Agilent G3183B Three-Way Splitter Kit

Installation and Operation Guide

Agilent Technologies

Notices

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Safety Notices

CAUTION

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WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

In this Guide. . .

This Installation and Operation Guide contains information for installing and using an effluent splitter on an Agilent 6890 gas chromatograph (GC). The G3183B splitter is intended for use with capillary columns and uses makeup gas to maintain adequate flows throughout the system.

1 Introduction

This chapter describes how the splitter works, the GC and software requirements of the system and the contents of the installation kit.

2 Hardware Installation

See this chapter for a detailed procedure for installing the splitter hardware and connecting the makeup gas supply.

3 Splitter Configurations

The split ratio (how the column effluent divides among the three detectors) is governed by three restrictors, which are lengths of deactivated fused silica tubing. This chapter presents a set of precalculated “typical” configurations. If desired, you can create a custom configuration to meet specific needs. The chapter describes a set of software tools, included in the kit, to assist you in designing such configurations. Finally, installation of the column and restrictors is covered.

4 Operation

This chapter contains a worked-out custom configuration, plus a few special topics.

Contents

1 Introduction

This manual covers the installation and operation of the G3183B effluent splitter with makeup kit on the Agilent 6890 series gas chromatograph (GC).

Overview

Splitter installation is done in three steps:

1. Hardware installation. This gets the hardware installed and the gas flows connected.
2. Restrictor configuration. You can choose to use a typical, precalculated configuration or create a custom one using software tools supplied on a CD.
3. Restrictor and column installation. Using the results of step 2, cut the appropriate lengths of the appropriate diameter tubing for the restrictors. Install the restrictors and the analytical column.

How It Works

The splitter divides the effluent from a column among three different detectors. The detectors can be operating at different pressures, that is, any mix of the following can be used:

- **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**
  MSD (mass selective detector)
- **Above atmospheric pressure**
  AED (atomic emission detector)

The split ratio is determined by the length and diameter of tubing connecting the splitter to the detectors. Tubing dimensions may be determined from Table 2 on page 26 in this manual or from a spreadsheet calculator that is included for calculating tubing dimensions for special situations.

Figure 1 shows the plumbing configuration for the G3183B splitter.

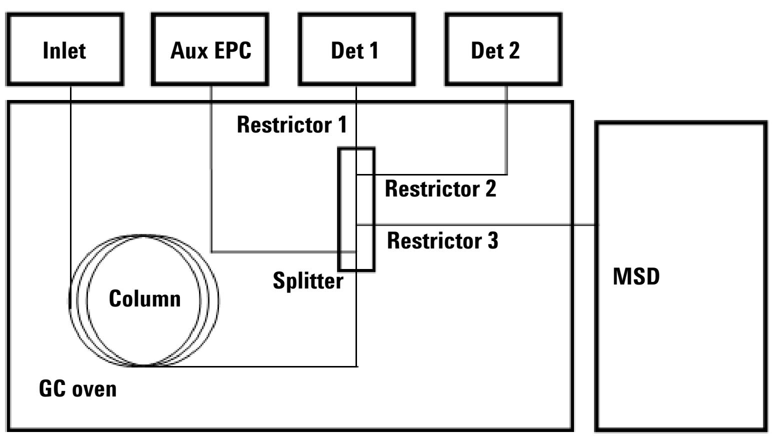


Figure 1 Splitter plumbing

The column flow mixes with the makeup flow in the splitter. This mixture then flows through lengths of uncoated, deactivated, fused-silica tubing to each detector. These tubes act as flow restrictors. While the flows through the restrictors change with oven temperature, the *ratio* of the flows at any temperature is constant.

Details

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

Metal ferrules

The splitter uses 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) that can result in chromatographic problems.

Microfluidic plate

The splitting hardware is based on microfluidic plate technology. This allows very low dead volume connections between the column end and the three detector restrictor tubes. The thin metal plate has fast thermal response and is mounted solidly on the oven wall for ease of use. The interior plate surfaces are deactivated to prevent adsorption of active compounds.

Constant pressure operation

The splitter uses a source of makeup gas supplied by electronic pneumatics control (EPC). This maintains the splitter at a known and constant pressure. Constant pressure allows easier splitting to vacuum detectors like the MSD. It simplifies choice of splitter parameters, allowing all aspects of the chromatographic setup to be calculated. Constant pressure makeup allows the column to be run in constant flow mode while still maintaining a constant split ratio between three detectors of different operating pressures such as the FPD and the MSD. Because the EPC pressure can be time programmed, useful operations like backflushing unwanted heavy materials from the column and changing columns in MSD systems without venting are possible.

Other uses

Other useful configurations can be set up as described in Chapter 4.

Calculation of chromatographic parameters

Because the pressure at the split point is known and constant, the chromatographic parameters can be calculated before setup. This is especially useful with GC/MSD setups, where there are limitations on the flow rates of carrier gas allowed into the MSD. If a method that was originally developed on an MSD is converted to a splitter setup, a new inlet pressure can be calculated to produce retention times very similar to the original method.

Gas 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, with one very important exception. **Do not use hydrogen or any other flammable gas as the makeup gas, regardless of the carrier gas choice**.

The splitter protects the entrance to the MSD when the column is disconnected by blanketing the connection with makeup gas. This gas flows into the column oven. Hydrogen or other flammable gas would present a serious fire and explosion hazard if allowed to flow into the oven.

WARNING **Hydrogen is both a fire and explosion hazard when mixed with air. For this reason, hydrogen must not be used as the makeup gas.**

GC Requirements

The splitter mounts in an Agilent 6890 series GC.

The splitter requires an electronically controlled pressure source such as the Three Channel Pressure controller (6890 option 301 or 308) or a Pneumatics Control Module (PCM).

Other Requirements

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

Parts Supplied

Parts in the G3183B kit are listed in Table 1.

Table 1 Parts supplied

Part number	Description	Quantity
G1530-01340	Capillary column spring clips	4
G2855-60140	Oven bracket assembly	1
G2855-60560	T-screw oven bracket retainer	2
0515-0374	Screw, M3 × 10 mm	7
G3183-60500	Three-way splitter with makeup gas assembly	1
G2855-80022	Manual and calculator CD	1
G3183-90120	Manual, G3183B	
0100-0241	Union, 1/8-inch to 1/16-inch reducing	1
0100-2354	Tubing, stainless steel, 1/16-inch od × 0.01-inch id, 1 m	1
G1580-00130	Valve box blanking plate	1
0100-0124	Union, stainless steel, 1/16-inch tubing	1
G2855-60150	Supplies and spares kit	1

