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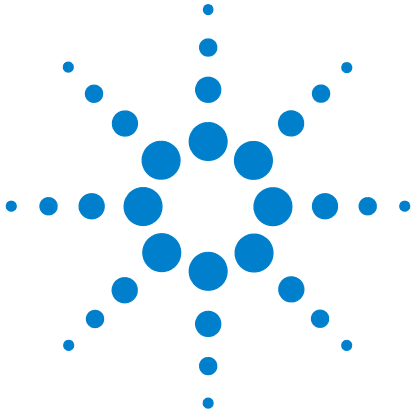

Agilent G3183B
Three-Way Splitter Kit

Installation and Operation Guide

Agilent Technologies

Notices

Manual Part Number

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Contents

1 Introduction

This manual covers the installation and operation of the G3183B three-way effluent splitter with makeup gas kit on the Agilent 7890A Series Gas Chromatograph (GC).

Overview

Splitter installation is done in three steps:

1. **Install the hardware**. Install the bracket and splitter plate on the wall of the GC oven. Connect the purge line to the EPC.
2. **Configure the restrictors**. Choose appropriate lengths and diameters of uncoated deactivated fused silica (UCDFS) tubing to obtain the desired split flows to each detector as described. Use the software tools supplied on the Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVD (Instrument Utilities DVD) to calculate the appropriate configuration for your GC system.
3. **Connect the column, splitter, and detectors**. Using the results of step 2, cut the appropriate lengths of the chosen diameter tubing for the restrictors. Install the metal ferrules onto the ends of the column and restrictor tubes (see *Swaging SilTite Ferrules* on the Instrument Utilities DVD for details) and connect them to the splitter plate. Connect the other ends of the restrictor tubes to the detectors.

How It Works

The splitter divides the effluent from a column among three different detectors (or other flow paths). The detectors can be operating at different pressures. A few examples are shown below:

- **Atmospheric pressure**
  FID (flame ionization detector)
  TCD (thermal conductivity detector)
  NPD (nitrogen phosphorus detector)
  ECD (electron capture detector)
  FPD (flame photometric detector)
- **Below atmospheric pressure**
  MS (mass spectrometer)
  MSD (mass selective detector)
  SCD (sulfur chemiluminescence detector)
  NCD (nitrogen chemiluminescence detector)
- **Above atmospheric pressure**
  AED (atomic emission detector)

The split ratio is determined by the relative dimensions (lengths and diameters) of tubing connecting the splitter to the detectors. Tubing dimensions may be determined from the spreadsheet calculator (included on the Instrument Utilities DVD), or independent software tools.

Figure 1 shows the plumbing configuration for the G3183B splitter.

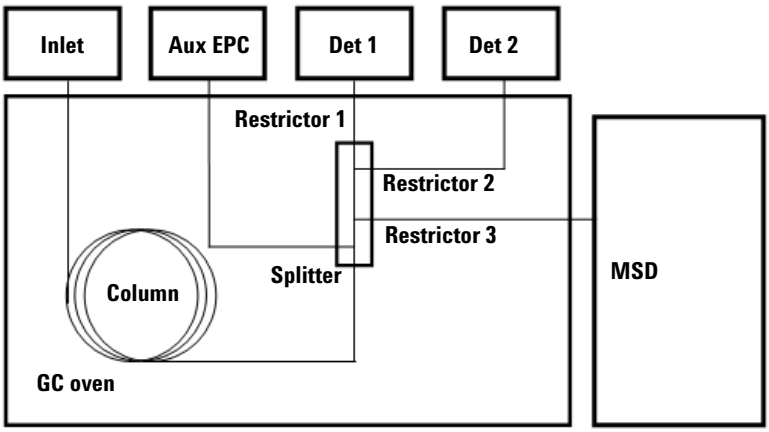

Figure 1 Splitter plumbing

The column flow mixes with the Aux EPC makeup flow in the splitter. This mixture then flows through lengths of fused-silica tubing to each detector (or alternate destination). These tubes act as flow restrictors. While the flows through the restrictors may change with oven temperature, the *ratio* of the flows at any temperature remains constant as long as all restrictors experience the same temperature change.

Details

The G3183B kit addresses several limitations of previous approaches to splitting column effluent between three detectors.

Metal ferrules

The splitter uses soft metal column ferrules which eliminate air leakage into the sample stream. Unlike polyimide, metal ferrules do not loosen upon thermal cycling of the oven. They also do not outgas contaminants or shed particles (like graphite), or extrude into fittings and cause chromatographic problems, nor do they require periodic retightening.

Capillary flow technology

The splitting hardware is based on Capillary Flow Technology (CFT). This provides very low dead-volume connections between the column end and the three detector restrictor tubes. The thin metal plate of the splitting hardware has fast thermal response and is mounted firmly on the oven wall for maximum ease of use. The interior plate surfaces are deactivated to prevent adsorption or decomposition of active compounds.

Programmable pressure and flow

The splitter uses a source of makeup gas supplied by electronic pneumatics control (EPC). The 7890A GC provides the ability to control the pressure of up to 6 channels, so the GC can adjust the makeup gas supply into the splitter as needed to maintain either constant or programmed pressure, or flow. Programmable control of pressure or flow at the column inlet provides the opportunity for constant flow control modes and backflush. It also simplifies choice of splitter parameters, allowing all aspects of the chromatographic setup to be accurately calculated. Because the EPC pressure can be time programmed, useful operations like backflushing unwanted heavy materials from the column and changing columns in MS systems without venting are possible.

Calculation of Chromatographic Parameters

When adding a purged splitter to the end of a column, the outlet pressure is higher than the original pressure. In order to regain the original retention times, the inlet pressure must be raised. Determining the new (higher) inlet pressure is a straightforward process, and with flow controlled method software the process is automated.

Because the pressure at the split point is known and controlled, the chromatographic parameters can be calculated/modeled before setup. This is especially important with GC/MS setups, to avoid exceeding recommended flow rates of carrier gas into the MS. If a method that was originally developed on an MS is converted to a splitter setup, a new inlet pressure can be calculated to produce retention times very similar to the original method.

Requirements

Carrier gas

The choice of carrier gas is dictated by the chromatographic requirements. Hydrogen and helium are the most common choices for capillary columns. See your GC documentation and other sources for additional carrier gas information.

Makeup gas

The makeup gas is usually the same as the carrier gas.

If connected to a Mass Selective Detector (MSD), the splitter purge gas protects the MSD when the column is disconnected by blanketing the open connection. In addition to the flow of blanket gas to the MSD, excess gas is vented through the opening to the GC.

Gas chromatograph

The splitter mounts in an Agilent 7890A GC.

The splitter requires an electronically controlled pressure source such as the Three Channel Pressure controller (option 301 or 308), a Pneumatics Control Module (PCM), or an Auxiliary Pressure controller (Aux EPC).

For effluent splitters, be sure that a low flow resistance, single-ring frit is installed in the Aux EPC module channel used for the splitter.

Microsoft Excel 97

The calculator requires Microsoft Excel 97 (or later), which is not supplied with this kit.

Parts Supplied

The G3183B kit contains the following parts (Table 1 and Table 2)

Table 1 Three-way splitter with makeup gas kit (G3183-64010)

Description	Quantity
3-way splitter with makeup gas, inert	1
Capillary column spring clip	4
Oven wall bracket for CFT plate devices	1
Union SS 1/16-inch tubing	1
Plug for microfluidic manifold or unions	1
Deans switch supplies and spares kit (Table 2)	1
Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVDs	1

Table 2 Deans switch supplies and spares kit (G2855-60150)

Description	Quantity
Ferrule pre-swage tool, capillary flow	1
Capillary tubing cutter, 4/pk	1
Magnifier, 3x, 6x paddle plastic	1
Fused silica, deactivated retention gap, 0.100 mm x 5 m	1
Fused silica, deactivated 0.200 mm x 5 m	1
Fused silica, deactivated 0.250 mm x 5 m	1
Fused silica, deactivated 0.080 mm x 10 m	1
Fused silica, deactivated 0.15 mm x 5 m	1
Siltite ferrules, 0.53 mm column, 10/pk	1
Siltite ferrules, 0.1-0.25 mm column, 10/pk	1
Siltite ferrules, 0.32 mm column, 10/pk	1
Internal nut, CFT capillary fitting	6
Column storage fitting	4
Stainless steel wire, 0.015-inch diameter x 40 mm, 10/pk	1
Plug for microfluidic manifold or unions	3
Protective cap, polyethylene, 1/16-inch id	4

Parts Identification

Most of the kit parts are easily recognized. The unique ones are identified in Figure 2.

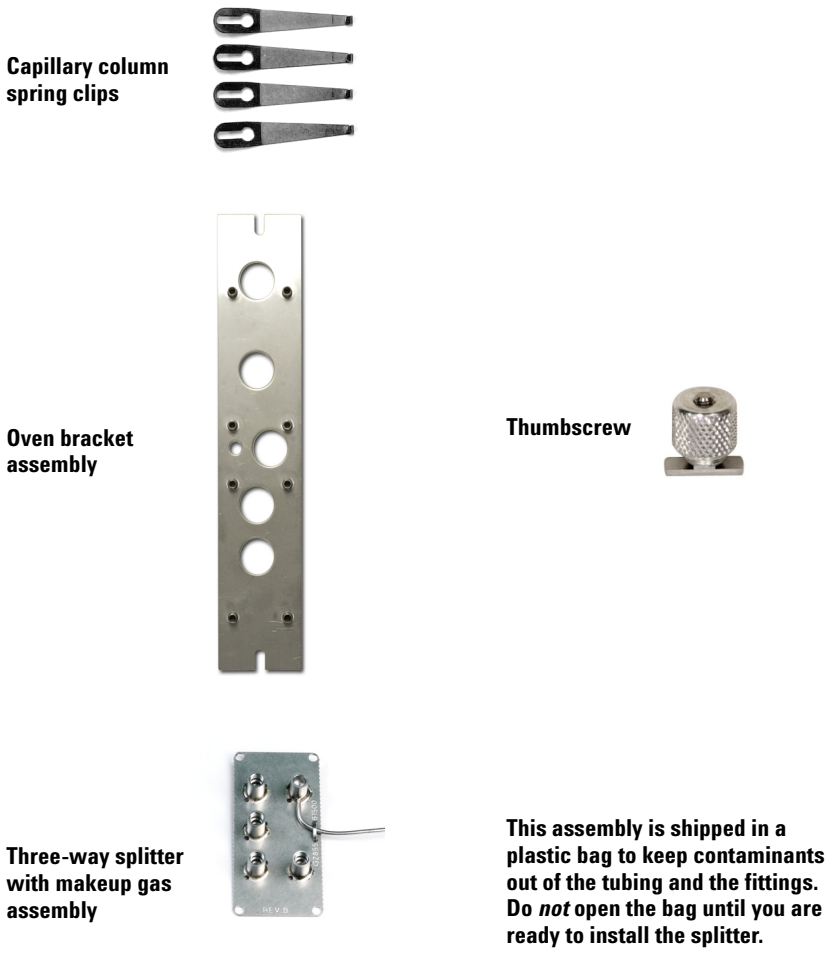

Figure 2 Parts identification

Tools Required

- Diagonal cutter, large
- Screwdrivers, Phillips
- 5190-1442 Precision Tubing Cutter (recommended)

Pressure Units

All pressure figures in this manual are given in pounds per square inch (psi).

