pre-column diameter use Hagen-Poiseuille relation to convert:

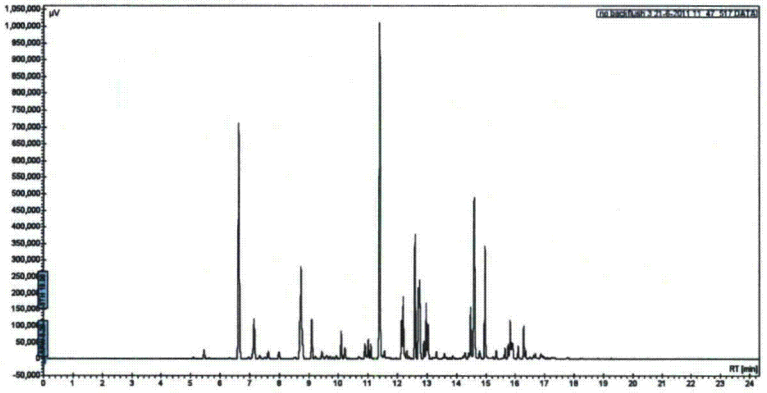
- 12.2 meter 0.32 mmID is equivalent to 1 meter 0.25 mmID
- 20.2 meter 0.53 mmID is equivalent to 1 meter 0.25 mmID

Obviously the more accurate the length is given the better the flow match will be.

For both EFC units the vacuum outlet needs to be selected in case of connection to MS.

An example:
Sample: premium gasoline
Oven program: 35 (5 min) > 15°/min > 250 (5 min).
Carrier helium
Constant flow: EFC21: 1 ml/min; EFC24: 1.01 ml/min.
Pre-column: 7 meter x 0.25 mm 0.25 µ BR1
Analytical column: 30 meter x 0.25 mm 0.25 µ BR1

Without backflush:



Backflush at 9 minutes (-30 psi/min to 4 psi)

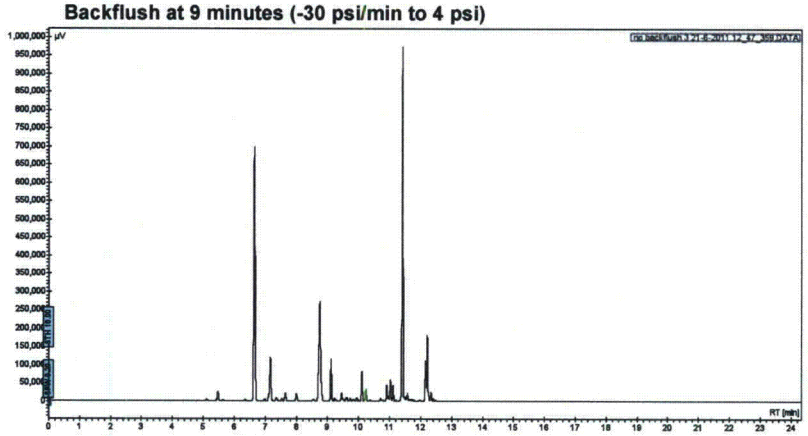
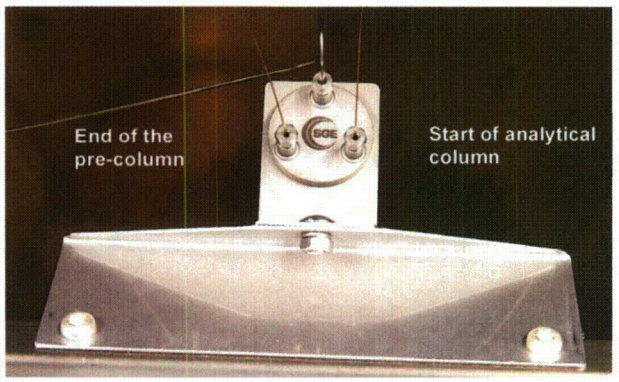