Part Identification

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

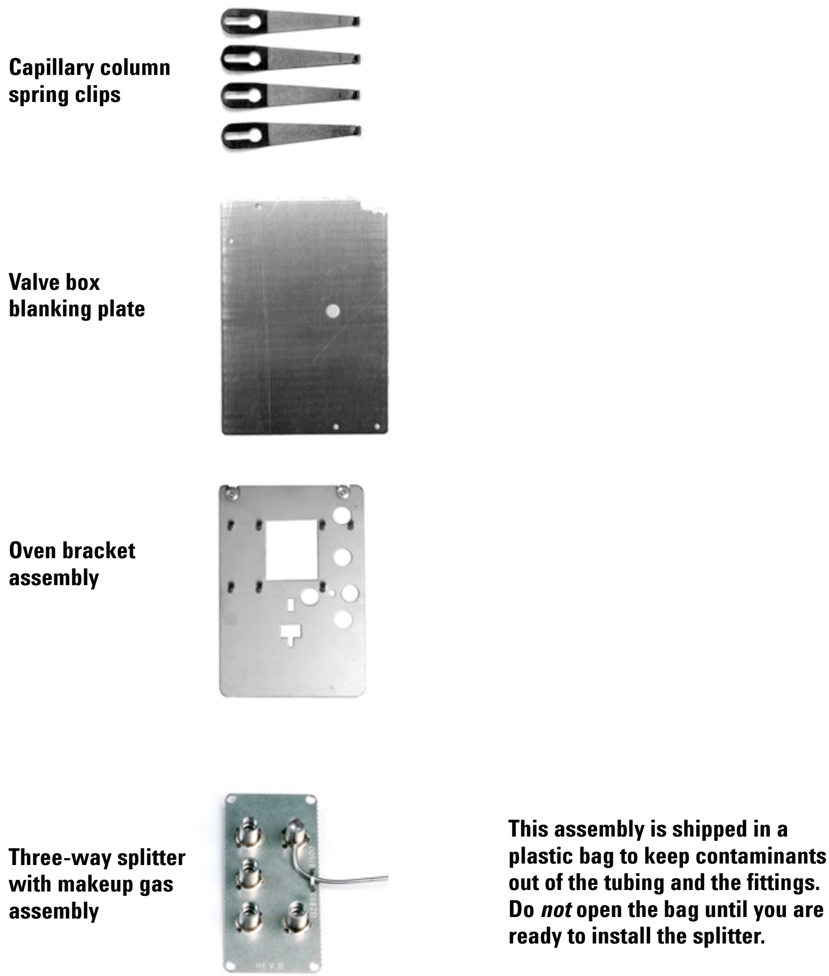


Figure 2 Part identification

Parts Not Supplied

Brown-dot frit (19231-60610)

Tools Required

Side cutter, large

Screwdrivers, Phillips

Pressure Units

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

Gauge pressure (psig)

This is the pressure as measured by most pressure gauge. 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 procedure for installing the splitter hardware and connecting the makeup gas supply.

Prepare the 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 3).

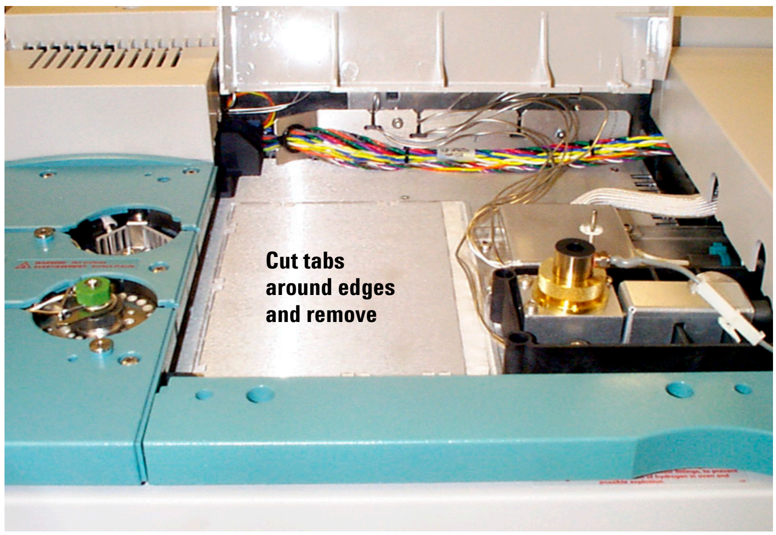


Figure 3 Remove the valve box cutout

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 4.

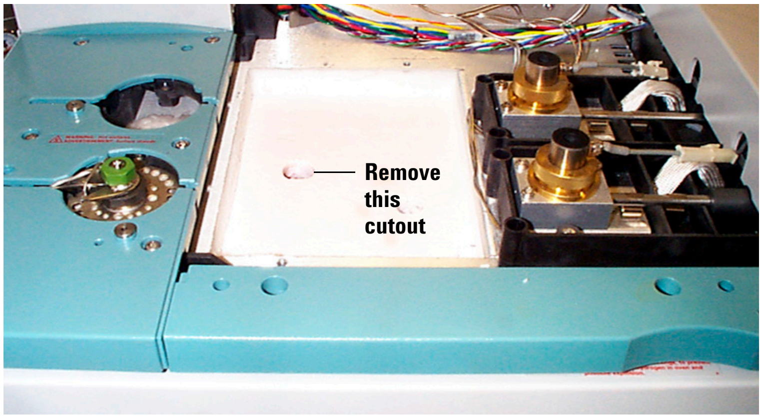


Figure 4 Remove the insulation cutout

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 5.

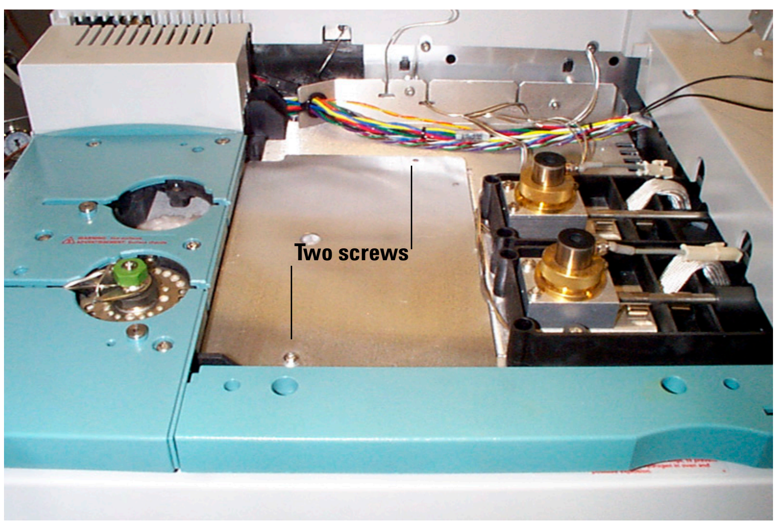


Figure 5 Install valve box blanking plate

Install the Column Clips

Install the four column clips on the oven shroud (Figure 6).

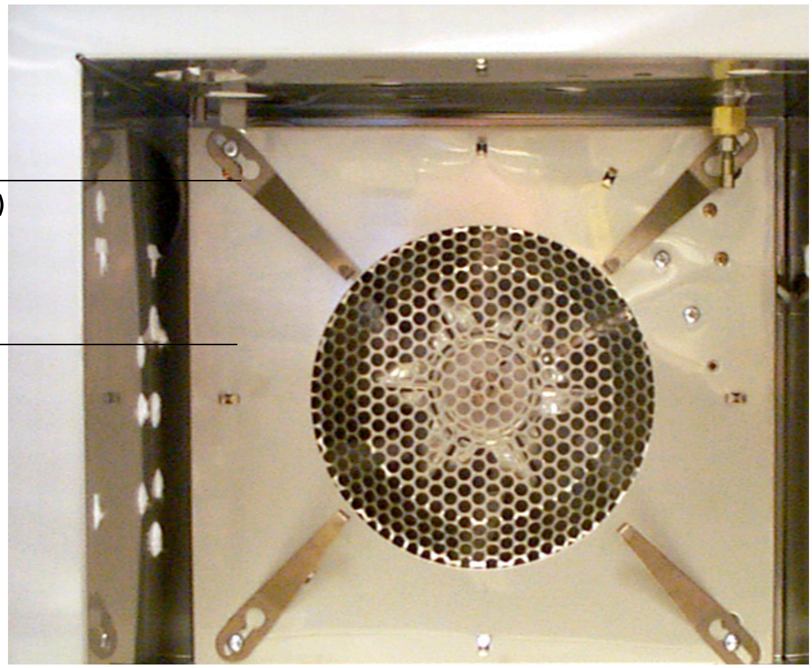


Figure 6 Oven clips

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 splitter is usually installed on the right side of the oven.