Gauge pressure (psig)

This is the pressure as measured by most pressure gauges. It is the pressure *in excess of* atmospheric pressure.

Absolute pressure (psia)

This is the actual pressure relative to vacuum. It is the sum of the gauge pressure and the atmospheric pressure.

Atmospheric pressure

Atmospheric pressure at sea level = 14.696 psia = 101.32 kPa

Conversion

kPa = psi × 6.8947

2 Installation

This chapter describes the procedures for installing the splitter hardware in a 7890A GC.

Safety

Many areas and parts of the GC operate at temperatures high enough to cause serious burns. These include, but are not limited to components inside the oven, and components connected to the oven such as:

- The inlet
- The detectors
- Any nut attaching a column to an inlet, to a detector, or to the splitter plate

Cool these areas to room temperature before working on or around them.

The inlet and detector cool faster if you set their temperatures to **OFF** and set oven temperature to room temperature (which keeps the oven fan and exhaust flaps in operation). Turn the oven off when room temperature is attained.

Also, be careful when working behind the instrument. During oven cooldown, the GC emits hot air exhaust which can cause burns.

Once cool, all pressure zones should be set to **OFF**. Caution should be exercised when venting residual pressure that may still be present.

Software Installation

You must install the provided calculator software in Table 3 to select the appropriate restrictors for your splitter and GC configuration.

Table 3 Calculator software

Software	Uses	Where to find it
GC front keypad or GC method editor	• Find current column flows and pressures.	• GC • Agilent data system method editor
Pressure Flow Calculator	• Estimate flow or pressure for a capillary column at given conditions, for example new splitter or inlet head pressure or flow rates. • Estimate minimum and maximum flows through restrictors.	• Agilent Instrument Utilities software *
Method Translator	• Estimate new flow or pressure conditions for a column so that you keep the same holdup time or elution order. • Estimate flow or pressure needed to achieve desired column flow.	• Agilent Instrument Utilities software *
Effluent Splitter Calculator	• Select diameters and lengths of restrictor tubing based on desired split ratio, temperature, and column outlet pressure.	• **G3183B_splitter_calc.xls**, located on the Agilent GC and GC/MS Hardware User Information and Instrument Utilities DVD in the G3183B Tools directory.

* Install the Instrument Utilities software from the Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVD.

Open the **G3183B_splitter_calc.xls file** directly from the Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVDs, or copy it to your computer as desired.

Hardware Installation

If using a 6890 Series GC see “Prepare the 6890 GC” on page 50.

WARNING **Cool the column oven, inlet, and MSD transfer line before starting. They may be hot enough to cause burns.**

WARNING **Turn the power off and disconnect the power cord before proceeding.**

Install the column clips

Install the four column clips on the oven shroud (Figure 3):

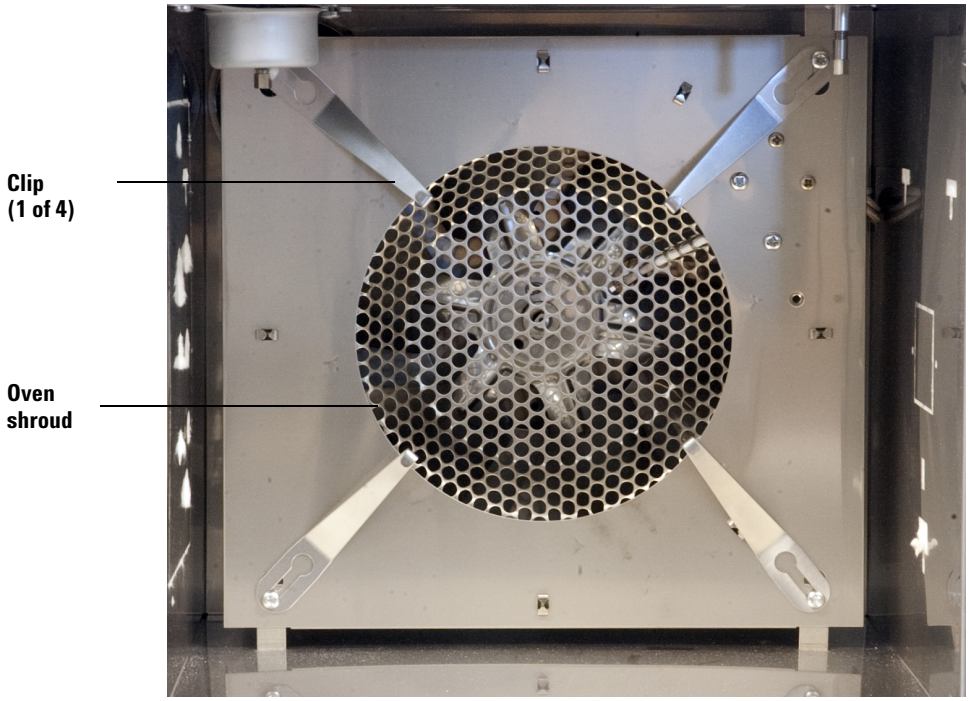

Figure 3 Oven clips

1. Loosen the four corner screws, but do not remove.
2. Slip each corner screw through the large hole on the clip.
3. Slide the clip so that the screw is positioned in the slot.
4. Tighten the screws enough to hold the clips in place. Once the column is installed, fully tighten the four corner screws to secure the clips and column to the oven wall.

Install the bracket and splitter

The body of the splitter may be discolored as a result of the deactivation process. This is not a defect.

The plate can accommodate several CFT devices. It can be positioned in any of four positions (left, right, front, back). The splitter can be positioned on the high or low position of the plate.

The splitter is usually installed on the front-right side of the oven, but can just as well be positioned in any of the other three positions based on the application needed.

1 Place the bracket against the side of the oven. The two notches should be up and the standoffs should face the center of the oven.

2 Use the T-shaped thumbscrew to fasten the bracket to the T-slot in the oven wall (Figure 4). Pull the thumb screw sleeve back to facilitate slipping it into the slot on the oven wall.

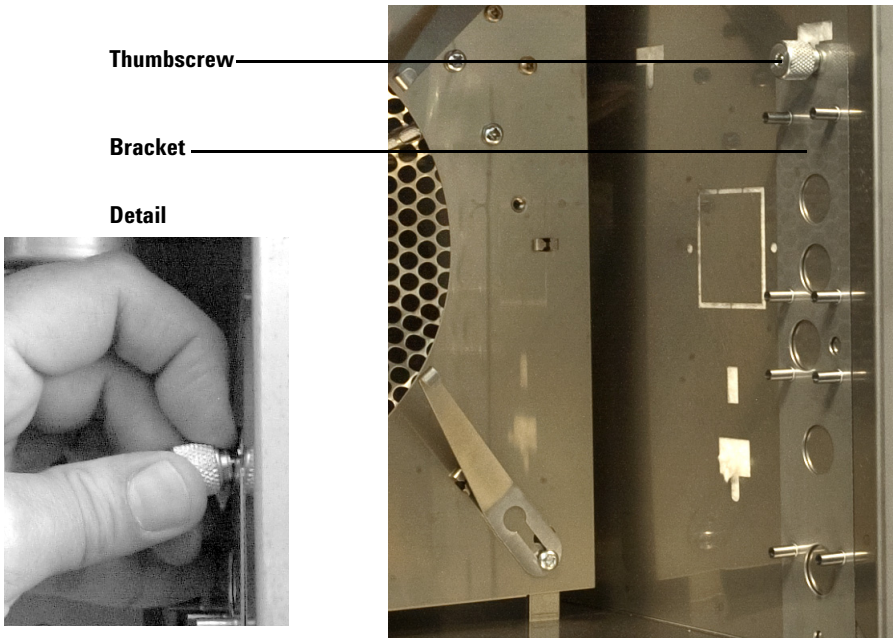

Figure 4 Installing the bracket

3 Tighten the thumb screw and push down on the plate to keep the plate firmly seated against the bottom of the oven. Be sure that the “T” behind the thumb screw clip is horizontal.

CAUTION Use extreme care to prevent any fragments of insulation or other material from entering the makeup gas tubing or the fittings on the splitter assembly. Such materials could block the internal passages in the splitter or the bore of the capillary restrictors.

4 Open the plastic bag and remove the splitter tube, but leave the assembly in the bag to protect it from dust. If not done already, install a plastic cap on the end of the makeup gas tubing. Place small pieces of tape or parafilm over the other open connections of the splitter to prevent dust from entering.

NOTE Use the large plastic bag to capture the insulation that falls from the oven ceiling. Lay the bag against the oven floor before running the tubing through the oven ceiling.

5 Poke an item such as a screwdriver down through the oven ceiling first to help create a passage for the makeup gas tubing.

6 Run the end of the makeup gas tubing up through the hole in the oven ceiling so that the end of the tubing comes out of the hole in the oven top. For a 6890 GC, run the tubing through the valve box blanking plate.

