Agilent G3181B Two-Way Splitter Kit

Without Makeup Gas

Installation and Operation Guide

Agilent Technologies

Notices

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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 Agilent G3181B Splitter is intended for use with capillary columns.

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.

3 Splitter Configurations

The split ratio (how the column effluent divides between the two detectors) is governed by two restrictors, which are lengths of deactivated fused silica tubing. This chapter presents simple configurations for atmospheric-pressure detectors. If desired, you can create other configurations to meet specific needs. This chapter also describes the use of software tools included in the kit to assist you in designing such configurations.

4 Restrictor and Column Installation

This chapter describes installation of the column and presents instructions for connecting restrictor tubing to, and disconnecting it from, the splitter.

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Introduction

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This chapter contains:

- An overview of how the splitter is installed and set up
- A description of how the splitter works
- A description of how limitations of previous splitters have been overcome
- Requirements for use of the G3181B Splitter
- A list of parts supplied in the splitter kit
- Identification of the parts in the kit

Overview

To install the splitter you will complete the following three processes:

1 **Install the hardware.** Install the bracket and splitter plate on the right-hand wall of the GC oven.

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.

3 **Connect the column, splitter, and detectors.** Using the results of step 2, cut the required lengths of the chosen diameter tubing for the restrictors. Using the instructions in the appendix, install the metal ferrules onto the ends of the column and restrictor tubes (see "Swaging SilTite Ferrules" on the CD for details) and connect them to the splitter plate. Connect the other ends of the restrictor tubes to the detectors in the usual way.

How It Works

The splitter divides the effluent from a column between two different detectors. Agilent offers two types of splitter hardware. The G3181B (covered in this manual) is a simple splitter without makeup gas. Another splitter (G3180B) is of similar construction, but includes the addition of makeup gas regulated by electronic pneneumatic control (EPC). The G3180B is also described on the CD that contains this manual.

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 G3181B Splitter without makeup gas is useful for labs that set up a single configuration and use it without changing columns or parameters frequently. It is best used with column flows greater than 2 mL/min and with atmospheric pressure detectors. While the G3181B can be used for splitting to an MSD if care is taken in the setup (see “Splitting to an MSD” on page 26), the G3180B is strongly recommended for MSD applications.

The *split ratio* of the G3181B is determined by the lengths and diameters of restrictor tubing that connect it to the detectors. In addition to the hardware, Agilent provides calculation software to determine restrictor tubing dimensions and to predict the chromatographic performance of a specific splitter setup, minimizing method development time.

Figure 1 shows the plumbing configuration for the G3181B Splitter.

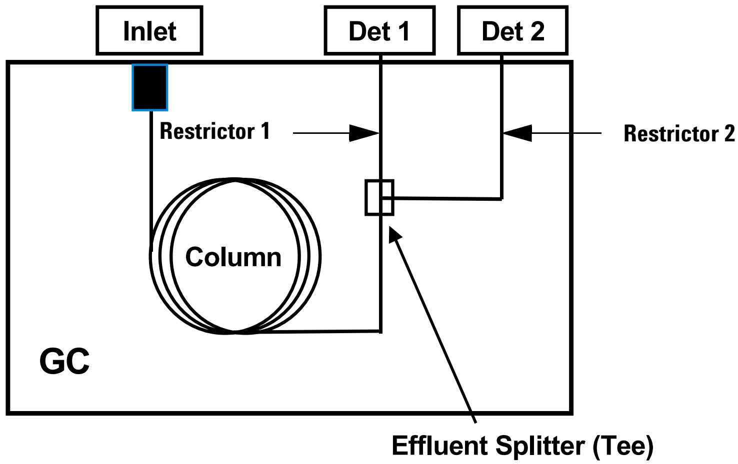

Figure 1 Splitter plumbing

The splitter splits the effluent flow from the column. Each part of the split flow travels through a length of UCDFS tubing to one of the detectors. These tubes act as *flow restrictors* since they restrict the flow to the detectors, controlling the relative portion that is received by each of them. While the flow through each restrictor changes with oven temperature, the *ratio* of the two flows is the same at any temperature.