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 two T-shaped thumbscrews to fasten the bracket to the T-slots in the oven wall (Figure 7).

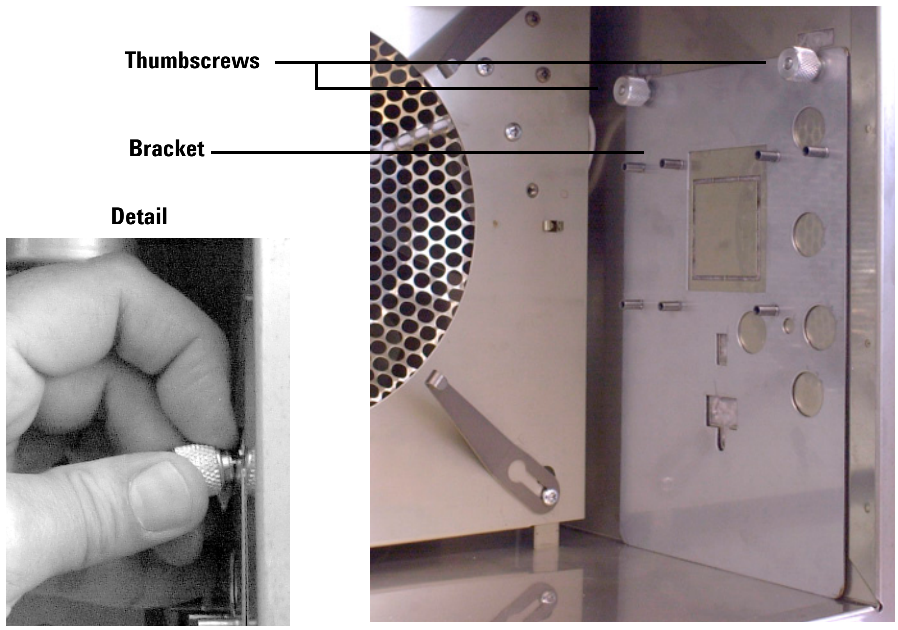


Figure 7 Installing the bracket

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.

CAUTION In the following steps, bend the tubing over an object such as your thumb to avoid kinks.

3 Open the plastic bag and remove the splitter assembly. Install a plastic cap on the end of the makeup gas tubing. Place small pieces of tape over the open end of the fittings.

4 Prebend the tubing according to Figure 8. This will make splitter installation much easier.

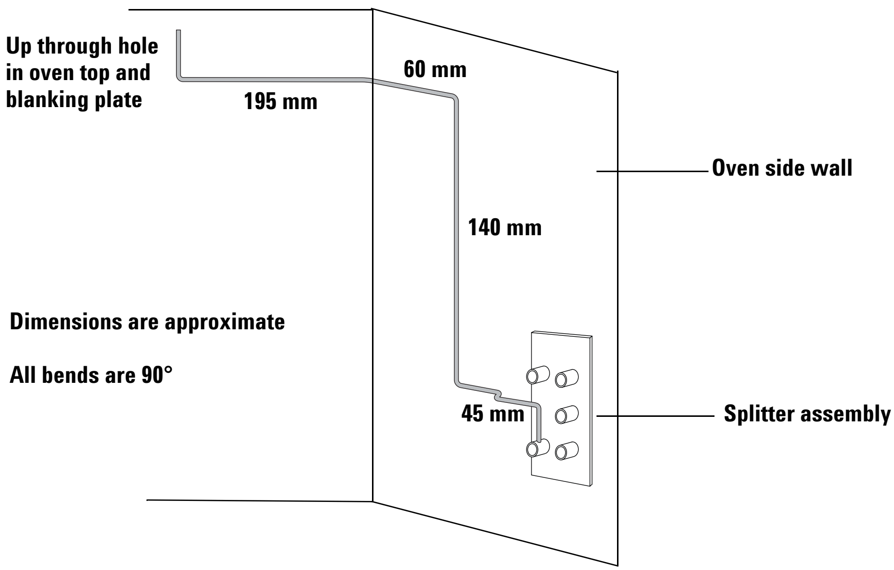


Figure 8 Prebending the splitter tubing

5 Push the end of the makeup gas tubing up through the top oven wall so that the end of the tubing comes out in the hole of the valve box blanking plate.

6 Route the prebent tubing against the oven wall and top to keep it clean for future maintenance. It should be behind the back detector location.

7 Screw the splitter assembly to the bracket (three screws). See Figure 9.

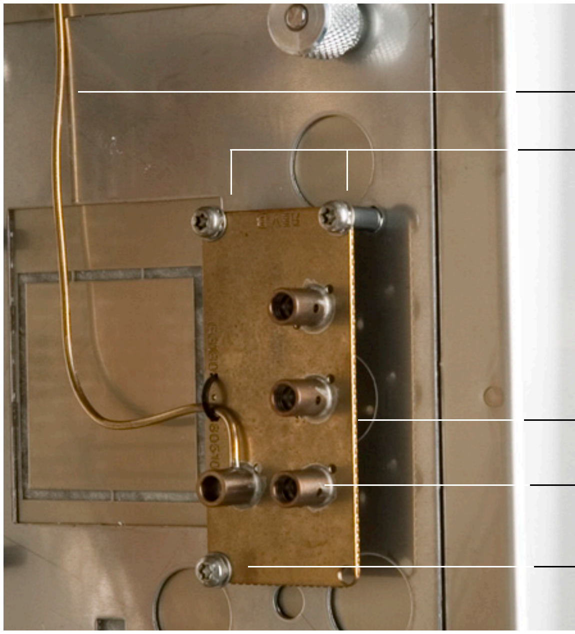


Figure 9 Installing the splitter assembly

8 After the splitter is installed and the makeup gas tubing is routed, trim the tubing above the oven top, leaving at least 10 cm.

Connect the Makeup Gas Supply

Connect the makeup gas source to the Pneumatic Control Module or Auxiliary Pressure controller.

To supply the makeup gas from a Pneumatic Control Module (PCM)

1 Connect the tubing from the PCM to the length of stainless steel tubing from the kit with a union.
2 Connect the free end of the stainless steel tubing to the tubing from the splitter assembly with a union. See Figure 10.

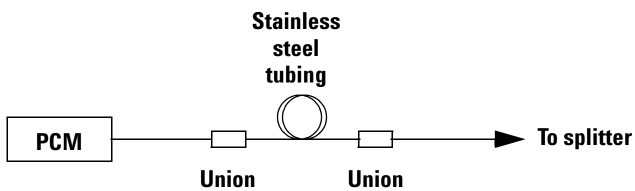


Figure 10 Plumbing a PCM makeup supply

To supply the makeup gas from an Auxiliary Pressure controller

1 Install the brown-dot frit (part no. 19231-60610) in the output channel. See your GC manual for details.
2 Connect the tubing from the Auxiliary Pressure controller to the tubing from the splitter assembly with the 1/8 to 1/16-inch stainless steel reducing union.

This completes the hardware installation.

3
Splitter Configurations

The combination of restrictor diameters and lengths determines how the column effluent is divided (the split ratio) among the three detectors. There are two approaches to setting up a splitter method.

- Use a typical configuration. A set of eight configurations is discussed beginning on the next page. They apply to a variety of detector combinations and split ratios. All of the flows have been calculated.
- Create a custom configuration. If the typical configurations do not meet your needs, you can create one that does. The CD shipped with the splitter kit provides tools for the necessary calculations.