7 Preliminarily route the tubing against the oven wall and top to keep it clean for future maintenance. It should run behind the back detector location.

8 Screw the splitter assembly to the bracket (four screws). See Figure 5.

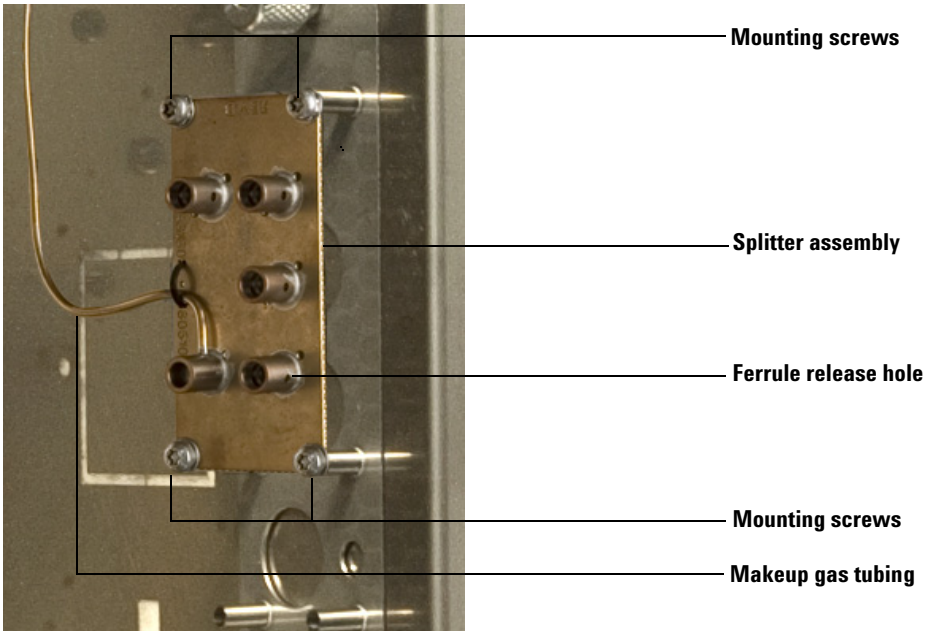

Figure 5 Installing the splitter assembly

9 Once you have installed the splitter and makeup gas tubing, route the tubing in the oven, securing as needed. Trim the tubing above the oven top, leaving at least 10 cm.

CAUTION To avoid kinks or sharp bends in the tubing, bend the tubing over an object such as your thumb.

When routing the tubing, be sure to consider the following:

- Even though it is important for best pressure control to minimize the length of connecting tubing, you should leave enough tubing for future relocation of the splitter and for easy connections.
- Be careful to not pinch or deform the inner diameter of the tubing.
- Bend corners with a minimum radius of 1 cm to avoid inner diameter crimping.
- To save excess tubing for future use, coil the excess tubing around an object like a can or bottle (see Figure 6).

Figure 6 Coil tubing around a can or other cylinder

Connect the makeup gas supply

Connect the makeup gas source to the Auxiliary Pressure controller or Pneumatic Control Module using the 1/16-inch straight union. Replace the stainless steel default ferrules (front and back) with the separate Vespel ferrules provided in the kit. Vespel ferrules provide a more reliable and leak-free connection that is also more reusable than the stainless steel ferrules.

Cut excess tubing (minimize the extraneous length of tubing between the pressure source and the splitter plate). However, be sure to leave enough tubing for a clean routing path along the top of the oven and away from the other devices that may be mounted there, and for easy assembly/disassembly when changing configurations or troubleshooting.

To supply the makeup gas from an Auxiliary Pressure controller or PCM

NOTE

For a clean union, make sure the tubing ends are cut as square as possible, and do your best to avoid crimping.

The use of Precision Tubing Cutter 5190-1442 facilitates proper tube cutting.

1. Ensure that the zero-restriction (open frit) is installed in the output channel. See your GC manual for details.
2. Connect the tubing block to the selected channel of the Auxiliary Pressure controller or PCM and route the tubing neatly across the back of the GC oven top. Cut off excess tubing, keeping enough for future potential reconfiguration and troubleshooting.
3. Zero the pressure sensor for the selected channel. (On the GC keypad, go to **Options > Calibration**, scroll to the module, then press **[On/Yes]**.
4. First remove the front/back ferrule set that comes installed with the union, then connect to the 1/16-inch straight union using Vespel ferrules.
5. Turn on the column inlet and Aux EPC/PCM sources, and leak check all fittings.
6. Use the Instrument Utility software to update the Auxiliary Pressure controller or PCM PID constants according to the application instructions.
   - Update only the channel used by the splitter.
   - If using the Instrument Utilities Backflush Wizard, the wizard may recommend PID values. Otherwise, use the QuickSwap PIDs with the zero restriction frit, or the standard PIDs with the low restriction 1-ring frit.

This completes the hardware installation.

3 Operation

This chapter describes how to correctly configure and operate the splitter using a 7890A GC.

Preparing the Splitter

To prepare the splitter for use with a method, do the following:

1. Select the appropriate splitter configuration (restrictor lengths) for your GC configuration. (See “Selecting a Splitter Configuration” on page 23.)
2. Add a SilTite ferrule to one end of the uncoated, deactivated fused silica tubing.
3. Cut the tubing to a length slightly longer than needed.
4. Add the nut and ferrule appropriate for the detector or other uses.
5. Trim the tubing to the final target length. (See your 7890A GC user documentation for details.)
6. Check your Aux EPC or PCM module to make sure the open frit is installed.
7. Check that the pressure sensors are at +/- 0.001 psi for the selected channels when the line is open and the EPC is off.
8. Install the restrictors and column to the splitter. (See “Backflushing the Column” on page 46.)
9. Install the restrictors to the detectors. (See your 7890A GC or MS/MSD user documentation for details.)
10. Configure the new columns in the 7890A GC using the data system method editor (or GC front panel for standalone setups).
11. Update your method for any new setpoints. Typically, the only change needed is to update the splitter flow or pressure. If correctly configured, the GC adjusts the analytical column flow or pressures accordingly.
12. Check for leaks.
13. Test the setup.

Selecting a Splitter Configuration

The combination of relative restrictor lengths and diameters, plus the GC flow and temperature programs, determines the split ratio leaving the splitter and entering the detectors. This section describes:

- The general procedure for determining reasonable restrictor sizes for a splitter in the 7890A GC
- The new method settings you will need and how to determine them
- The flow characteristics that affect your decisions

What impacts the split ratios?

In addition to the restrictor characteristics, the following also impact the splitter split ratio:

- Column dimensions, gas type, pressures, and restrictor temperature zones
- Flow mode for the column and splitter restrictors

What impacts overall success?

Getting the correct relative flows into the detectors that need them.

If using a flow-sensitive detector, such as an MS or MSD, the resultant flow rate should be in the optimal range for the detector. For the Agilent Triple Quadruple MS and MSD, the optimal flow rate is 1.0 – 1.5 mL/min. Using lower or higher flow rates will reduce sensitivity. Refer to the detector's user documentation for details.

Split ratios

The split ratio should be such that flow sensitive detectors, such as an MS or MSD, get their optimal flow. Next, you want concentration-sensitive detectors, such as the TCD and ECD, to receive as little makeup gas as reasonable. Using less makeup gas with these detectors keeps sensitivity higher.

The amount of makeup gas added in the splitter

Gas chromatography works better when the compressibility of gas can be ignored. The amount of makeup gas used, even for mass-sensitive detectors that can accept high flow rates (such as the FID), can sometimes cause changes in detector behavior. If possible, it is best to use the smallest amount of makeup gas as necessary to move the column effluent through the splitter and into the detectors.

Using too little makeup gas can lead to problems with backpressure or insufficient flow to the detectors.

The ability to calculate flow rates

It is difficult to determine column flow in columns which exist in more than one thermal zone. An example of this type of column is a restrictor that runs from a splitter and into the MSD through the transfer line. The GC oven contains half the transfer line, and can be temperature programmed. The MSD transfer line contains the other half of the restrictor, and is typically isothermal. The transfer line temperature is also typically higher than the maximum oven temperature used. Newer 7890A GC firmware, A.01.11 and higher, can calculate flows through columns which exist in more than one thermal zone.

With older 7890A GC firmware and with other GC types, we can only estimate the flow through this type of restrictor by calculating the boundary values:

Case (1): flow assuming the whole restrictor is in the internal transfer line

Case (2): flow assuming the whole restrictor is in the GC oven at the low oven temperature, and

Case (3): flow assuming the whole restrictor is in the GC oven at the highest oven temperature.

This estimation provides upper and lower limits on the flow. The real flow rate will vary during the oven program between cases (1) and (2). The lowest actual flow rate will be between cases (1) and (3). So, we try to select a restrictor where we are reasonably sure that case (2) does not exceed the MSD maximum flow rate, and that cases (1) and (3) are both in the optimal flow range for the MSD.

Control modes

On the 7890A GC, you can control the splitter and the analytical column using either pressure or flow control modes. If using a mixed mode system, where you control the splitter using pressure but the column using flow rate (or vice-versa), you may get into trouble if not careful. For example, consider using constant flow control for the column and constant pressure control for the splitter. As the oven temperature increases, the inlet head pressure will increase to maintain the main column flow. However, due to increasing temperature, flow through the restrictors at a constant pressure will go down. If the total flow out of the restrictors becomes less than the constant column flow, the setpoint pressure will be exceeded and no gas will flow from the EPC. This in turn can cause peak tailing, ghost peaks, etc.

NOTE Split ratio remains the same (approximately) in all scenarios.

Therefore, it is highly recommended that you use the same mode for both the column and restrictor. Remember that only one of the restrictors (the first one listed in the configuration) can be in constant flow mode. If in this example we used all constant flow control modes, then the splitter pressure would increase with the temperature program. The inlet and splitter EPC modules would independently but correctly compensate for the temperature changes.

Guidelines

Given the known issues, here are some guidelines for selecting restrictors.