Details

Splitting the column effluent between two detectors has long been used to improve GC analyses, since using two detectors gives a much clearer picture of the sample makeup than using a single detector. For example, using only an FPD for detection of phosphorus pesticides works well for most samples. However, for samples that have extremely large non-phosphorus matrix peaks, these peaks could appear as small responses on the FPD signal and erroneously be interpreted as phosphorus compounds. By splitting the effluent to an FID, the FID would show those matrix peaks that are large enough to exceed the selectivity of the FPD, thus eliminating the confusion.

While the use of splitters is an old concept, the technique has been limited in its use due to technical challenges with the splitting hardware. For optimal performance, a splitter must be inert, have low dead volume, be leak free, have no outgassing from sealing materials, and be capable of withstanding the temperatures used in the GC analysis.

For instance, glass “Y” connectors meet most of the criteria, except that they sometimes become unattached from one of the tubes upon thermal cycling of the oven. Other splitters have used polyimide ferrules, which can outgas at high temperatures and develop leaks upon thermal cycling due to shrinkage of the polymer. Graphite ferrule splitters eliminate the thermal cycling problems, but may have inertness problems—they can outgas contaminants at high temperatures and can shed particles into the sample path.

The G3181B Splitter overcomes all these limitations of previous approaches to splitting column effluent between two detectors.

Metal ferrules

All connections in the G3181B are made using metal ferrules. There is no outgassing, particle shedding or leakage from the metal ferrules, even with oven temperatures of up to 350 °C.

Microfluidic plate technology

The splitting hardware is based on microfluidic plate technology. The splitters are constructed by etching micro channels into small stainless-steel plates. The interior plate surfaces are deactivated to prevent adsorption by active compounds. The thin metal plate has fast thermal response and is mounted solidly on the oven wall for ease of use. The entire approach allows very low dead volume connections between the column end and the two detector restrictor tubes, resulting in splitter hardware that meets both chromatographic and reliability requirements.

Calculation of chromatographic parameters

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.

Requirements

GC

The splitter mounts in an Agilent 6890 series GC.

Other

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

Parts Supplied

The G3181B kit contains the following parts (Table 1).

Table 1 Parts supplied

Part number	Description	Quantity
G1530-01340	Capillary column spring clips	4
G2855-80022	Manual and calculator CD	1
G3181-90120	Manual, G3181B	1
G3181-60500	Compact splitter, inert (kit)	1
G2855-60140	Oven bracket assembly	1
G2855-60560	T-screw oven bracket retainer	2
0515-0374	Screw, M3 × 10 mm	7
G2855-60150	Supplies and spares kit	1

Parts Not Supplied

SilTite ferrules

Tools Required

Screwdriver, Phillips

Open-end wrenches

Parts Identification

Figure 2 identifies the parts unique to the G3181B Splitter Kit.