We suggest examining the typical configurations first, since they cover a wide variety of splitter applications and require no calculations.

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 2 lists typical splitting configurations. Table 3 shows the resulting gas flows. All calculations assume helium as the carrier gas and a splitter pressure setting of 3.8 psig.

Table 2 Splitting configurations

Configuration	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 3 Splitter flows

	40 °C			200 °C			300 °C		
Configur-ation	**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 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 2 and proceed to “Restrictor and Column Installation" on page 35.

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 1 can be used but peak broadening and/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 1 are very limited due to these flow considerations and should be avoided if possible.

Custom Configurations

The CD supplied with this kit contains three software tools:

Effluent Splitter Calculator (with Makeup) Calculates dimensions (length and inside diameter) of restrictors to obtain a desired split ratio (Figure 11).

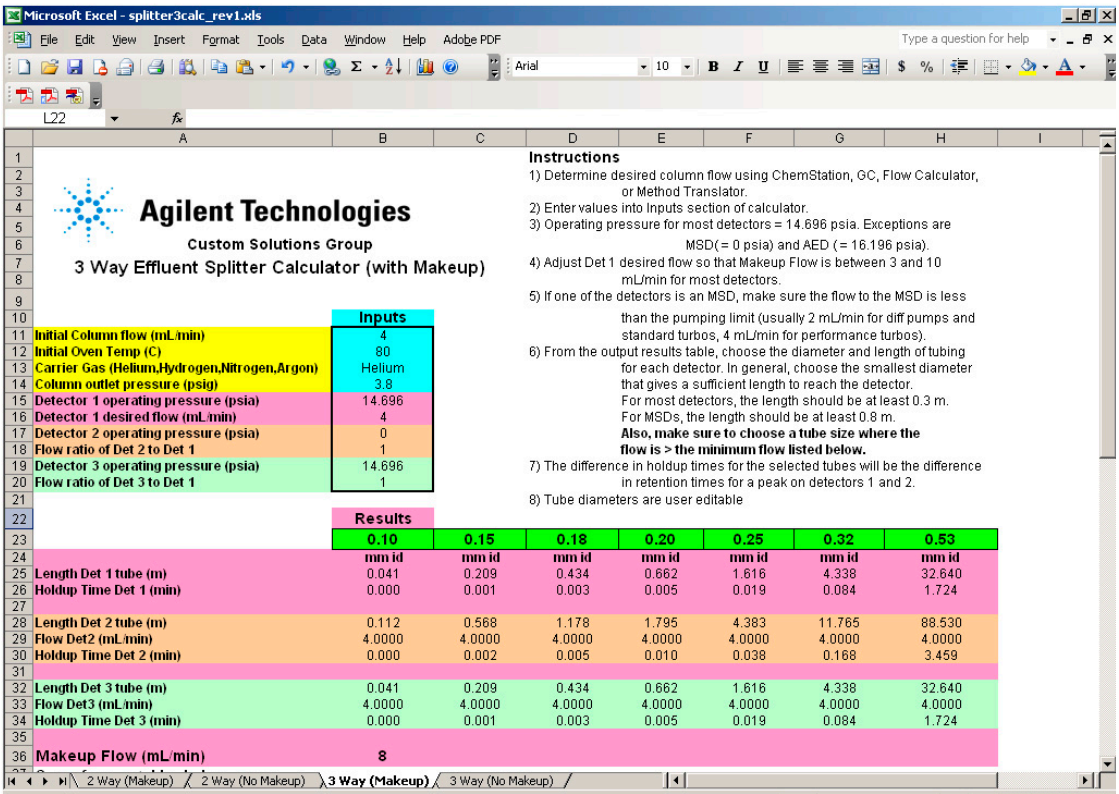


Figure 11 Effluent Splitter Calculator

GC Method Translation Converts an analytical method from one set of operating conditions to another (Figure 12).

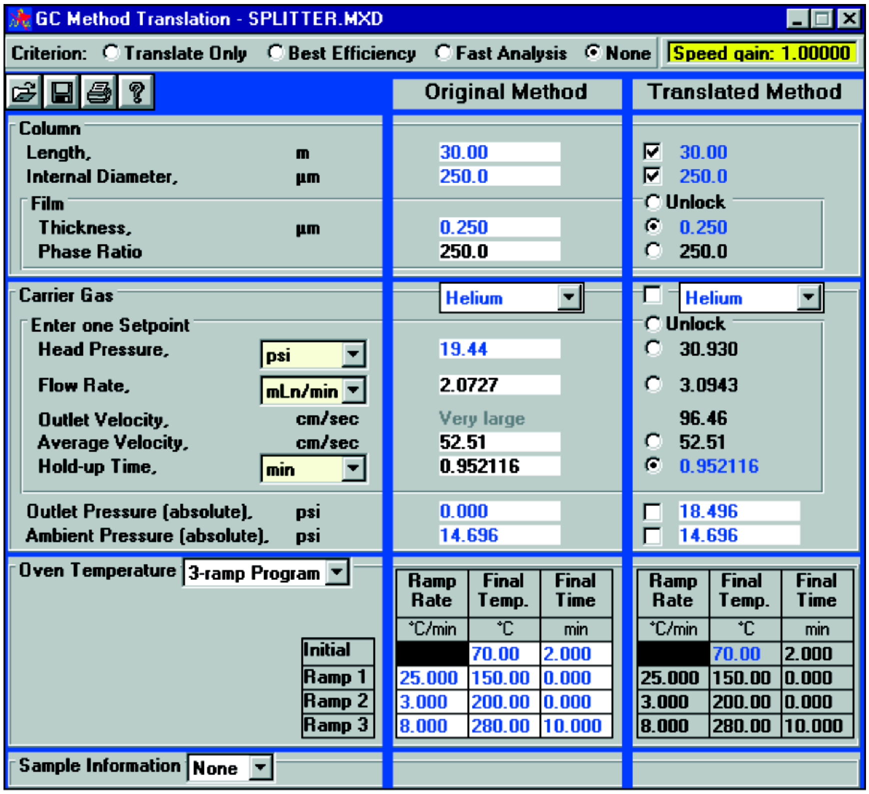


Figure 12 GC Method Translation

Column Pressure/Flow Calculator Calculates flows and pressures for a given set of column (or restrictor) dimensions (Figure 13).

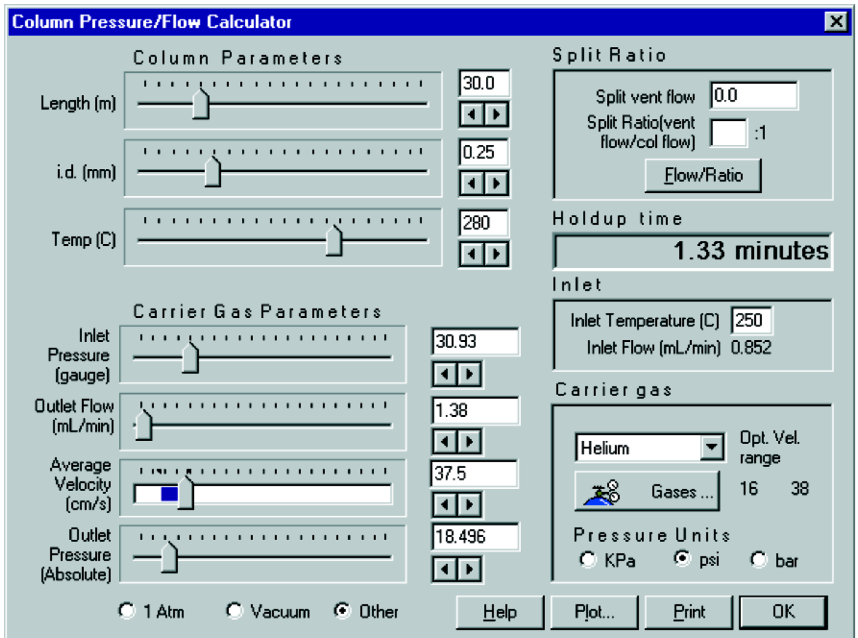


Figure 13 Column Flow/Pressure Calculator

These tools allow you to perform all the calculations needed to create a custom splitter configuration. We recommend that you load the CD software into your PC.