1. If using a flow-sensitive detector, select a restrictor so that this detector receives its optimal flow. Then select the other restrictor to achieve desired split ratio(s) to any other detector(s), whether concentration or mass-sensitive.
2. If using a concentration sensitive detector, such as a TCD or ECD, select its restrictor to minimize the addition of makeup gas. Then select restrictors for any remaining mass-sensitive detectors.
3. Use the same control mode for the splitter (Aux EPC or PCM) and the analytical column. If not, select restrictor sizes that maintain acceptable flow rates and ratios throughout all phases of the temperature program. (Highest flows will be at lowest oven temperatures.)
4. Select restrictors so that the flow rates or pressures are within acceptable operating ranges of all detector types throughout the oven temperature program.
5. The splitter total output flow needs to be greater than 10% higher than the analytical column flow. This guideline ensures that there is always a positive flow of makeup gas through the splitter, and provides a safety margin to compensate for inaccuracies in tubing dimensions. This guideline applies to the total splitter output flow both at the initial and at the highest oven temperatures.
6. The restrictors need to be long enough to install between the splitter plate and the detectors or transfer line assembly. For an MS or MSD connection, typically you need at least 0.8 m length. For most other detectors, typically you need at least 0.3 m of length.

Typical Configurations

The important parameters when setting up a splitter are the lengths and diameters of the restrictor tubes that go to the three detectors. The dimensions of the restrictors are chosen to give the desired flow (split) ratio, flow to the detector, and to minimize peak broadening.

The splitter restrictors are chosen based on:

- The range of column flows that will be used with the method
- The operating pressure of the three detectors
- The flow rate requirements of the three detectors

Table 4 lists typical splitting configurations. Table 5 on page 27 shows the resulting gas flows. All calculations assume helium as the carrier gas and a splitter pressure setting of 3.8 psig.

Table 4 Splitting configurations

Config.	Detector type			Flow ratio		Restrictor 1		Restrictor 2		Restrictor 3	
				Det2 to Det1	Det3 to Det1	id, mm	length, m	id, mm	length, m	id, mm	length, m
1	MSD-D*	atm†	atm	1	1	0.18	2.89	0.18	1.06	0.18	1.06
2	MSD-T‡	atm	atm	1	1	0.18	1.44	0.18	0.53	0.18	0.53
3	MSD-D	atm	atm	1	0.1	0.18	2.89	0.18	1.06	0.10	1.01
4	MSD-T	atm	atm	1	0.1	0.18	1.44	0.18	0.53	0.10	0.51
5	MSD-D	atm	atm	2	2	0.18	2.89	0.18	0.53	0.18	0.53
6	MSD-T	atm	atm	2	2	0.18	1.44	0.20	0.41	0.20	0.41
7	MSD-D	atm	atm	0.5	0.5	0.18	2.89	0.18	2.13	0.18	2.13
8	MSD-T	atm	atm	0.5	0.5	0.18	1.44	0.18	1.06	0.18	1.06

* MSD-D MSD with diffusion pump or standard turbo pump (2 mL/min flow capability)

† atm Atmospheric pressure detectors such as FID, TCD, ECD, FPD, and NPD

‡ MSD-T MSD with performance turbo pump (4 mL/min flow capability)

Table 5 Splitter flows

Config.	40 °C			200 °C			300 °C		
	Flow R1, mL/min	Flow R2, mL/min	Flow R3, mL/min	Flow R1, mL/min	Flow R2, mL/min	Flow R3, mL/min	Flow R1, mL/min	Flow R2, mL/min	Flow R3, mL/min
1	2	2	2	1	1	1	0.71	0.71	0.71
2	4	4	4	2	2	2	1.42	1.42	1.42
3	2	2	0.2	1	1	0.1	0.71	0.71	0.07
4	4	4	0.4	2	2	0.2	1.42	1.42	0.14
5	2	4	4	1	2	2	0.71	1.42	1.42
6	4	8	8	2	4	4	1.42	2.85	2.85
7	2	1	1	1	0.5	0.5	0.71	0.36	0.36
8	4	2	2	2	1	1	1.42	0.71	0.71

To use the tables, select the configuration you wish to set up. For example, **Configuration 1** splits column effluent equally between two atmospheric pressure detectors (FID, TCD, ECD, FPD, and NPD) and an MSD. To plumb this system, connect a 2.89-m length of 0.18-mm id uncoated deactivated fused silica tubing to the MSD and two 1.06-m lengths of the same tubing to the two atmospheric pressure detectors. This configuration is specifically designed for MSDs with a diffusion pump or standard turbo pump (2 mL/min maximum flow).

The makeup supply (either Aux EPC or PCM module) is set to 3.8 psig. This will add sufficient makeup flow to the column flow to maintain the splitter (and thus the column outlet) at 3.8 psig. Column flow can be varied from 0 to a maximum flow which is determined by the upper temperature of the GC oven program.

If **Configuration 1** is used with a method that programs to 200 °C using helium, the flow through each restrictor at 200 °C will be 1 mL/min. The total flow will be 3 mL/min. The maximum column flow should be equal to the total flow minus about 1 mL/min to ensure that there is some flow for the makeup supply to regulate with.

The column flow at 200 °C should be no more than 2 mL/min. This becomes important when the column is run in constant flow mode. If constant flow mode is used with **Configuration 1** and the method is programmed to 300 °C, the column flow should not exceed 1.13 mL/min ([0.71 + 0.71 + 0.71] –1).

For constant pressure methods, first find the maximum flow as above. Use the GC, ChemStation, Flow Calculator Software or the Method Translation Software to find the inlet pressure that gives the maximum flow at the upper temperature of the method (make sure the column outlet pressure is set to 3.8 psi for the calculation).

For example, if a 30 m × 0.32-mm id column is used with **Configuration 1**, using helium carrier and programming to 300 °C, the pressure that gives a flow of 1.13 mL/min is 28.4 psig. This is the maximum pressure at which the inlet should be set. The inlet should not be set at or below 3.8 psig.

The performance turbo on the MSD is preferred because it allows splitter configurations with higher permissible column flows. **Configuration 2** is set up for the performance turbo and allows almost three times the column flow ([1.42 + 1.42 + 1.42] – 1) = 3.26 mL/min at 300 °C).

If you decide to use a typical configuration, note the restrictor dimensions from Table 4 on page 26 and proceed to "Installing a Column into a Splitter" on page 42.

Splitting to an MSD

Note that the maximum column flows for an MSD are quite low. This limit is imposed by the rating of the turbo or diffusion pump. Configurations with split ratios greater than one can be used but peak broadening or tailing may occur if the flows to the other detectors becomes too low.

In practice, the column flow can be set to within 0.5 mL/min of the total flow if necessary.

Split ratios to the MSD greater than one are very limited due to these flow considerations and should be avoided if possible.

Restrictor id and length

1. Run the Effluent Splitter Calculator and enter the following information. The calculator provides a list of possible restrictors.
   - **Column flow**. Use the ChemStation, GC, Flow Calculator, or Method Translation Software to determine the column flow in mL/min (with the column outlet at 3.8 psig) at the initial oven temperature.
   - **Initial oven temperature**. This is the temperature setpoint for an isothermal method or the initial temperature for a programmed method.
   - **Carrier gas type**. Enter Helium, Nitrogen, or Argon.
   - **Detector operating pressures (psia)**. The operating pressure must be in absolute units. Most detectors (FID, TCD, ECD, NPD, and FPD) operate at atmospheric pressure (14.696 psia). Exceptions are the MSD (0 psia) and AED (16.196 psia).
   - **Flow Ratios, Detector 2 to Detector 1 and Detector 3 to Detector 1**. These are the desired split ratios among the detectors. Usually this number is one, meaning the effluent divides equally among the detectors. This can be adjusted to higher values, but should normally not exceed five.
   - **Splitter (column outlet) pressure (psig)**. This is the desired pressure at which the splitter (and thus the end of the column) will operate. It can be set between 2 and 4 psig, but is usually set to 3.8 psig. This number can be varied to obtain an acceptable combination of restrictors that will have sufficient flow velocity to give good peak shapes.
2. Choose the id tubing that gives a length closest to (and at least) 0.3 m for most detectors and 0.8 m for MSDs. The green fields with tubing diameters in mm can be edited if you have other sizes of deactivated tubing available.

Maximum and minimum flows

The maximum suggested flow for MSDs depends on the vacuum pump used. For diffusion pump and standard turbo systems, the flow should not exceed 2 mL/min. For performance turbo systems, the flow should not exceed 4 mL/min. These flow limits restrict the column flows and split ratios that can be used with MSDs.

Try to have the flow through each restrictor tube at least equal to the suggested minimum flow in Table 6. Restrictors that have less flow will still work, but peak broadening or tailing may result.

Table 6 Suggested minimum restrictor flows

	Minimum carrier gas flow, mL/min			
Restrictor internal diameter, mm	**Helium**	**Hydrogen**	**Nitrogen**	**Argon**
0.10	0.400	0.500	0.125	0.110
0.18	0.720	0.900	0.225	0.198
0.20	0.800	1.000	0.250	0.220
0.25	1.000	1.250	0.313	0.275
0.32	1.280	1.600	0.400	0.352
0.45	1.800	2.250	0.563	0.495
0.53	2.120	2.650	0.663	0.583

1. The makeup flow is listed in cell B 36 of the effluent splitter calculator. You should have at least 0.5 mL/min for stable pressure regulation. Note that this value will decrease as the oven temperature programs up.
2. Use the **Column Pressure/Flow Calculator** to determine the flow through each restrictor at the maximum oven temperature of the method, add them and subtract the calculated column flow at that temperature. This value should be greater than 0.5 mL/min.

Column outlet pressure

The GC needs to know the pressure at the end of the column to be able to calculate column flows. Use either the GC keyboard or the ChemStation to set the outlet pressure for the column to 3.8 psig.

Inlet pressure

If this is a method used previously, you may want to reset the inlet pressure to give similar retention times with the new column outlet pressure. Do this by calculating the inlet pressure needed to keep the void (holdup) time the same as the previous method. For constant inlet pressure methods, this will also keep the elution order the same. The Method Translation Software tool or the Flow Calculator tool can be used to do this calculation.