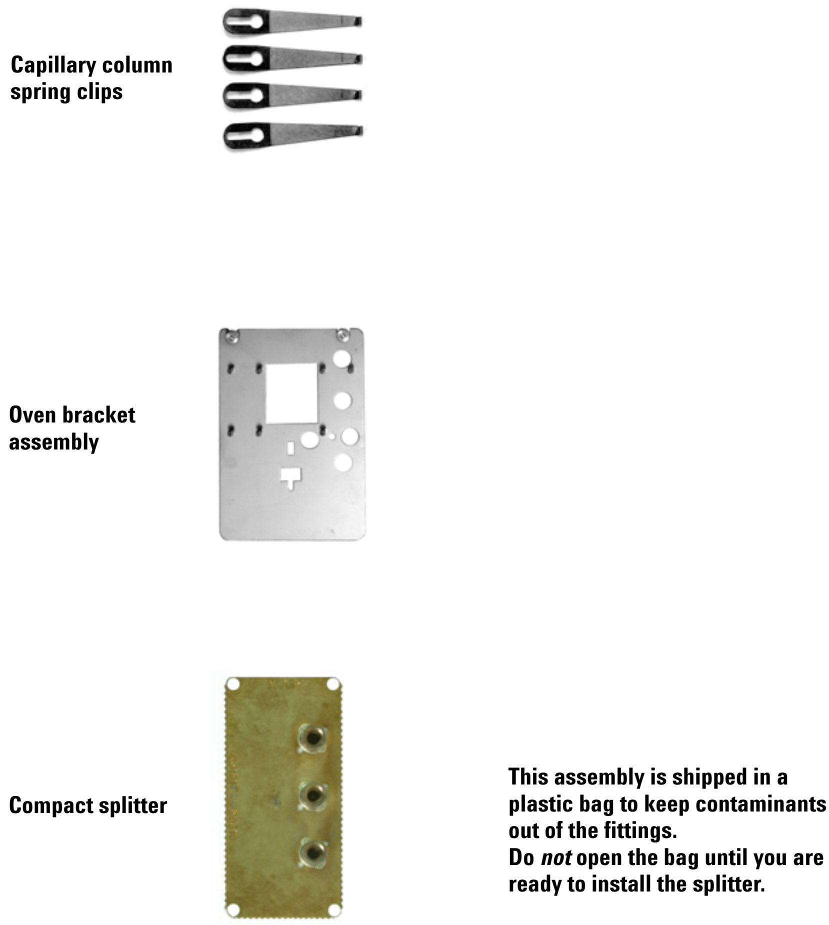

Figure 2 Parts identification

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Hardware Installation

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This chapter describes the procedure for installing the splitter hardware.

Install the Column Clips

1 Set the GC oven and detector temperatures to 35 °C and allow them to cool.

2 Turn off the GC and disconnect the power cord before proceeding.

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

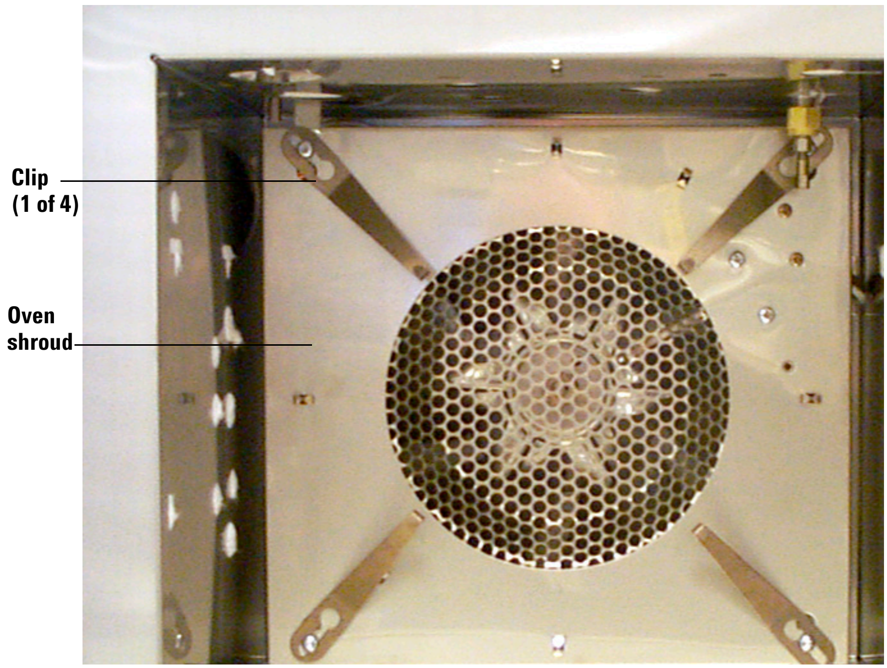

Figure 3 Oven clips

Install the Bracket and Splitter

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

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

4 Place the bracket against the side of the oven. The two notches should be in the top edge and the standoffs should point toward the middle of the oven.