1 Insert the CD into the drive and click the **Start** icon in the bottom left of the screen. Select **Run** and type **X:\ Setup,** where **X** is the letter assigned to the CD drive.

2 Click **Start**, then select **Programs** and the program you wish to run.

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 1, 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 4. Restrictors that have less flow will still work, but peak broadening and/or tailing may result.

Table 4 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 6890 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. The ChemStation screen where the column outlet pressure is set is shown in Figure 14.

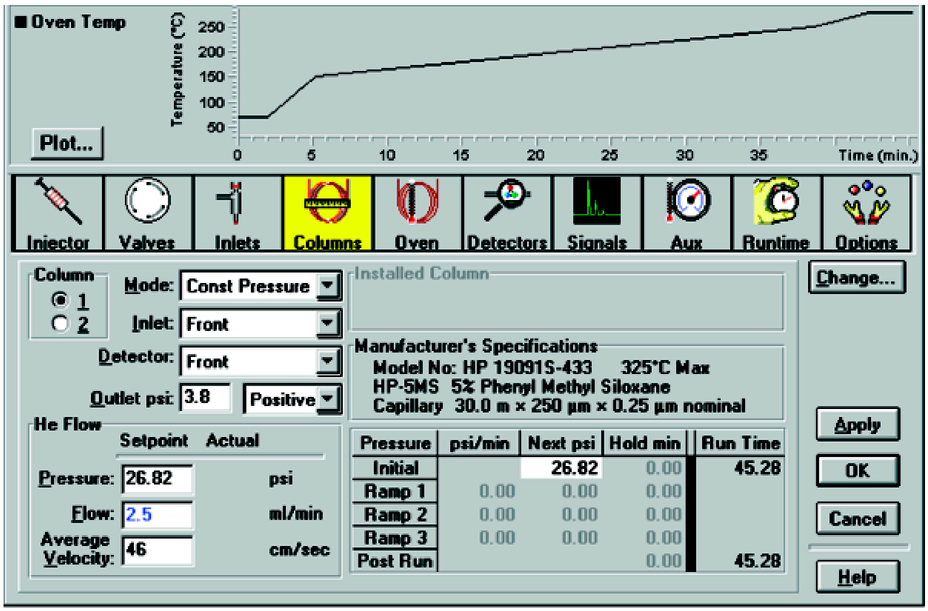


Figure 14 Column outlet pressure screen

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.

Restrictor and Column Installation

Restrictors and the column exit are connected to the splitter assembly using internal nuts and SilTite ferrules. Install fittings on the column exit and one end of each restrictor as described in "Swaging SilTite Ferrules" on the CD.

Install the column

1 Hang the analytical column on the column clips. The clips hold the outside of the wire basket that supports the column. Adjust the clips if necessary.

2 Connect the column to the inlet fitting.

Connect the splitter

1 Preswage the SilTite ferrules to the restrictors and columns. See "Swaging SilTite Ferrules" on the CD for details.

2 Connect the restrictors to the connectors on the splitter (Figure 15). Finger-tighten until just snug, then tighten with a wrench an additional 15°. Install the back restrictor first.

3 Connect the restrictors to the detector inlet fittings. See your GC documentation for details.

4 Connect the column exit to the splitter. 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.

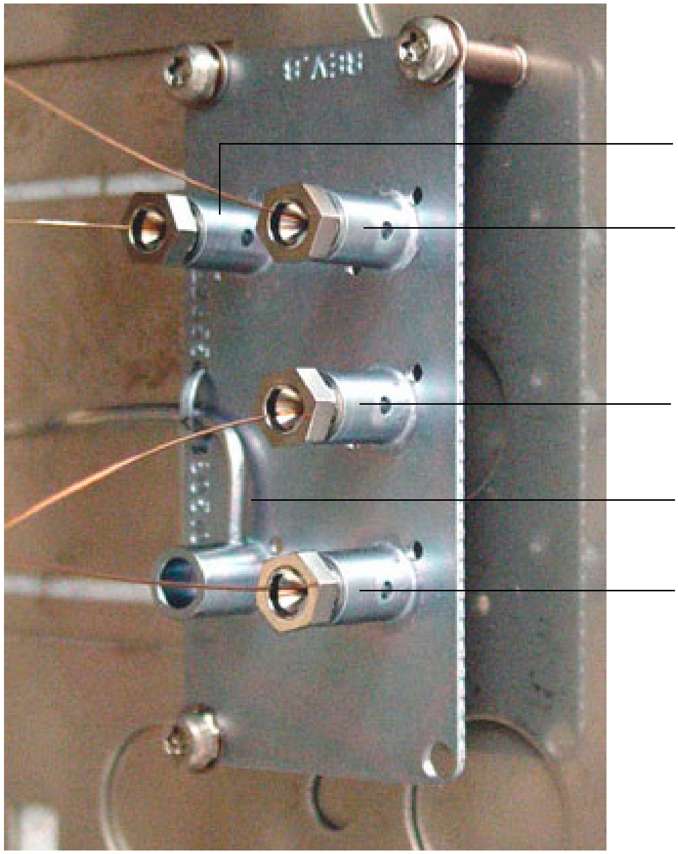


Figure 15 Detector and column connections

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

Disconnect tubing from the 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 16). Press firmly. The ferrule will click when it breaks free.

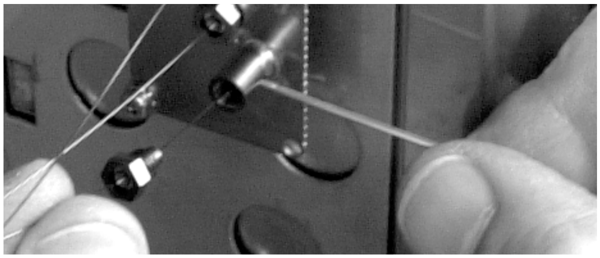


Figure 16 Releasing a ferrule

Protect the column and restrictors

Column and restrictor tubes with swaged metal ferrules can be disconnected and reconnected several times. To protect the tubing end, use one of the brass-sealing caps from the kit. Tighten to finger-tight plus 15 degrees.

Protect the splitter

Seal the ports of the splitter assembly with plugs when the splitter is not connected. This keeps particulates and contamination out. To make a plug, cut about 5 cm (2 inches) of the stainless steel wire and swage it as you would a column. Use the metal ferrule that fits 0.25-mm id columns. After swaging, clip the wire to within 0.5 mm of the ferrule end with a small high-quality wire cutter.

If the splitter is not connected but the GC is to be used, either remove the splitter plate from the oven or plug three of the holes and leave a restrictor on the fourth. Leave the makeup pressure at 3.8 psig to provide a flow through the splitter. Leaving an unplugged splitter in the oven when heating may damage the deactivation layer inside.

4
Operation

This chapter contains a worked-through custom configuration, plus some special topics.

An Example

Assume we have a method that uses an HP-5MS 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 17) is useful for this. Use the **None** mode and check the button to make the hold-up times the same.