An Example

Assume we have a method that uses an HP-5MS (p/n 190915-433) column (30 m × 250 µm id × 0.25-µm film thickness) to measure pesticides with an MSD performance turbo system. The initial oven temperature is 70 °C and is programmed to 280 ^{o}C. The method runs in constant pressure mode at 19.44 psig inlet pressure and the carrier gas is helium. The initial column flow listed by the ChemStation is 2.1 mL/min.

We want to create a new splitter method with the column effluent split 1:1:0.1 between the MSD (detector 1), an FPD (detector 2), and an ECD (detector 3). We would also like to preserve the retention times and relative elution order in the new method.

Column flow

Since the column outlet pressure will be much higher in the new method, the first step is to calculate the new inlet pressure and the resulting column flow. The Method Translation software (Figure 7 on page 32) is useful for this. Use the **None** mode and check the button to make the hold-up times the same.

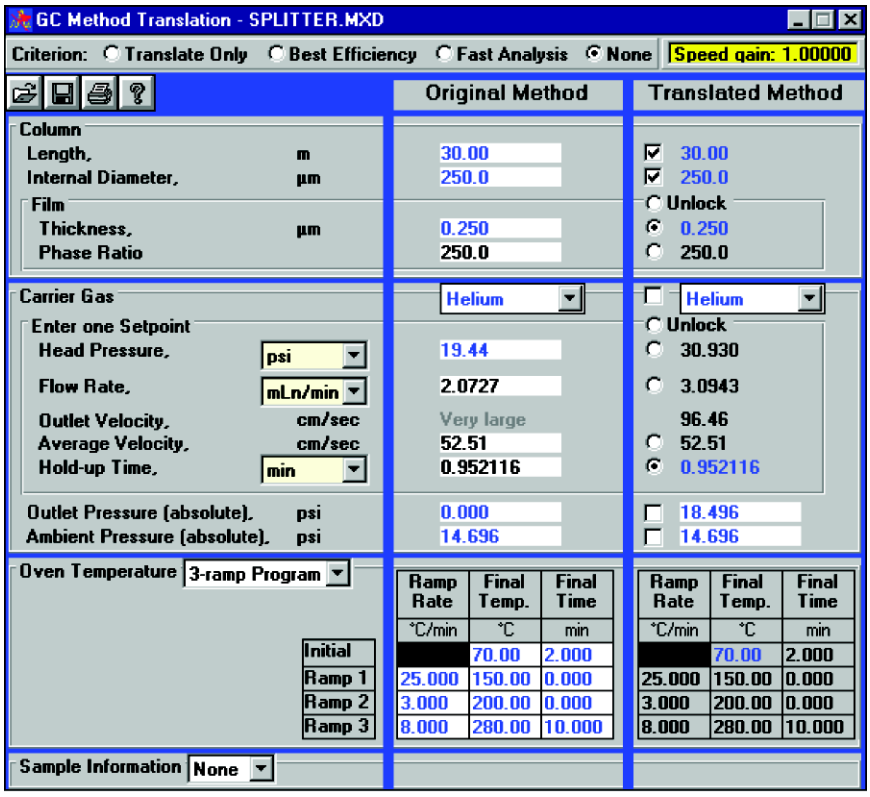

Figure 7 Calculating column flow

The outlet pressure entered for the new splitter method must be in absolute pressure units. Since the outlet of the column will be 3.8 psig, we need to convert this to psia for the method translator. Absolute pressure = gauge pressure + 14.696. Therefore, 3.8 + 14.696 = 18.496 will be entered.

The calculated inlet pressure for the new splitter method is 30.93 psig and the new column flow is 3.09 mL/min.

Select restrictors

Open the **G3183B_splitter_calc.xls** file directly from the Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVDs, or copy it to your computer as desired.

We will choose to have 4 mL/min go to the MSD initially. This flow is acceptable with a performance turbo system. Fill in the input column as shown (Figure 8) with the MSD assumed to be Detector 1, the FPD as Detector 2, and the ECD as Detector 3.

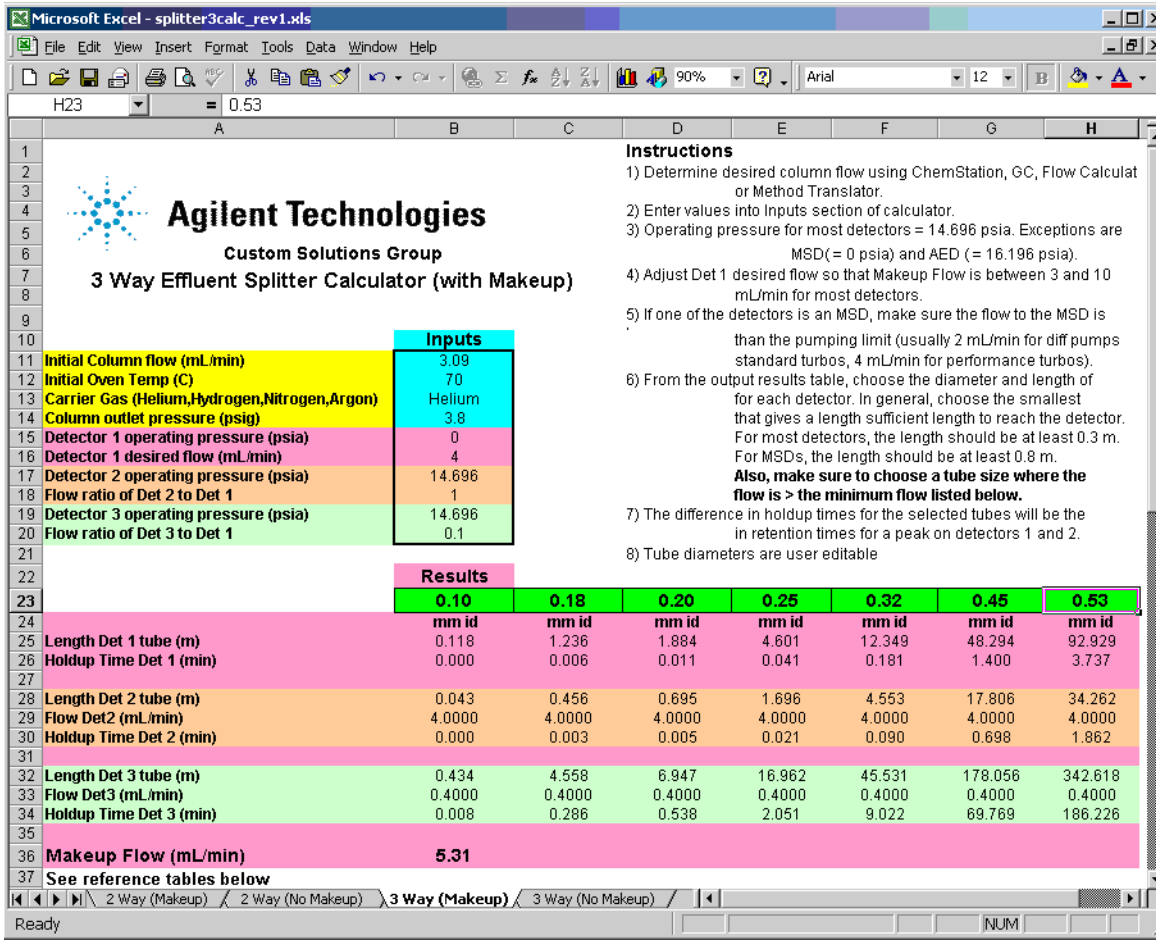

Figure 8 The Effluent Splitter calculator

The calculator lists the lengths required for the different sizes of uncoated, deactivated, fused-silica restrictor tubing available. Choose the id tubing that gives the shortest length of at least 0.3 m for most detectors and 0.8 m for MSDs. In this case 0.18-mm id is the choice for the MSD and the FPD. A length of 1.236 m is calculated for the MSD restrictor and 0.456 m for the FPD restrictor. For the ECD, a 0.434-m length of 0.1-mm id tubing is calculated.

Table 5 on page 27 shows that in all cases the flow is higher than or equal to the suggested minimum flow for the tubing diameter and gas type.

Calculate column flow

To find the makeup flow at 280 °C, first find the column flow at 280 °C. The Flow Calculator software (Figure 9) requires that the output pressure be entered in psia. Therefore 18.496 psia (3.8 psig) is entered.

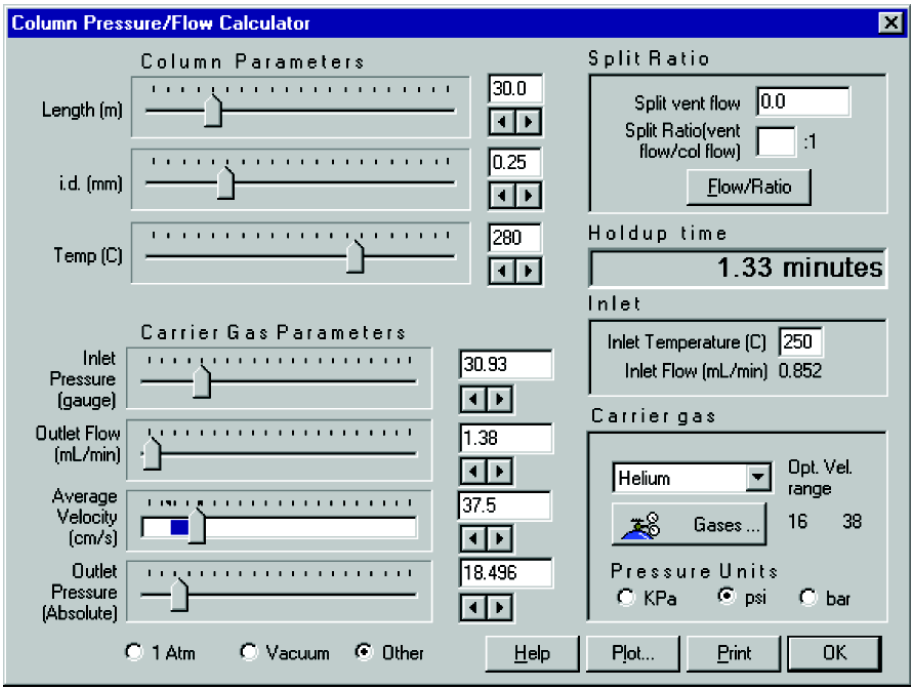

Figure 9 Column flow calculation

The column flow drops to 1.38 mL/min at 280 °C.