5 Use two T-shaped thumbscrews to fasten the two bracket notches to the T-slots in the oven wall (Figure 4).

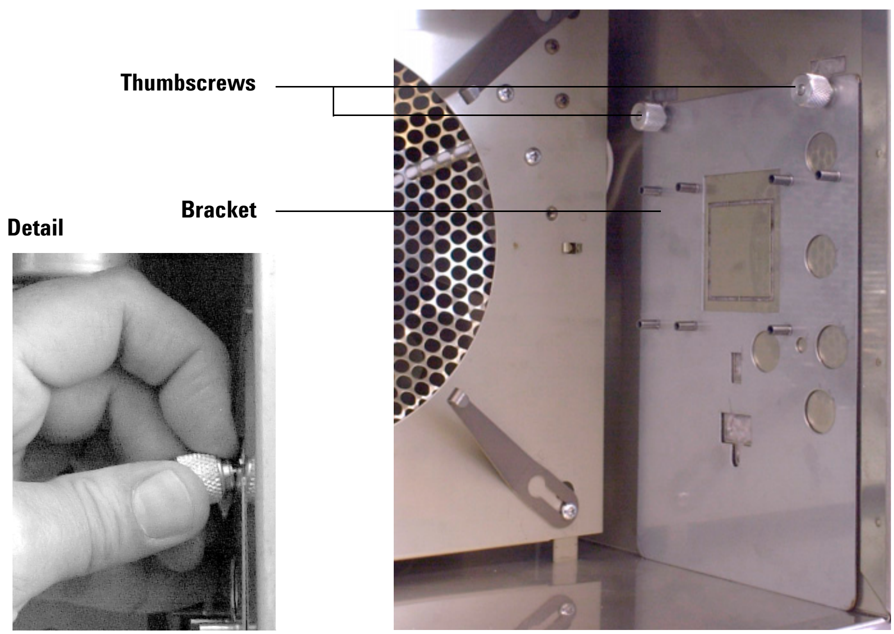

Figure 4 Installing the bracket

6 Screw the splitter assembly to the bracket (three screws). See Figure 5.

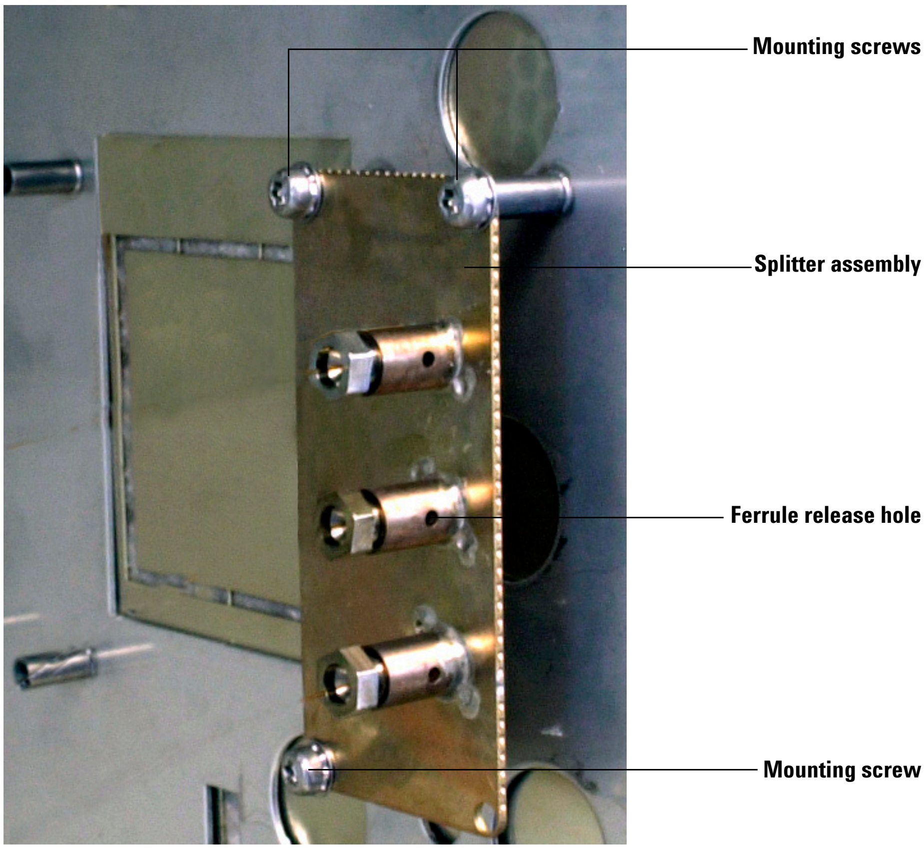

Figure 5 Installing the splitter assembly

This completes hardware installation.

3
Splitter Configurations

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The combination of restrictor diameters and lengths determines how the column effluent is divided (the split ratio) between the two detectors.

This chapter describes different setup approaches. Select the approach that most closely matches your situation.

Simple Setup for Atmospheric Pressure Detectors

A simple setup can be used for most situations where both detectors operate at atmospheric pressure (TCD, FID, FPD, ECD, NPD) and you want a 1:1 split ratio. For splitting to detectors with reduced operating pressure, see “Splitting to an MSD”, page 26.

Base your choice of the lengths and diameters of the restrictor tubes on:

- The column flow used
- The minimum length required to reach from the splitter plate to the detector
- The amount of pressure (if any) desired in the splitter

For most methods that use two atmospheric-pressure detectors, make your choices as follows:

1 **Choose diameter**—Choose restrictor tubing of the same diameter as that of the column. If, for example, the column is 0.25-mm inside diameter, choose 0.25-mm id tubing. The restrictor tubes can either be uncoated deactivated fused silica (UCDFS) tubing, or can be made from pieces cut off from the end of the column. Use this approach when analyzing very active compounds that require special columns.