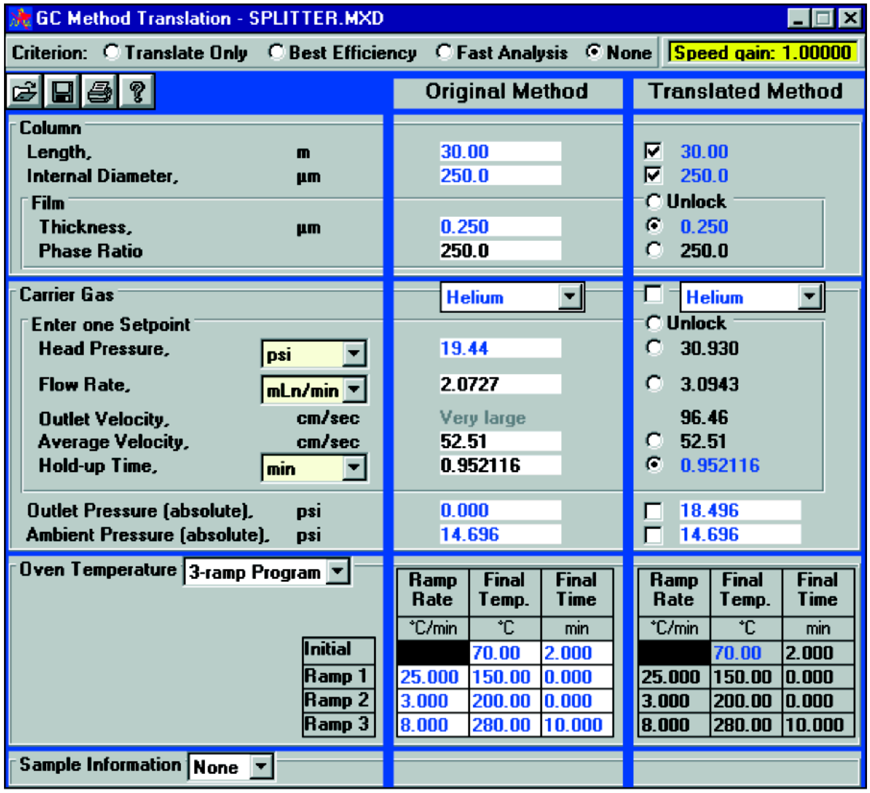


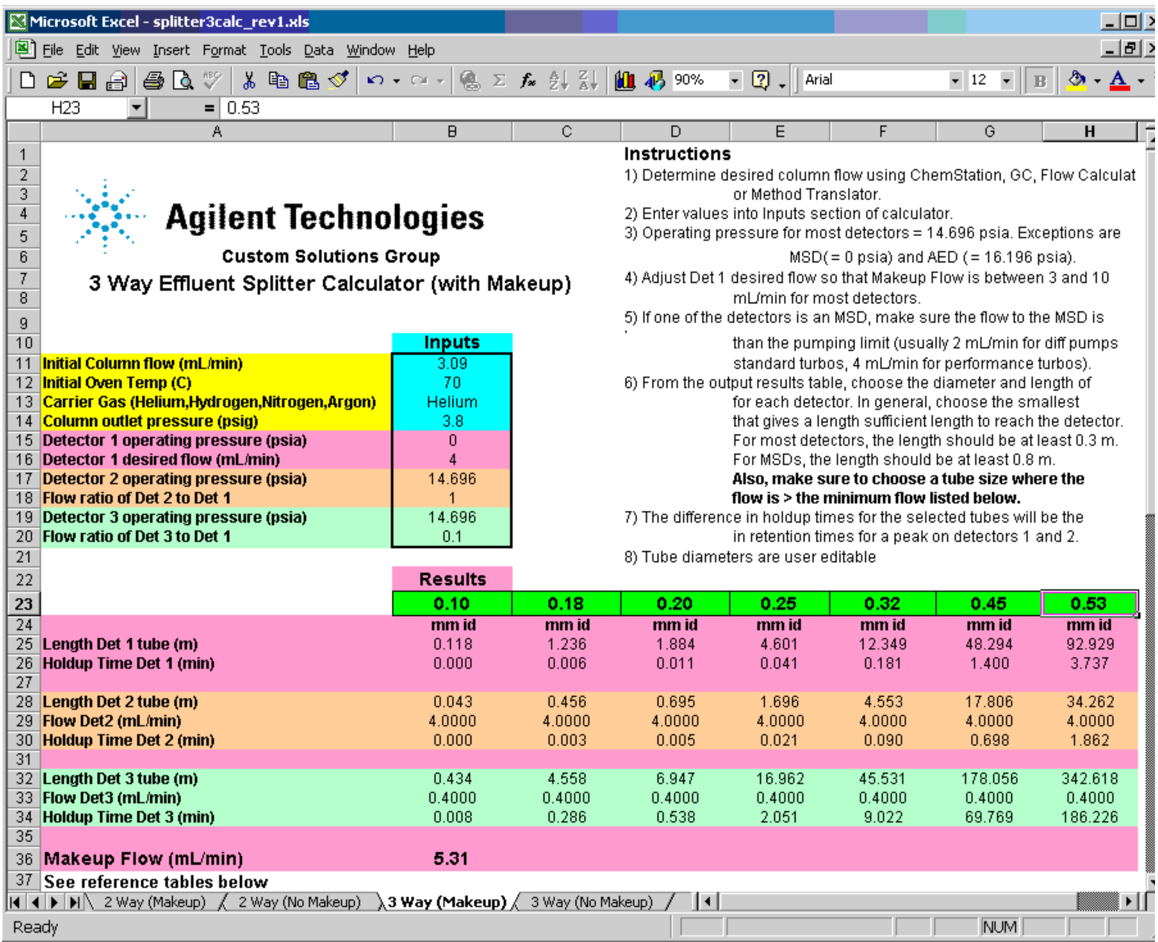
Figure 17 Calculating column flow

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. Hence, 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

Start up the spreadsheet "splitter3_calc_rev1.xls" in Excel. Select the “3-Way (Makeup)” worksheet. 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 18) with the MSD assumed to be Detector 1, the FPD as Detector 2, and the ECD as Detector 3.



	Inputs
Initial Column flow (mL/min)	3.09
Initial Oven Temp (C)	70
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Column outlet pressure (psig)	3.8
Detector 1 operating pressure (psia)	0
Detector 1 desired flow (mL/min)	4
Detector 2 operating pressure (psia)	14.696
Flow ratio of Det 2 to Det 1	1
Detector 3 operating pressure (psia)	14.696
Flow ratio of Det 3 to Det 1	0.1

Results	0.10 mm id	0.18 mm id	0.20 mm id	0.25 mm id	0.32 mm id	0.45 mm id	0.53 mm id
Length Det 1 tube (m)	0.118	1.236	1.884	4.601	12.349	48.294	92.929
Holdup Time Det 1 (min)	0.000	0.006	0.011	0.041	0.181	1.400	3.737
Length Det 2 tube (m)	0.043	0.456	0.695	1.696	4.553	17.806	34.262
Flow Det2 (mL/min)	4.0000	4.0000	4.0000	4.0000	4.0000	4.0000	4.0000
Holdup Time Det 2 (min)	0.000	0.003	0.005	0.021	0.090	0.698	1.862
Length Det 3 tube (m)	0.434	4.558	6.947	16.962	45.531	178.056	342.618
Flow Det3 (mL/min)	0.4000	0.4000	0.4000	0.4000	0.4000	0.4000	0.4000
Holdup Time Det 3 (min)	0.008	0.286	0.538	2.051	9.022	69.769	186.226
Makeup Flow (mL/min)	5.31						

Figure 18 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 4 on page 33 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 19) requires that the output pressure be entered in psia. Therefore 18.496 psia (3.8 psig) is entered.