Calculate MSD restrictor flow

The flow through the MSD restrictor at 280 °C is calculated to be 1.77 mL/min (Figure 10).

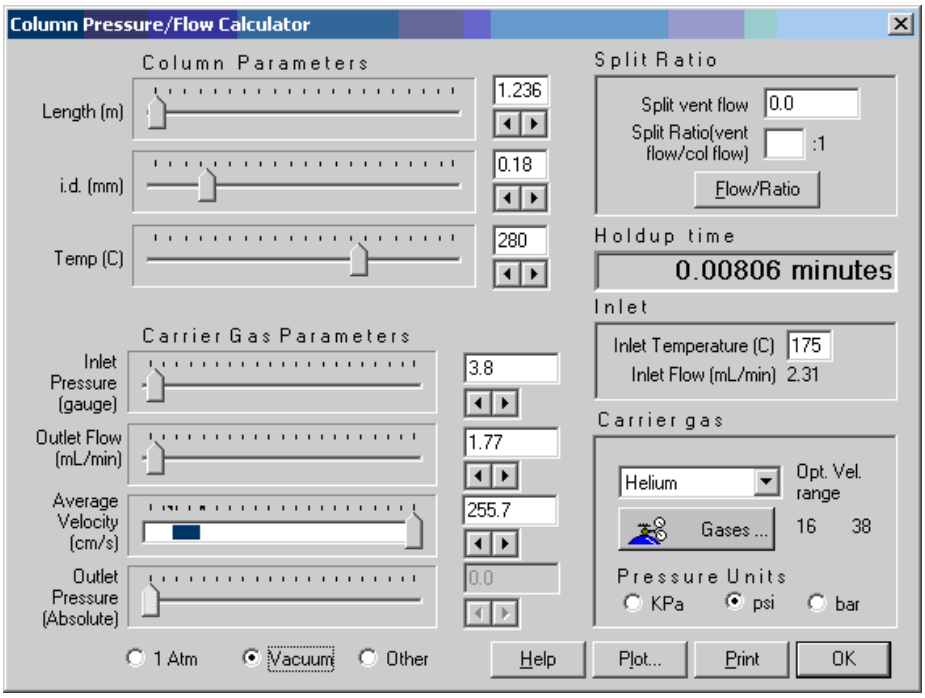

Figure 10 MSD restrictor flow calculation

This flow is higher than the minimum 0.72 mL/min suggested for helium in 0.18-mm id tubing.

Calculate FPD restrictor flow

The flow through the FPD restrictor (Figure 11) is also 1.77 mL/min at 280 °C.

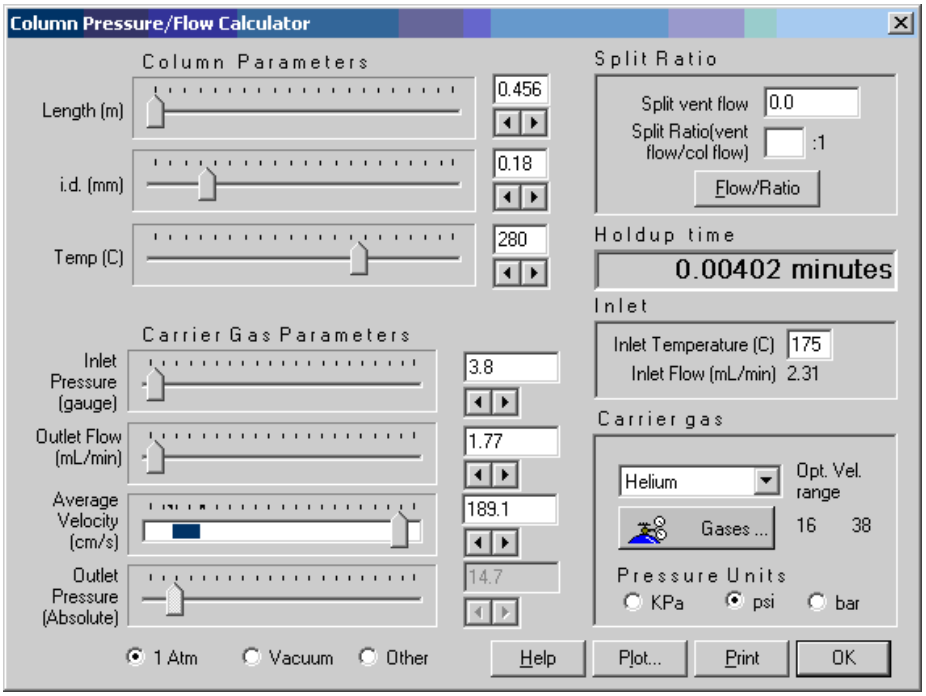

Figure 11 FPD restrictor flow calculation

This flow is higher than the minimum 0.72 mL/min suggested for helium in 0.18-mm id tubing.

Calculate ECD restrictor flow

The flow through the ECD restrictor (Figure 12) is 0.18 mL/min at 280 °C.

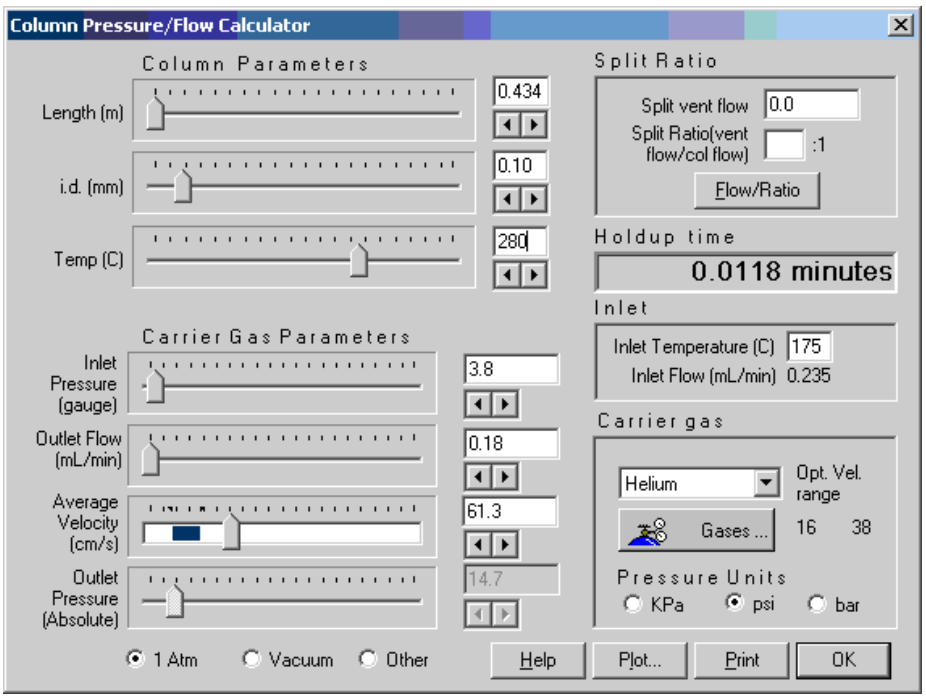

Figure 12 ECD restrictor flow calculation

This flow is below the minimum 0.4 mL/min suggested for helium in 0.10-mm id tubing. In practice, this setup still gives acceptable peak shapes on the ECD, so it will be used.

The calculated makeup flow is then [1.77 + 1.77 + 0.18] - 1.38 = 2.34 mL/min. This should work well.

The configuration can now be installed and used.

Configuring splitter columns

All the splitter columns can be configured on the 7890A GC, allowing it to directly calculate flows into the MSD. For the example in this section, configure the columns as shown in Table 7.

Table 7 Configuring splitter columns

Column	Inlet	Outlet	Heated By
190915-433 HPS-MS 30 m × 250 µm × 0.25 µm	Front inlet	Aux Pressure 1	Oven
Restrictor for MSD 1.236 m × 0.18 mm	Aux Pressure 1	Vacuum	Oven
Restrictor for µECD 0.434 m × 0.10 mm	Aux Pressure 1	Back Detector	Oven
Restrictor for FPD 0.456 m × 0.18 mm	Aux Pressure 1	Front Detector	Oven

Other Uses for Three-Way Splitter Setup

While the most common use of the hardware discussed here is as a three-way splitter, the device can also be used for other purposes. The following configurations are examples.

Three-way splitter

Figure 13 shows the typical three-way splitter configuration for reference.

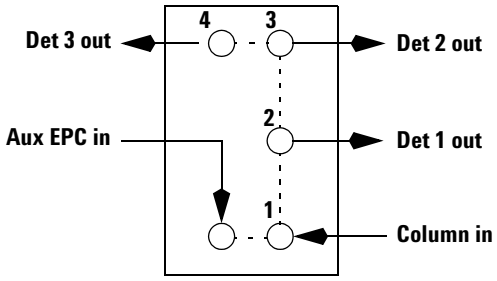

Figure 13 Three-way effluent splitter

Two-way splitter

Figure 14 shows the configuration for a two-way splitter.

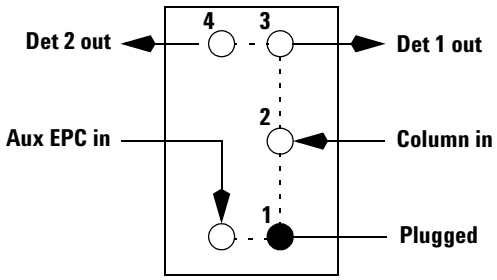

Figure 14 Two-way effluent splitter

Make the plug from a nut and ferrule plus a length of the stainless steel wire from the kit.

In this case, port 1 is plugged and the column is connected to port 2. The two detector restrictors are connected to ports 3 and 4. Calculations for a two-way splitter are similar to those for the three-way. See the manual for the G3180B Two-way Splitter with Makeup Gas provided on the Instrument Utilities DVD for information on setting up a two-way split.

Two columns in/Two detectors out

Figure 15 shows the configuration for a two-column combiner/two-way splitter.