2 **Choose length**—Make the lengths of both tubes equal to obtain an equal split between the two detectors. Use at least 30 cm for all atmospheric-pressure detectors, except the FPD. For an FPD, use a length of at least 50 cm to accommodate the length of the transfer line.

3 **Connect the splitter tubes and column**—Attach the column and restrictor tubes as described in Chapter 4.

4 **Turn on the GC and check for leaks.**

5 **Set up the GC, ChemStation, or Cerity**—Be sure to set the GC or ChemStation to collect the signals from both detectors. All other parameters can be the same as in the original unsplit method.

The setup is now complete.

Other Split Ratios for Atmospheric Pressure Detectors

For split ratios other than 1:1, the setup is the same as described on page 22 ***except*** for the choice of restrictor tubing diameter and length. The split ratio between different detectors is determined by the lengths and diameters of the two restrictor tubes. When using tubes of the same diameter to two detectors operating at atmospheric pressure, the split ratio is inversely proportional to the tube lengths. To obtain a 3 to 1 split between detectors A and B, make the tube to B three times longer than that to A.

For larger split ratios, such as 10:1 between an FID and ECD, use narrower tubing to the ECD to get increased restriction at a reasonable tubing length. The flow through a tube is directly proportional to the diameter to the fourth power.

As an aid to choosing restrictor tubes of the appropriate length and diameter, the CD included with the G3181B Splitter Kit contains an Excel spreadsheet calculator. This calculator requires Microsoft Excel 97 or later (not supplied).