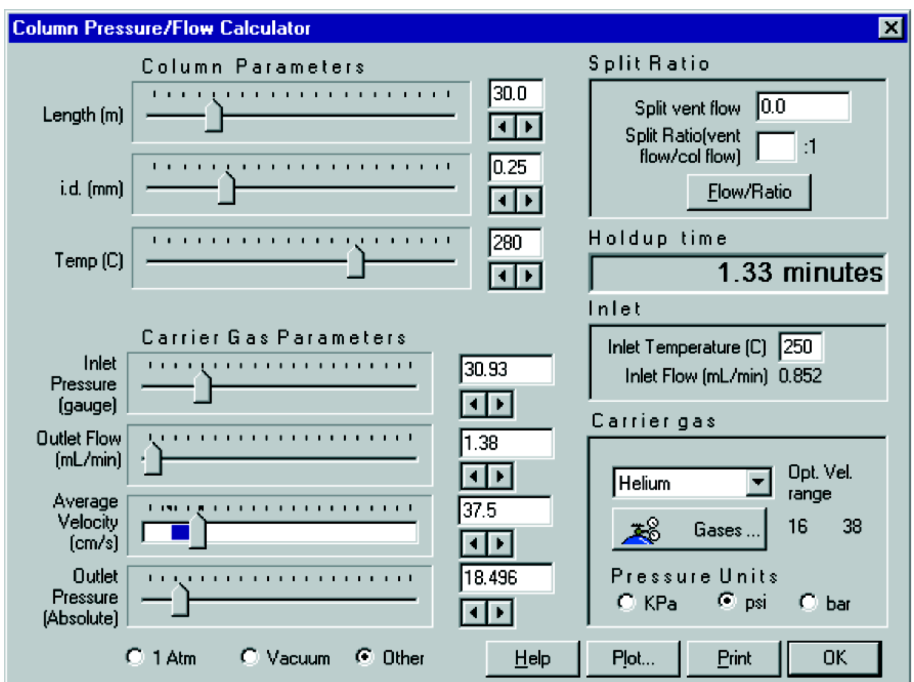


Figure 19 Column flow calculation

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 20).

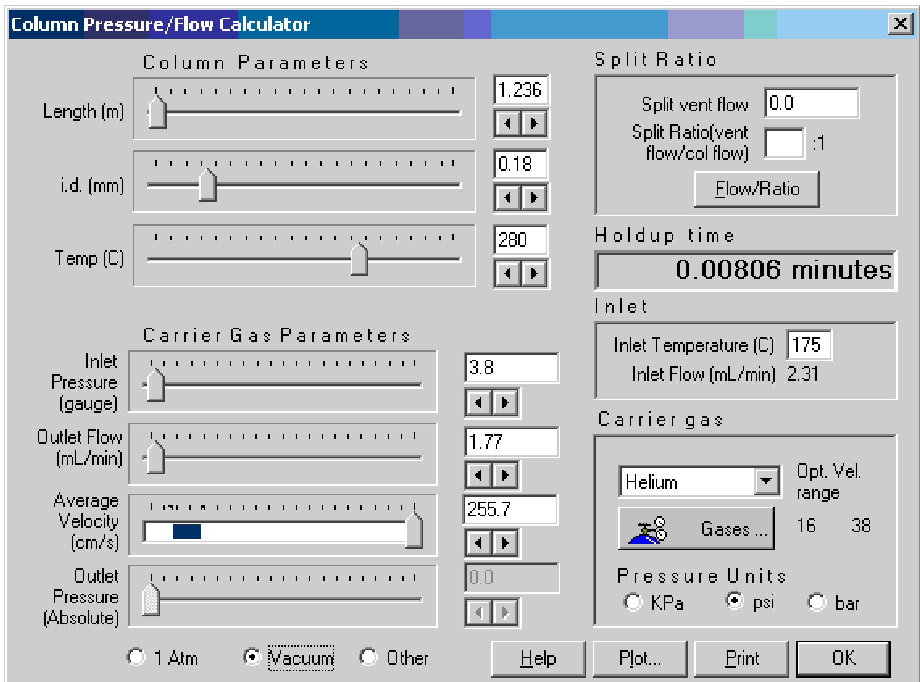


Figure 20 MSD restrictor flow calculation

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 21) is also 1.77 mL/min at 280 °C.

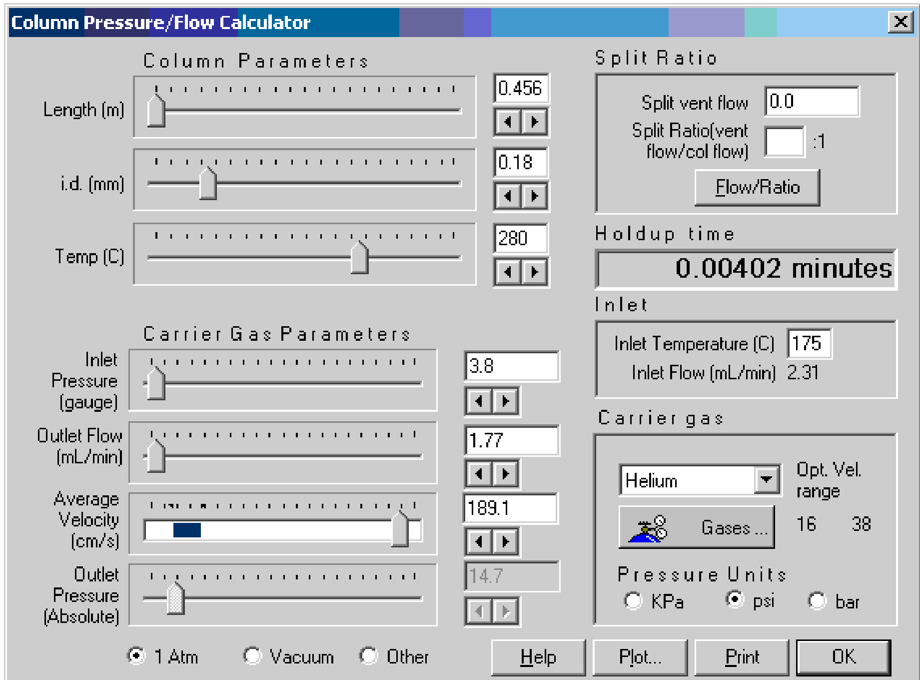


Figure 21 FPD restrictor flow calculation

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 22) is 0.18 mL/min at 280 °C.