Figure 32: Chromatogram with backflush

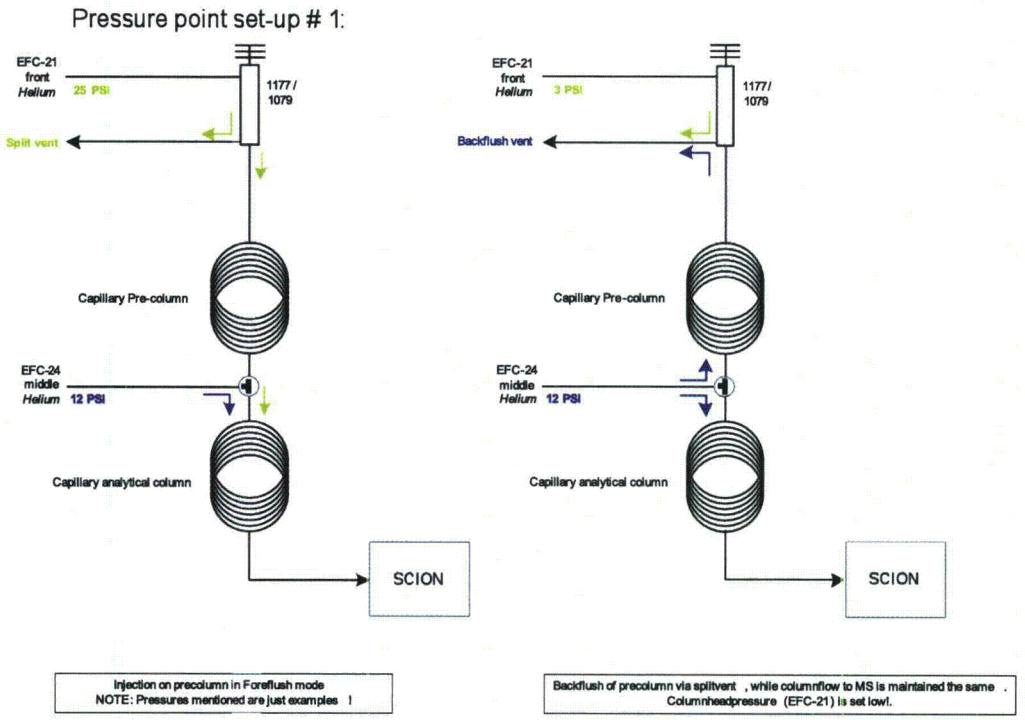
The actual coupling of EFC24 and the two columns is done with the SGE splitter using the SGE SilFlow[tm] technology.



This assembly is mounted (pre-installed) on the bottom of the oven with the (top) connection made to the EFC24.
Capillary column connections are on the front and the connections are fingertite connections (Refer to appropriate installation instructions; special tool is included!).
The capillaries connected are from the pre-column and the analytical column; their respective positions are not fixed. Refer to drawings on page 205.

The SGE SilFlow [tm] has certain advantages above the Valco piece:

- It is possible to mount larger inner diameter tubing representing the mid-pressure point, and the reducing union may be omitted. This will result in a more accurate pressure reading.
- Easy connection of the capillary-ends; no interferences.
- Above mentioned drawbacks are solved.



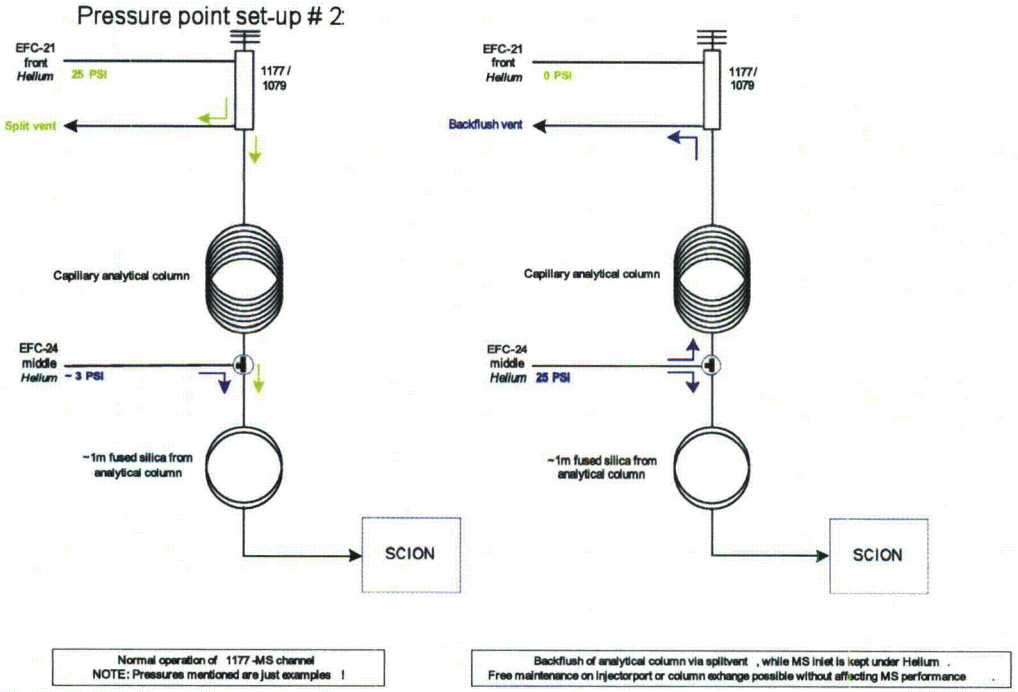


Figure 35: Backflush Connection Examples

DETECTORS

INTRODUCTION

The 436-GC/456-GC can accommodate up to three installed detectors and have all three running simultaneously.