Make your choices as follows:

1 **Choose restrictor tubing diameter and length**–Use tubing lengths of at least 30 cm for all atmospheric-pressure detectors except the FPD. For an FPD, use a length of at least 50 cm to accommodate the length of the transfer line. As described in “Example 1, Split 3:1 between NPD and FID”, page 24, it will be necessary to adjust the diameter of this tubing to achieve the desired split ratio.

2 **Connect the splitter tubes and column** as described in Chapter 4.

3 **Turn on the GC and check for leaks.**

4 **Set up the GC, ChemStation, or Cerity** to collect the signals from both detectors. All other parameters can be the same as in the original unsplit method.

The setup is now complete.

Example 1, Split 3:1 between NPD and FID

In this example:

- You are splitting between an FID and an NPD.
- You desire a flow to the NPD of three times that to the FID so that 75% of the sample will go to the NPD.
- You have chosen 0.35 m of 0.18-mm id tubing to connect the splitter to the FID.

You wish to run 0.18-mm id tubing to the NPD. What length will you need for a 3:1 flow ratio?

1 Choose a diameter and length combination.

To calculate this, start the spreadsheet calculator **3181BsplitterCalc.xls**. Go to the worksheet tab named **Equivalent Length and Misc**. and locate the **Restrictor Length Calculator** (Figure 6).

a Enter the desired split ratio in cell **B23**, **3.00**.

b Enter the FID restrictor tubing length and diameter (**0.350** m, **0.180** mm) in cells **B24** and **B25**.

c Enter the **0.180** mm for the NPD restrictor tubing diameter in cell **B26**. **Enter lengths in meters and diameters in millimeters.**

	A	B	C	D
20	**Restrictor Length Calculator**			
21	**(Use only for splits between atmospheric pressure detectors)**			
22				
23	**Desired Flow ratio of Det 2 to Det 1**	**3.00**		
24	**Length Detector 1 tube (m)**	**0.350**		
25	**Diameter Detector 1 tube (mm)**	**0.180**		
26	**Diameter Detector 2 tube (mm)**	**0.180**		
27	**Length Detector 2 tube (m)**	**0.117**		
28				

Figure 6 Restrictor Length Calculator

d Review the results and recalculate as necessary.

Cell **B27** displays the calculated length of 0.18-mm id tubing required to give the desired flow to the NPD, in this example, 0.117 m. This is too short to reach from the splitter to the NPD, however. For a longer length of tubing, increase the tubing diameter. A larger diameter tube requires an increased length to achieve an equivalent pressure drop.

e Enter increasingly larger diameters in cell **B26** until you find a suitable length. As shown in , a 0.25-mm id gives a usable length of 0.434 m.

	A	B	C	D
20	**Restrictor Length Calculator**			
21	**(Use only for splits between atmospheric pressure detectors)**			
22				
23	**Desired Flow ratio of Det 2 to Det 1**	**3.00**		
24	**Length Detector 1 tube (m)**	**0.350**		
25	**Diameter Detector 1 tube (mm)**	**0.180**		
26	**Diameter Detector 2 tube (mm)**	**0.250**		
27	**Length Detector 2 tube (m)**	**0.434**		
28				

Figure 7 Finding a suitable length

In summary, to achieve a 3:1 split between an NPD and an FID, you have determined the following configuration:

- 0.434 m of 0.25-mm id restrictor tubing between the splitter and the NPD
- 0.35 m of 0.18-mm id tubing from the splitter to the FID

2 **Connect the splitter tubes and column** as described in Chapter 4.

3 **Turn on the GC and check for leaks.**

4 **Set up the GC, ChemStation, or Cerity** to collect the signals from both detectors.

Splitting to an MSD

Splitting the output of a GC between an atmospheric-pressure detector and a mass-selective detector (MSD), which operates at reduced pressure, requires careful calculation of restrictor tubing lengths and diameters.

Splitting to an MSD requires:

- Correct flow limits (maximum flow of 2 mL/min or 4 mL/min, depending on vacuum pump type) into the MSD.