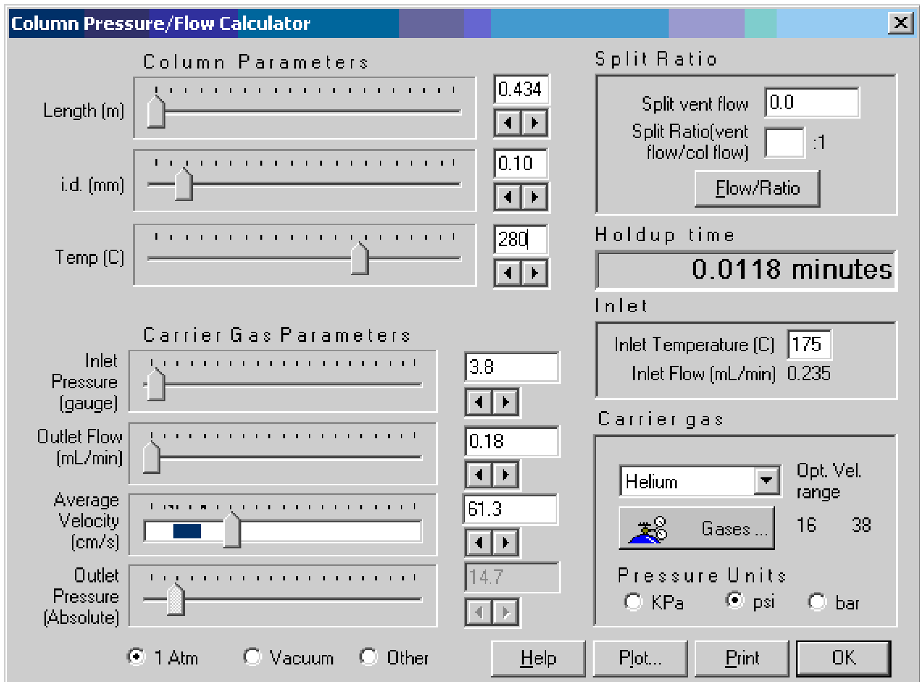


Figure 22 ECD restrictor flow calculation

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.

Changing Columns Without Venting the MSD

For systems that use an MSD attached to the splitter, one added advantage is the GC column can be changed without venting the MSD. When the column is disconnected from the splitter plate, the makeup gas forces air out of the fitting, preventing air from reaching the MSD.

To change columns with the splitter, the recommended steps are:

1. Cool the inlet to which the column to be removed is connected.
2. Disconnect the column from the splitter plate.
3. Immediately install a plug in the plate where the column was connected.
4. Change the column in the inlet and turn on carrier gas to displace air from the column.
5. Preswage a metal ferrule on the outlet end of the column.
6. Remove the plug from the connector.
7. Connect the new column to the splitter.

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 be used. Backflushing reduces the hold at the end of the run to clean out the column.

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.

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

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.

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.

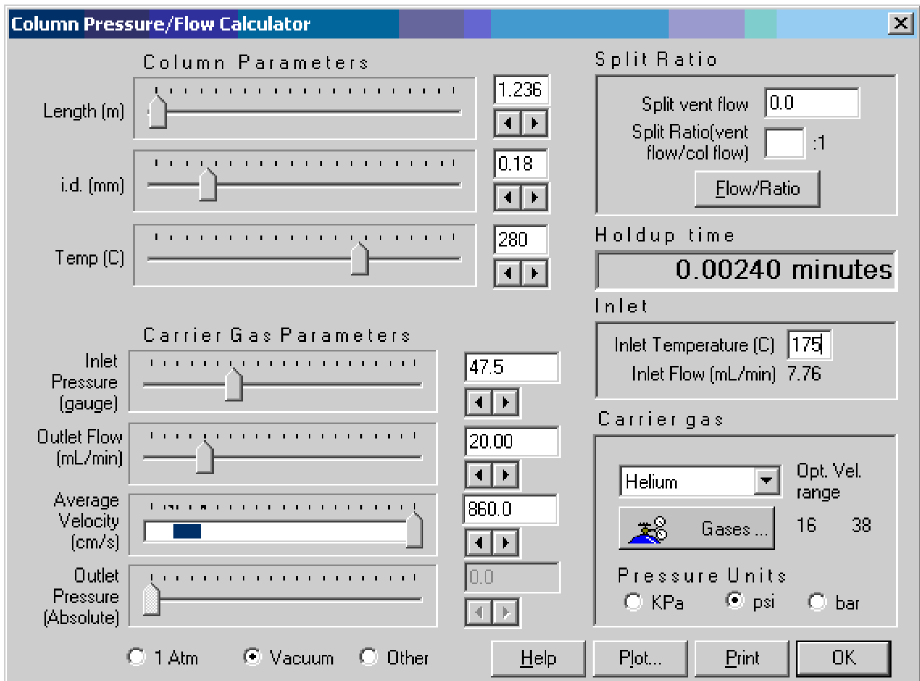


Figure 23 Column backflush flow calculation

The time required for complete backflushing of heavy materials is then determined empirically. Blank runs after samples with different backflush hold times are used to determine the minimum time to remove all heavy material.

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 24 shows the typical three-way splitter configuration for reference.

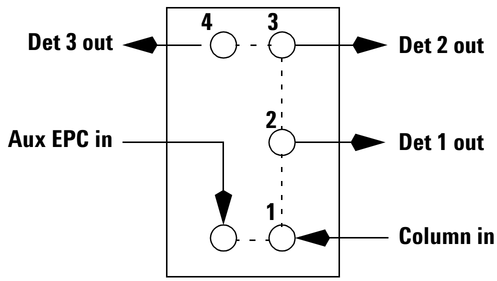


Figure 24 Three-way effluent splitter

Two-way splitter

Figure 25 shows the configuration for a two-way splitter.

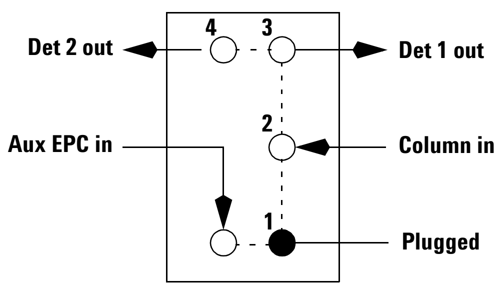


Figure 25 Two-way effluent splitter

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 Micro-Fluidic Effluent Splitter with Makeup Gas provided on the CD that came with this product for setting up a two-way split.

Two columns in/Two detectors out

Figure 26 shows the configuration for a two-column combiner/two-way splitter.

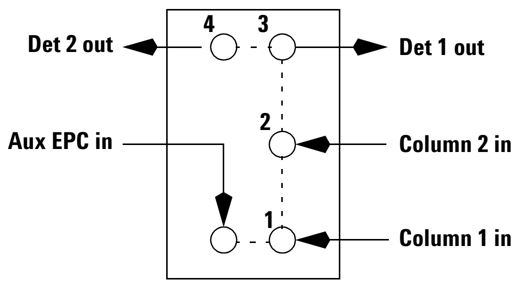


Figure 26 Two columns in/two detectors out

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

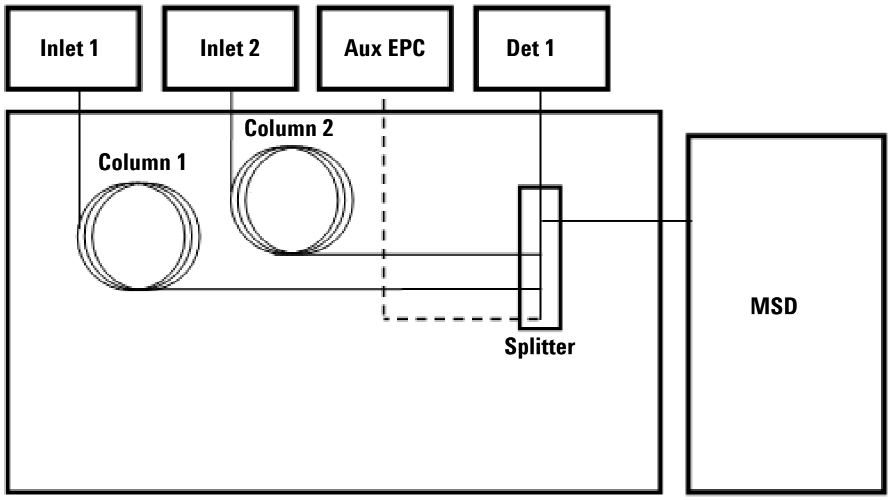


Figure 27 Two different columns in/two detectors out

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.

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