The standard detectors available on the 436-GC/456-GC are:

- The Flame Ionization Detector (FID), on page 209.
- The Pulsed Discharge Helium Ionization Detector (PDHID), on page 217.
- Nitrogen Phosphorus Detector (NPD), on page 224.
- Thermal Conductivity Detector (TCD), on page 233.
- Electron Capture Detector (ECD), on page 242.
- Pulsed Flame Photometric Detector (PFPD), on page 251.

Except for the TCD and PFPD any combination of three detectors can be installed.

In case of the PFPD, only two may be installed but any one additional detector may also be installed including a TCD.

In the case of the standard TCD, only two can be installed and then no additional detectors can be installed.

However, a dual TCD option is available as a custom solution which allows installation of an additional ionization detector.
The dual TCD consists of 2 detector cells in one housing but using only one heating zone.

Detectors are mounted on the top of the 436-GC/456-GC, above the left side of the column oven.
The position of the detector, however, is determined by the location of the detector's electrometer in the electronics cabinet. Detector electrometers are installed on the left side of the GC accessed by removing the left side panel.
All cables connecting the electrometers to the detectors are accessible by removing the 2 covers on the top left of the 436-GC/456-GC, i.e. the detector top cover and the cover over the display/display.

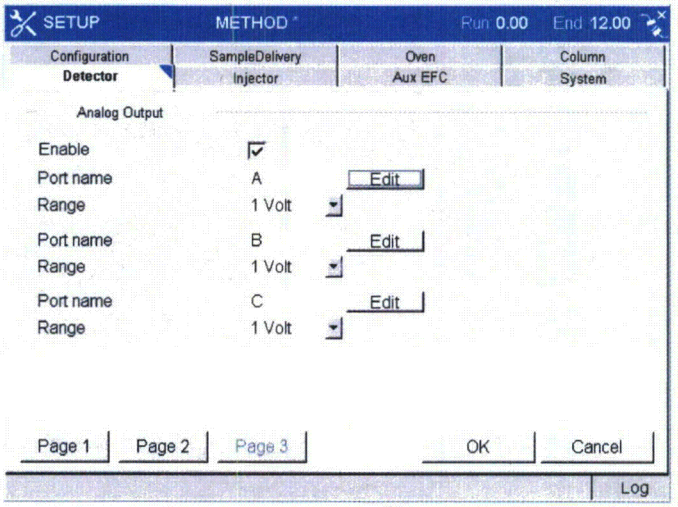
For connecting a capillary column to a detector, see the procedure Connect Capillary Column to Detector (available in the Installation Manual).

ANALOG OUTPUT

The 436-GC/456-GC can be equipped with an option board. On the option board 3 analog-output signals are present. Also 8 external events output are available. Connection details can be found here on page 359.

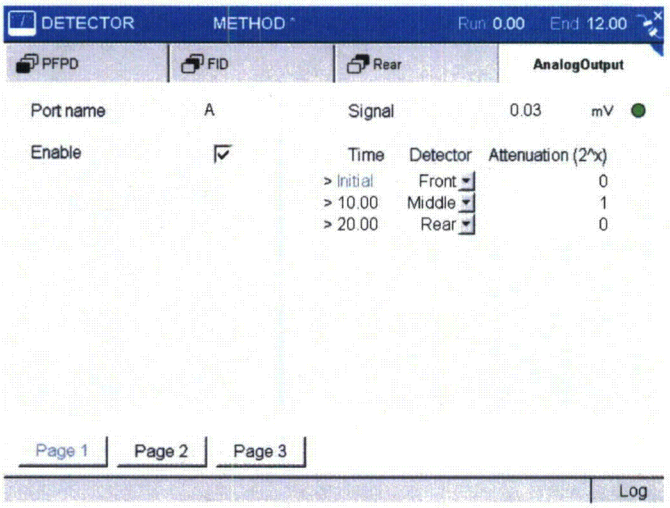
In the SETUP page by the detector tab on the second page (Page 2 or page 3) the Detector Analog Output screen will be visible, only if the Analog option board is present.

The Analog Output Port name can be changed by the button "Edit".