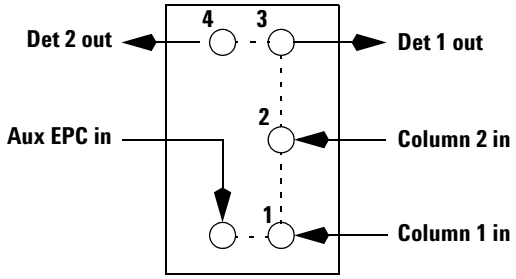

Figure 15 Two columns in/two detectors out

This configuration could be used in a system that allows injection into either inlet 1 or inlet 2.

Two different columns in/Two detectors out

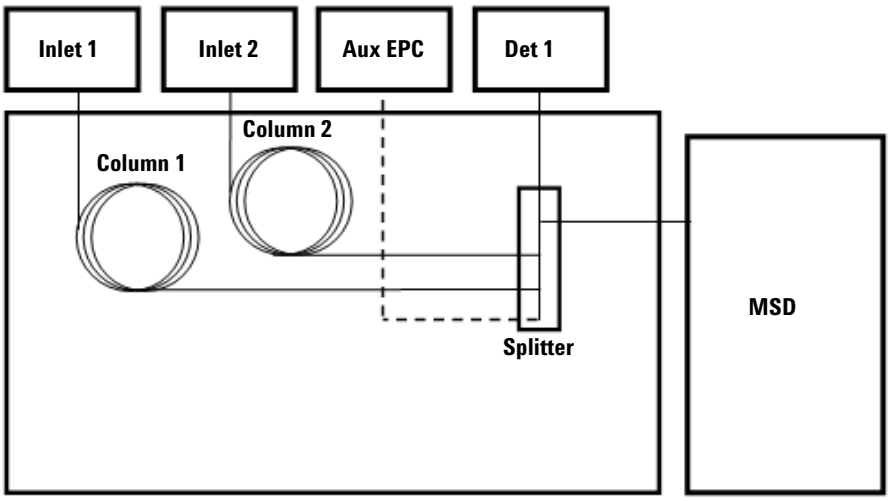

Figure 16 Two different columns in/two detectors out

With this setup, a method can inject into Inlet 1 (and Column 1) while Column 2 is unused. Inlet 2 pressure is set at a low level that produces a small makeup flow (at least 0.5 mL/min) through Column 2. A second method can reverse the situation, purging Column 1 while analyzing with Inlet 2 and Column 2.

This can be useful in laboratories that frequently need to use columns with different phases. Setup is the same as for the two-way splitter, except that column flow in the calculations is now the SUM of the flows from Columns 1 and 2.

Since the bleed from both columns enters the detector simultaneously, low-bleed stationary phases should be used.

Installing a Column into a Splitter

Install the column

1. Hang the analytical column on the column clips or column hanger. The clips hold the outside of the wire “basket” that supports the column. Adjust the clips if necessary.
2. The hanger attaches to the union bracket on the top of the oven. Loosen the bracket, spread and clip in the hanger from each side, and retighten.
3. Connect the column to the inlet following column installation instructions (See your 7890A GC user documentation for more information).

Connect to the splitter

1. Remove any plugs from the splitter connectors.
2. Pre-swage Siltite ferrules on restrictors.
3. Connect the restrictors to the fittings on the splitter (Figure 17 on page 43). Finger-tighten until just snug, then tighten with a wrench an additional 10-15 degrees (Figure 18 on page 43).
4. Connect the restrictors to the appropriate detectors.
5. Connect the column exit to the splitter fitting closest to the incoming purge line (bottom of Figure 17 on page 43). Tighten as you did the restrictors.

CAUTION Arrange the tubing (restrictors and column) so that it does not touch the oven walls. This could create a cold spot.

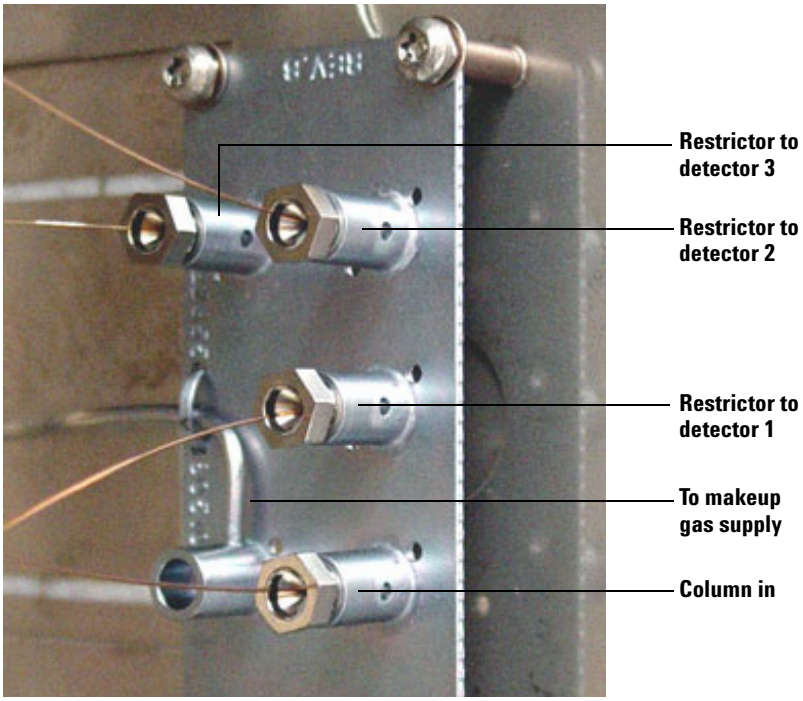

Figure 17 Detector and column connections

CAUTION Do not overtighten the fittings. One-eighth of a turn (about 15 degrees clockwise from finger tight) is usually enough.

Figure 18 Tightening the connections

Disconnecting a Column from a Splitter

Loosen and remove the internal nut from the splitter fitting. Usually the tubing and ferrule will fall out of the fitting.

Occasionally the ferrule will stick in the fitting. If this happens, use a pointed object like a pen or a paper clip and insert it in the ferrule-release hole in the side of the fitting (Figure 19). Press firmly to rock the top of the ferrule. The ferrule will click when it breaks free and will then be easily removed.

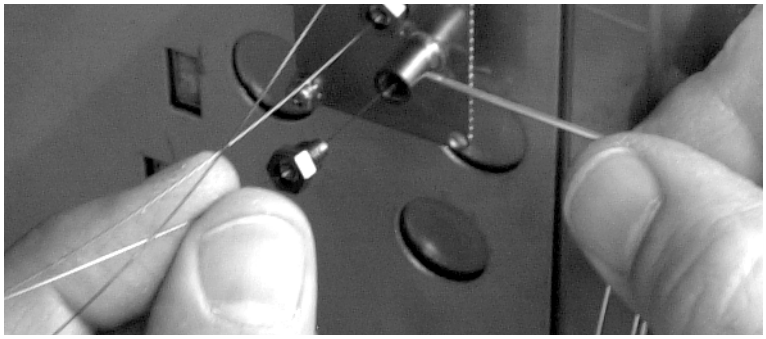

Figure 19 Releasing a ferrule

Protect the column and restrictors

Columns and restrictors with swaged metal ferrules can be disconnected and reconnected several times providing that they have not been overtightened. To protect the tubing end, use one of the sealing caps from the kit. Tighten to finger-tight.

Protect the splitter

Seal the ports of the splitter assembly with plugs when the splitter is not in use. This keeps particulates and contamination out.

Changing Columns without Venting a MS or MSD

For systems that use a MS or MSD attached to the splitter, one added advantage is the GC column can be changed or maintained without venting the MS/MSD. When a column is disconnected from the splitter plate, the makeup gas purges air out of the fitting, preventing air from reaching the MS/MSD providing it is of sufficient flow rate. If disconnecting the column from the inlet with the column installed to the plate, purge gas flows backwards through the column, protecting it from air infiltration.

To perform column maintenance with the purged splitter, the recommended steps are:

1. Cool down the inlet to which the column to be replaced or maintained is connected.
2. Ensure that the pressure to the splitter plate is in the range of 5-10 psig.
3. Disconnect the column from the splitter plate.
4. Replace the column or trim from the inlet end to remove contaminants.
5. If replacing the column, reattach one end to the inlet and turn on the carrier gas to purge air from the column.
6. Swage a metal ferrule on the outlet end of the column.
7. Connect the swaged end of the column to the splitter plate.
8. If finished trimming the column, reattach to the inlet and resume flow.
9. Return the splitter pressure to the original setpoint.

Backflushing the Column

One useful feature available with EPC control of the makeup is the ability to backflush unwanted higher boiling analytes from the column. Use of this feature requires that the split/splitless inlet or multimode inlet be used. Backflushing can reduce overall run time to clean out the column. With purged splitters, backflushing happens just after the last compound of interest has eluted. Temperature is held at that temperature (usually lower than the original ending temperature) and the run ends. Backflushing takes place as part of a post-run program.

As long as you backflush after each run, backflushing is complete after only a few holdup times (in the reverse flow direction). Therefore, it makes most sense to reasonably maximize the reversed flow to minimize the backflush time. Typical holdup times range from 1 to 2 minutes as original pressure drops. This can be significantly reduced by raising splitter pressure. Confirmation of a complete backflush can be empirically visualized by using standards that contain some later eluting markers. No later-eluting compounds will be present in the analysis of a blank that is run just after the test backflush run in a properly tuned backflush.

The process of setting up a post-run backflush program is greatly simplified by using the Backflush wizard provided in any Agilent data system for 7890A GCs. To access the Backflush wizard, select **Edit GC Acquisition Parameters**, then the **Backflush icon**. The wizard allows you to control all the appropriate parameters and limits (such as oven temperature, flows, and void volumes) and to automatically add them to your method.

When developing the backflush portion of your method, consider the following:

- Since the backflush runs as a post-run program, set the backflush initiation time a tenth of a minute or so after the last peak of interest returns to baseline, or after reaching the last temperature of interest.
- Ensure that the split vent flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.
- If using gas saver, ensure that the gas saver flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.

NOTE If using a 6890 Series GC see "Backflush the 6890 Series GC" on page 51.