NOTE

The pressure should not exceed 4 psig (18.696 psia), because the 6890 GC and ChemStation column outlet pressure values used in column flow and inlet split ratio calculations cannot be set above 4 psig.

- Splitter pressure above atmospheric pressure at all times
- Inlet pressures corrected to match existing GC or GC/MSD methods, so the splitter method retention times remain similar to those in the original method.
- Constant pressure column flow mode. Constant pressure mode allows the ChemStation or GC to maintain constant splitter pressure so the split ratio between the MSD and the other detector remains constant with changing oven temperature.

The requirement to *always* operate the splitter above atmospheric pressure prevents the MSD from sucking in gases from the other detector, which would happen if the splitter pressure dropped below atmospheric pressure (or the operating pressure of the other detector). Sucking gases from the other detector into the MSD not only stops the splitter from working, it can also result in damage to the source in the MSD

The provided spreadsheet **3181Bsplitter.Calc.xls** contains calculators to determine the appropriate splitter tubing sizes, operating pressures, and method changes.

To set up a split between an MSD and another detector:

1 **Determine the flow limit** of the MSD you are using. The limit is 2 mL/min for diffusion pumps and standard turbo systems, and 4 mL/min for performance turbo systems.

2 **Calculate original (unsplit) method properties.**

   a Open the file **3181Bsplitter.Calc.xls**.

   b Select the **Splitter Pressure Calculator** worksheet.

   c Locate the **Original GC Method calculator** and enter the original method settings (cells **B9–B14**).

   d Note the original method's **Column average linear velocity**, **Column flow**, and **Column holdup time**.

3 **Calculate new method settings.**

   a Change the **Absolute column outlet (detector) pressure** to the value desired for the split method.

   b Determine the new **Column flow** and **Inlet pressure** required to make the **Column average linear velocity** the same as the original method. See "Example 2, Split 1:3 between MSD and FID", page 28, for details.

4 **Calculate acceptable restrictor tube dimensions.**

   a Go to the **Splitter Tubing Selector** worksheet and locate the **Effluent Splitter Calculator**.

   b Following the instructions on the worksheet, enter the data required. Use the new column flow determined in step 4b as the initial **Column flow**.

   The worksheet displays options for restrictor ("splitter") tubing id and lengths at the desired split ratio, operating pressure, and MSD flow.

5 Select the most appropriate restrictor tube dimensions from the calculator table.

6 Size the selected splitter hardware using the calculated parameters, then assemble as described in Chapter 4. Check for leaks.

7 Set the MSD ChemStation to collect the signals from both detectors, if desired.

The method is now ready for use.

NOTE

Make sure to use *constant-pressure mode* when splitting to an MSD. It is important to maintain constant pressure in the splitter. Constant-pressure mode allows the ChemStation or GC to maintain constant splitter pressure so the split ratio between the MSD and the other detector remains constant with changing oven temperature.

CAUTION

The calculators in the spreadsheet 3181BsplitterCalc.xls do not account for the temperature of detector heated zones in the calculations. This simplifying assumption can lead to errors in the calculated flows, pressures, or lengths of as much as 20%. The calculators are still useful, but designing a splitter setup right on the edge of a limit is not recommended. Leave some margin for error.

Example 2, Split 1:3 between MSD and FID

A splitter is to be used with an MSD and an FID. The original method was developed on an FID setup. The new setup will operate the splitter at 3 psig (17.696 psia). The MSD is a performance turbo system with a maximum flow of 4 mL/min. The desired split ratio is 1:3 MSD to FID.