Analog output is disabled by default. Meaning that a GC with analog output hide the analog output tab. If analog output is enabled, the detector frequency is fixed to 50 Hz (local automation and Workstation).
If the GC is connected to a Workstation the analog output frequency is depending of the detector frequency in the method, up to a maximum of 50 Hz.

The analog output ports are programmed in the 436-GC/456-GC method by pressing the DETECTOR key on the display, select Analog Output.
This brings up a menu screen from which you choose one of the three ports to program; Page 1, Page 2 or Page 3. The following screen appears when Page 1 is chosen.



The Analog Output Port name in this example is A, but can be changed in the Setup screen. The individual analog ports can be assigned to any installed detector on the 436-GC/456-GC. The ports are identified as A, B and C with the default assignment being front, middle and rear detector respectively. The Analog output full-scale range can be set (in Setup, on page 35) to 0-1 Volt or 0-10 Volt (jumpers on the option-board must be set to "auto" (default).
In the 0-10 Volt full-scale, peaks will be 10 times higher than in the 0-1 Volt full-scale range.

The right-side of the screen is used to time program the signal source of the analog output. By default analog output A is assigned to the front detector. In the example above, the analog port is programmed to transmit the Front detector signal for the first 10 minutes of the run and then switch to the Middle detector. After 20 minutes the switch will be switched to the rear detector for the remainder of the run. This mode of operation is known as detector switching.

Note that the attenuation[9] should normally be set to 0. However for optimum resolution (less electronic noise) the best results are achieved by setting the attenuation to a negative value if small peaks are detected. For overloaded peaks set the attenuation to a positive value (attenuation range from -16 till +16).

The 436-GC/456-GC has a number of analog cable options on page 58, depending on the device to which the cable is connected. All cables have a 15 pin D-shell connector on one end to attach to J401 on the 436-GC/456-GC and have the appropriate connectors on the other end of the cable for the devices to which they are being attached.

---

[9] **The analog output formula:** $Analog\ output\ signal = \frac{detector\ signal}{2^{attenuation}}$

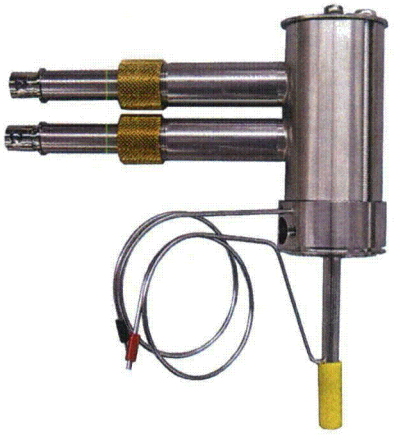
If attenuation > 0, the output signal will be decreased.

If attenuation = 0, the output signal equals the detector signal.

If attenuation < 0, the output signal will increase.

Example: to gain the signal with a factor 10, set the attenuation to -3.32 ($2^{-3.32}$ = 1/10).

FLAME IONIZATION DETECTOR (FID)



The flame ionization detector (FID) is the most sensitive gas chromatographic detector for hydrocarbons such as butane or hexane. With a linear range of 6 or 7 orders of magnitude (10^6 to 10^7) and limits of detection in the low picogram or femtogram range, the FID is the gas chromatographic detector for volatile hydrocarbons and many carbon containing compounds.

The FID detector employs hydrogen as the combustion gas which is mixed with Air and the column eluent (helium, nitrogen or other appropriate gas) and burns at a small jet situated inside a cylindrical electrode. A potential of a few hundred volts is applied between the jet and the electrode and when a carbon containing solute is burnt in the jet, the electron/ion pairs that are formed are collected at the jet and cylindrical electrode.

The following instructions refer to the operation of a 436-GC/456-GC Flame Ionization Detector (FID). The FID is installed on a detector base, directly above the column oven. The FID electrometer is installed in the electronics cabinet on the left of the instrument. The electronics of the FID are controlled from the 436-GC/456-GC display, the gas flows are set and controlled either from the 436-GC/456-GC display (if detector EFC is installed) or at the manual pneumatics panel.