Using the Effluent Splitter Calculator

The Effluent Splitter Calculator can be used to determine the optimal diameter and length of restrictor tubing for each detector or MS/MSD so that the flow requirements for all devices are met.

To use the Effluent Splitter Calculator, you must first determine the desired column flow using either your data system's method editor, the GC front keypad, the Pressure Flow Calculator, or the Method Translator. (See "Software Installation" on page 14 for more information.)

Then, enter the following parameter values into the **Inputs** section of the Effluent Splitter Calculator:

- **Initial Column flow** - The column flow prior to the splitter installation.
- **Initial Oven Temp** - The starting oven temperature.
- **Carrier Gas** - The carrier gas (Helium, Hydrogen, Nitrogen, or Argon) used to carry your sample throughout the GC.
- **Column outlet pressure** - The column outlet pressure used in your configuration prior to the splitter installation.
- **Detector 1/2/3 operating pressure** - The standard operating pressure for your detectors. Operating pressure for most detectors is 14.696 psia, with exceptions being the MS/MSD (0 psia) and the AED (16.196 psia). See your GC user documentation for details.
- **Detector 1 desired flow** - Enter the desired flow for Detector 1.
- **Flow ratio of Det 2 to Det 1** - Enter the desired flow ratio of Detector 2 to Detector 1.
- **Flow ratio of Det 3 to Det 1** - Enter the desired flow ratio of Detector 3 to Detector 1.

Once you have entered the input values, use the output results table to choose the diameter and length of tubing for each detector. In general, choose the smallest diameter that gives a long enough reach to the detector. For most detectors, the length should be at least 0.3 m, and for MSDs, the length should be at least 0.8 m (for a splitter on the right side of the oven). Also, be sure to choose a tubing size that allows the flow to be greater than the calculated minimum Makeup Flow listed at the bottom of the calculator.

6890 Series GC Supplemental Guide

This appendix describes the procedures for installation and operation of the splitter hardware that are specific to a 6890 Series Gas Chromatograph (GC).

Prepare the 6890 GC

WARNING **Turn the power off and disconnect the power cord before proceeding.**

1 Raise the GC top cover to expose the oven lid.

2 Remove the valve box cutout using a side cutter (Figure 20).

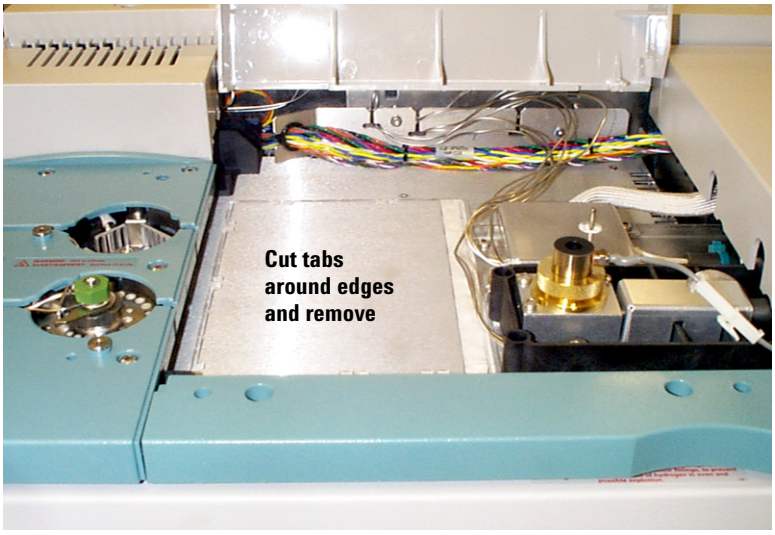

Figure 20 Remove the valve box cutout (6890N GC shown)

3 This exposes a layer of soft insulation. Remove it to expose the hard oven insulation. Remove the precut insulation piece at the location shown in Figure 21.

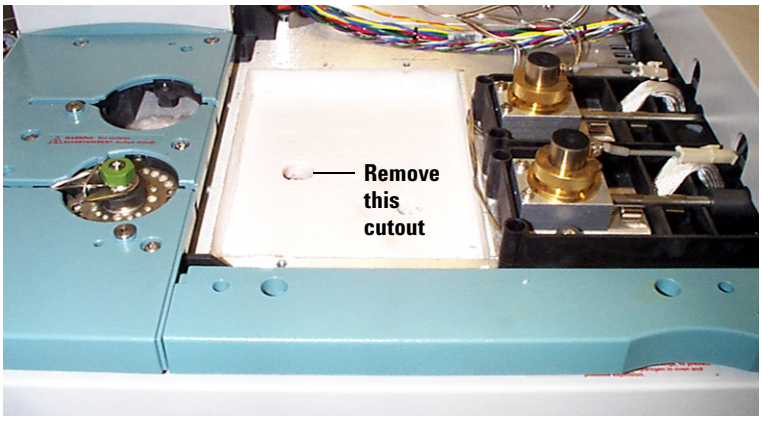

Figure 21 Remove the insulation cutout (6890N GC shown)

4 Replace the soft insulation. Install the valve box blanking plate, using one screw at the front and one at the rear to secure it. See Figure 22.

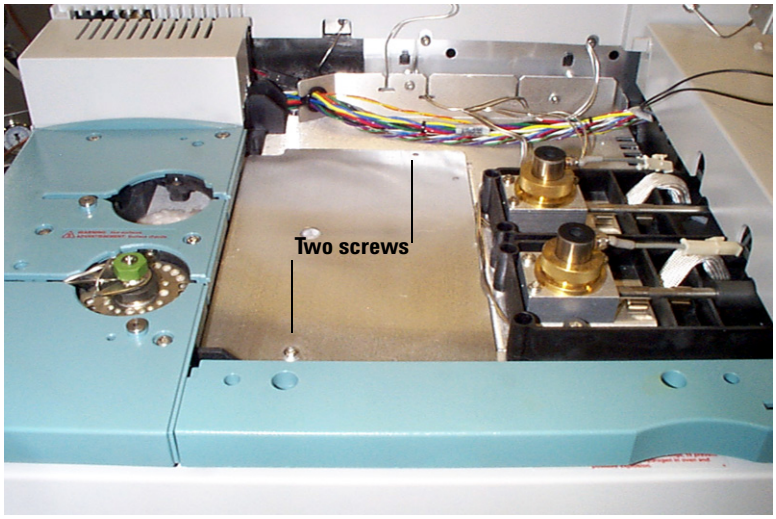

Figure 22 Install valve box blanking plate (6890N GC shown)

Backflush the 6890 Series GC

To backflush after elution of the last peak of interest, the MSD is time-programmed to stop collecting data, the splitter makeup pressure is time-programmed to rise rapidly, and the inlet pressure is reduced rapidly. These pressure changes reverse the flow through the column. Heavy materials are then carried out the split vent of the inlet.

The inlet pressure is programmed to decrease to 0.5 psig. The makeup pressure is programmed to rise to a maximum pressure determined by the detectors and cleanout temperature used.

Using the example above, the MSD will limit the flow, and thus pressure, that can be used for backflushing. The flow allowed to go to the MSD should be no more than 25 mL/min with a standard turbo and 100 mL/min with the performance turbo. Diffusion pumps cannot be used with backflushing. The backflushing conditions must be calculated to not exceed this.

We need to use the MSD restrictor tubing dimensions and the backflushing temperature to find the backflushing pressure. For this example, we will use 20 mL/min going to the MSD.

CAUTION Make sure that MSD acquisition is OFF while backflushing to prevent possible damage to the ion source.

The restrictor to the MSD was 1.236 m of 0.18-mm id tubing. The backflushing temperature used here is the hold temperature at the end of the run in the original method (280 °C). The flow calculator (Figure 23) shows that the makeup pressure can be programmed to 47.5 psig at 280 °C.

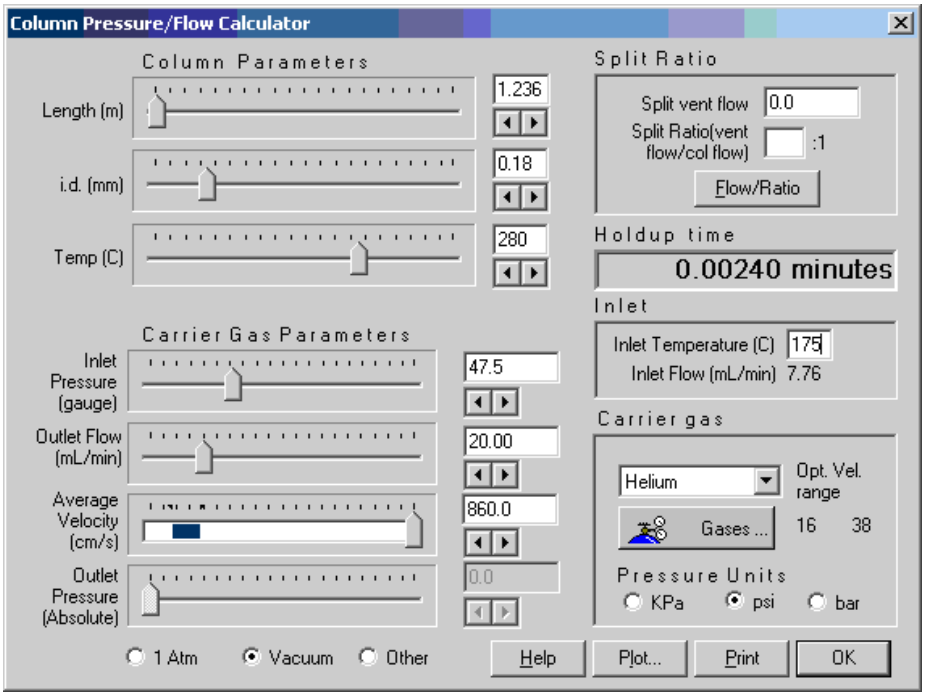

Figure 23 Column backflush flow calculation

When developing the backflush portion of your method, consider the following:

- Ensure that the split vent flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.
- If using gas saver, ensure that the gas saver flow setpoint is at least 25 mL/min and at least 50% more than the column backflush flow rate.

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