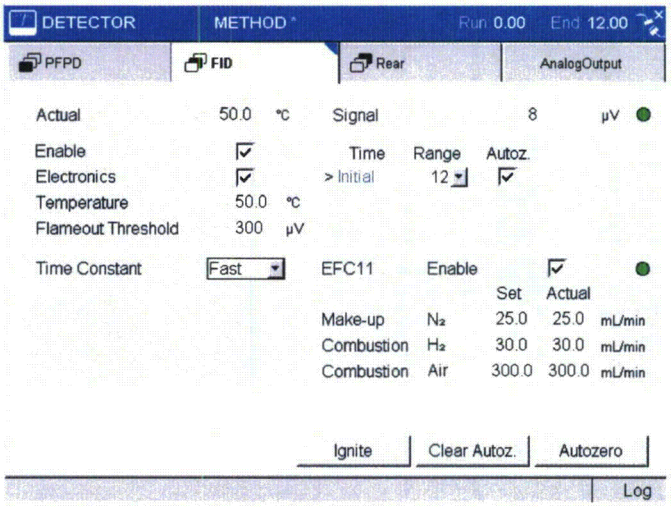
Initial Set-Up

To set-up the FID, proceed as follows. Note that when the 436-GC/456-GC is first powered up all heated zones with the exception of the column oven are powered off and all detector electronics are turned off.

1. Refer to the Pre-Installation Instructions for additional information on gas supply requirements.

2. Press the DETECTOR key on the 436-GC/456-GC display to display the DETECTOR screen. The FID should be listed in position Front (), Middle () or Rear (). If your GC is equipped with detector EFC verify the makeup gas you will be using with your FID.

3. Install a chromatographic column in the GC. If the analytical column is not pre-conditioned, use a no-hole ferrule in the detector column nut while conditioning the column and leave the detector end of the column loose in the oven. If the analytical column is pre-conditioned, follow the usual procedure for column installation.



4. If detector EFC is present, set the following flow rates in the adjustments section of the FID method:

   - Make-up flow to 25 mL/min (recommended gas is N_2, Helium reduces the FID sensitivity half).
   - The hydrogen flow to 30 mL/min.
   - Air flow to 300 mL/min.

5. Note: For optimum performance the combined column + make-up flow should be 30 mL/min, so some adjustment of the make-up flow may be necessary.

6. If manual pneumatics is installed, attach a bubble or digital flowmeter (AL5700) to the top of the FID tower using the adapters supplied in the FID accessory kit (Note that the most reliable measure of flow is directly from the flame tip. This prevents wrong-adjustment due to leaks in the overall tower assembly). Check the following flow rates and set them if necessary: combined carrier gas + make-up flow to 30 mL/min, the hydrogen flow to 30 mL/min and the air flow to 300 mL/min.

7. Press the DETECTOR key on the 436-GC/456-GC display, select the FID by its location and turn on the FID oven power. Set the oven temperature to 300 °C. Verify that the FID electronics is turned OFF.

1. Check the detector temperature status on the DETECTOR/FID screen. Check the FID range setting and set to range 12, if necessary.

Generally, to prevent water condensation in the detector assembly, the detector should be operated at a temperature above the column temperature and not below 150 °C. If the detector is operated at a lower temperature, condensation can lead to excessive noise.

2. After the FID oven has reached its setpoint temperature, turn on the FID electronics. Note that the FID will ignite automatically when auto-ignite conditions are fulfilled (for detailed information about auto-ignite conditions see here, on page 212). The 436-GC/456-GC monitors the FID background current continuously and will attempt to ignite the flame (only if in Setup/Detector the checkbox on page 48 "check for flame out" is checked) if the current drops below a specified threshold. Up to 3 attempts to ignite the flame will be made before the 436-GC/456-GC reports a flame-out fault. If a flame-out fault is reported, correct the cause of the flame-out and then clear the fault in Instruments Log.

   The FID will automatically ignite after the fault is cleared.

WARNING: EYE HAZARD ***DO NOT look directly into the detector tower when attempting to ignite the flame.***

You can verify that the flame is lit by monitoring the FID signal in the status region of the FID display.
Uncheck the box "AutoZ." and press "Clear AutoZ."
Initially the signal will read a very high value but should then stabilize. Typically the signal will be > 50 mV at range 12 when the flame is first lit but should drop to < 10 mV within 30 minutes.

WARNING: EXPLOSION HAZARD ***To avoid a possible fire or explosion, always turn off the flow of hydrogen when the column is removed or when the detector is not being used. This prevents the accumulation of hydrogen.***

3. Set the Time Constant to "Fast". The default setting for the time constant is "Fast".

   The time constant has 2 settings: SLOW and FAST.

   Fast time constant:
   a. Used with capillary columns (0.10/0.15 ID columns).
   b. Reduces high frequency noise.
   c. Used for peaks widths at half height less than 1 second.

   Slow Time Constant:
   a. Used with columns diameter from 0.25 and higher (and also packed columns).
   b. Used for broad peaks (widths at half height larger than 1.5 second).

4. The "Ignite" button applies power to the FID igniter filament for 5 seconds.

5. The "Autozero" button applies an immediate zeroing of the detector signal.

FID auto-Ignite flow diagram

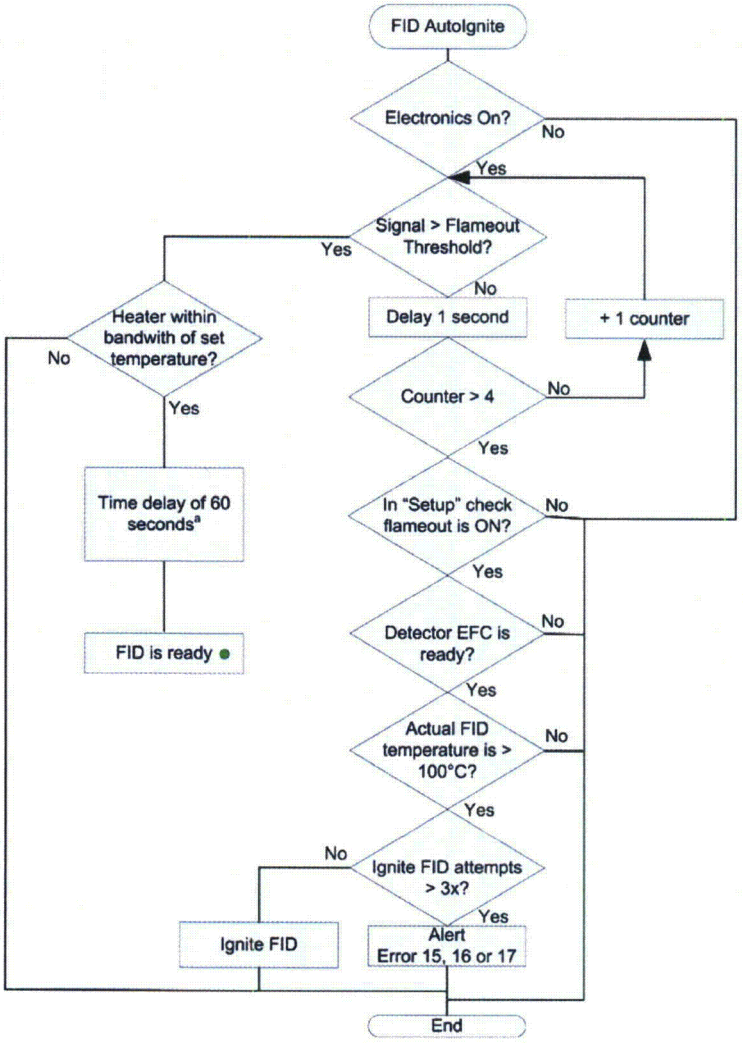
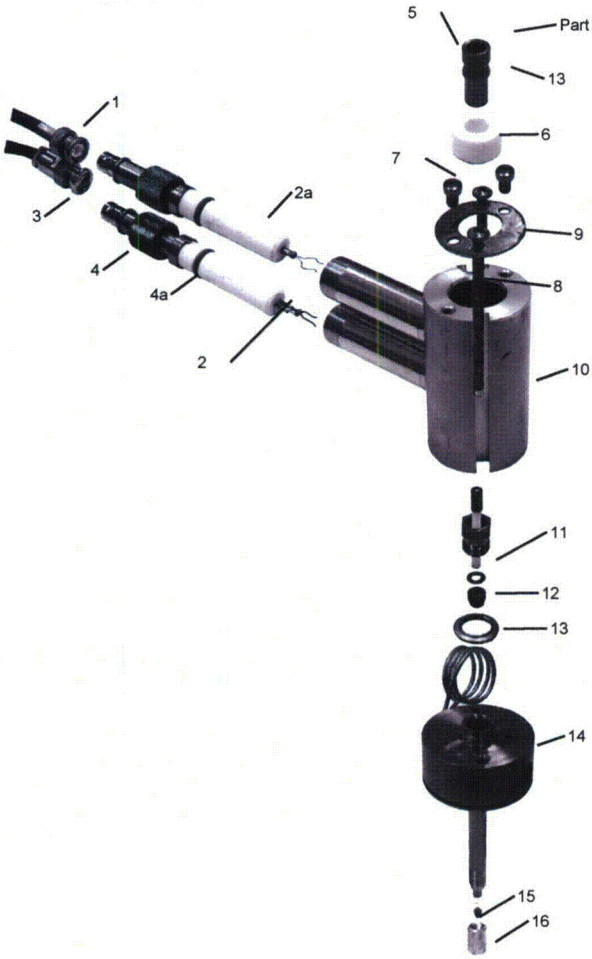


Figure 36: FID auto-Ignite flow diagram

Installation/Disassembly

At times, it is necessary to remove the detector components to inspect, clean, replace parts, or to install another detector. Follow the disassemble/reassemble instructions on the next pages for both the FID and the detector oven.

Exploded View



1 Signal Cable (391783000)
2 Signal Probe (200187300)
2a O-ring (baked, 0390596004)
3 Ignitor Cable (392548701)
4 Ignitor Probe (200187200)
4a O-ring (baked, 0390596004)
5 Collector Tube (394958700)
6 Ceramic Insulator (2100003200)
7 Screws, 8-32 x ¼" (1214200804)
8 Tower Screws, 8-32 x 3" (391866302)
9 Tower Top Plate (3400073100)
10 Detector Tower Body (100099300)
11 Flame Tip, 0.01 in. Capillary Tip (200187500)
11 Flame Tip, 0.02 in. Standard Tip (200193800)
12 Ferrule: Graphite/Vespel 40/60 max. 400°C (2869462201) Default
Graphite (100%) max. 450°C (CR211200)
13 Aluminum Seal Washer (1500334701)
14 Detector Base (392547501)
15 Column Ferrule, see table 7 "1/16" Column Ferrules" in Installation Manual
16 Column Nut 0.9 mm brass for capillary connections (394955100)
Column Nut 1.6 mm brass for 1/16" connections (CP742351)
Column Stainless Steel Nut (0.9mm) for High temperature applications (CP743117)

--- Flow Tube (200187600)
--- Large Stopper (394958600)

Figure 37: FID Exploded View

WARNING: SHOCK HAZARD

Before removing Ignitor cable (item 3, figure: above); cool down detector oven and oven temperature to <50 °C. Turn off any detector gases at their supply. Then turn off the main power switch and unplug the power cord.

Disassemble the FID

To disassemble the FID, proceed as follows:

1. Set the FID to 50 °C and wait for it to cool before disassembly. Turn the detector electronics and detector oven OFF in the active method. Remove the GC Top-Covers.
2. Turn off the main GC gas supplies to the detector at the pneumatics panel. These are the make-up, hydrogen and air supplies.
3. Disconnect the signal and igniter cables from their probes on the detector tower.
4. Remove the signal and igniter probes from the detector tower. *DO NOT* rotate the probes as you disconnect them from the electrical contacts in the tower. Place the probes on a clean surface such as a lint-free laboratory tissue.
5. Remove the 2 tower mounting screws from the top of detector tower.
6. Remove the tower assembly from the detector oven, lifting straight up until clear of the flame tip. Remove the collector tube and insulator from the detector tower. Avoid contamination of the ceramic insulator and probes. If the detector is not completely cool, use a metal tool (such as tweezers or a hooked wire) to remove parts from the tower assembly. Place parts on a clean Kimwipe®. Never place them on a counter or painted surface.
7. Remove the flame-tip assembly from the detector base. Take care not to break the ceramic flame tip tube or the Vespel®/graphite or graphite ferrule.
8. Remove and discard the aluminum seal washer from the detector base. Always use a new aluminum seal washer each time you reassemble the detector. Note that the aluminum seal washer may remain on the detector tower bottom when the tower is removed from the base.

Reassemble the FID

Refer to Installation/Disassembly on page 213 to reassemble the FID detector. To reassemble the FID, proceed as follows:

1. Re-assembly is the reverse of removal.
2. Install the flame tip in the detector base. The FID and NPD flame tips are identical.
   a. If you are installing the flame tip with a new Vespel/graphite ferrule, tighten the assembly finger-tight plus an extra 1/6-turn.
   b. If you are installing the flame tip assembly with a used graphite ferrule, tighten about 1/3-turn past finger tight.
3. The Vespel/graphite ferrule supplied with the GC has a maximum temperature limit of 400 °C. If it is necessary to operate the detector above 400 °C, you may need to replace the Vespel/graphite ferrule with a graphite ferrule (450 °C).
4. Install a new aluminum seal washer onto the shoulder of the detector base. To ensure a reliable tower seal, use a NEW aluminum washer each time you install the detector.
5. Place the detector tower on the detector base and secure it with the 2 tower mounting screws. Alternately tighten these screws a ½-turn as the tower tightens into place.

Handle the ceramic insulator and probes with tweezers to avoid contamination.

6. Carefully insert the igniter probe into the lower arm of the detector tower. Align the probe key with the tower arm slot. Check the orientation of the igniter elements by looking down through the top of the detector tower. The spring clip should slip around the flame tip and make good contact (see Figure 38, below). The igniter coil must not touch the flame tip assembly nor be positioned directly above it. Tighten the knurled nut to secure the probe.

7. Make sure the notch in the detector tower arm does not cut the O-ring seal.

8. Insert the insulator into the detector tower, then insert the collector tube into the tower. The collector tube must not touch the igniter coil.

9. Insert the signal probe into the upper arm of the detector tower. The probe clip should fit around the tapered section on the collector tube tightly enough to exert a downward force (see Figure 39). Secure the signal probe by tightening the knurled nut. Make sure the notch in the detector tower arm does not cut the O-ring seal.

10. Connect the igniter cable to the igniter probe and the signal cable to the signal probe.

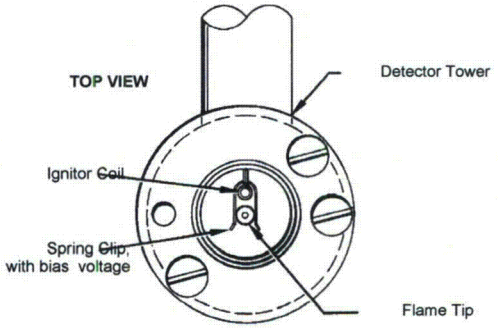


Figure 38: Orientation of Igniter Probe Elements in FID Tower

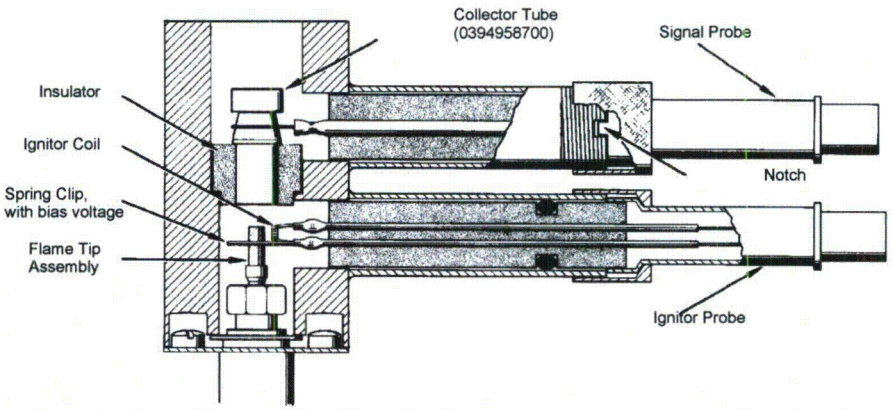


Figure 39: FID Cross-sectional View

MAINTENANCE

When the performance of the FID has degraded you may want to replace the flame tip ferrule, or clean the deposits from the internal parts, including the flame tip.

Clean the Flame Tip and Internal Parts

If the FID noise increases with frequent spikes, the internal parts may be contaminated and need to be cleaned.
To clean the flame tip and the internal parts of the FID, proceed as follows:

Always handle detector components with tweezers to avoid contamination.

WARNING:
EYE HAZARD

WARNING:
FIRE HAZARD

Use proper eye and skin protection. Methanol and acetone are toxic and flammable chemicals. Exercise appropriate care when you use and dispose of these substances.

1. Remove the detector and internal parts (see disassemble the FID, on page 214).
2. Scrape the deposits from the bore of the collector tube, the insulator and the metal part of the flame tip with emery cloth (e.g., SiO_2).
3. If the flame tip is plugged, insert a wire through the flame tip orifice to clear it.
4. If you have access to an ultrasonic cleaner, clean the collector, insulator and flame tip with distilled water.
5. Rinse the cleaned components with methanol or acetone and air dry.
6. Wipe the detector tower with acetone.
7. Clean the probe arms with methanol and air dry or dry in an air oven (maximum drying temperature of 150 °C).
8. Reassemble the FID, see Reassemble the FID, on page 214.

Replace the Flame-Tip Ferrule

After a period of prolonged use, the flame-tip ferrule may deteriorate and crack. As a consequence, a leak may develop around the base of the flame tip assembly that causes unstable baseline, noise and reduced sensitivity. In this situation, or when the detector is to be operated at temperatures above 400 °C, you must replace the ferrule. Be sure to select the proper ferrule for the application. The Vespel/graphite ferrule has a maximum temperature limit of 400 °C see exploded view FID on page 213.
Bruker recommends to NOT using other ferrules than those listed. Due to small dimensional variations common in these ferrules, leaks on higher temperatures are possible.

FID Test Chromatogram

For an FID test chromatogram see section Detector Test Chromatograms, on page 302.