The original method parameters were:

Carrier gas	Helium
Initial oven temperature	50 °C
Inlet pressure (constant-pressure mode)	20.00 psig
Column dimensions	30 m × 0.25-mm id
Column outlet pressure (FID)	14.696 psia (0 psig)

Determine the flow limit of your MSD

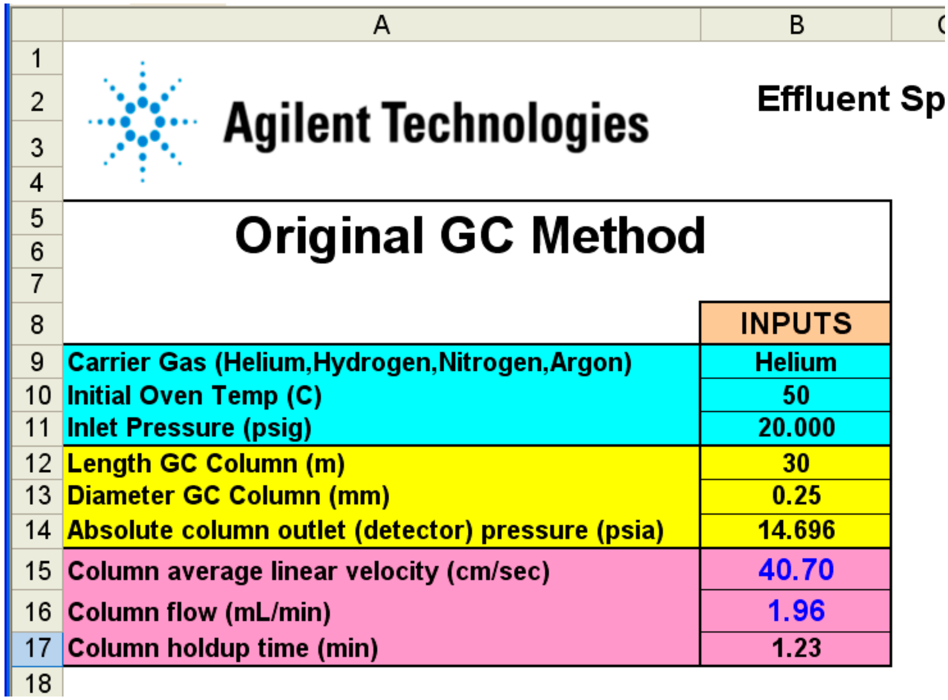
As stated above, the limit is 4 mL/min.

Calculate original (unsplit) method properties

1 Open **3181Bsplitter.Calc.xls**.

2 Select the S**plitter Pressure Calculator** worksheet and go to cell **B9** in the **Original GC Method** calculator.

3 Enter the original method settings (inputs). In this case, enter **Helium**, **50**, **20.00**, **30**, **0.25**, and **14.696** in cells **B9–B14** as shown in Figure 8.



	A	B
1		
2	Agilent Technologies	Effluent Sp
3		
4		
5	Original GC Method	
6		
7		
8		INPUTS
9	Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
10	Initial Oven Temp (C)	50
11	Inlet Pressure (psig)	20.000
12	Length GC Column (m)	30
13	Diameter GC Column (mm)	0.25
14	Absolute column outlet (detector) pressure (psia)	14.696
15	Column average linear velocity (cm/sec)	40.70
16	Column flow (mL/min)	1.96
17	Column holdup time (min)	1.23
18		

Figure 8 Original method properties 1

4 Cells **B15 – B17** display the original method's settings:

- **Column average linear velocity** 40.7 cm/s
- **Column flow** 1.96 mL/min
- **Column holdup time** 1.23 min

Calculate new method settings

Since 3 psig (17.696 psia) is the desired splitter pressure, it is necessary to find the new inlet pressure and flow that will match the column average linear velocity of the original method.

1 Change the column outlet pressure in cell **B11** from **14.696** to **17.696** psia (Figure 9).

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Original GC Method

		INPUTS
9	Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
10	Initial Oven Temp (C)	50
11	Inlet Pressure (psig)	20.000
12	Length GC Column (m)	30
13	Diameter GC Column (mm)	0.25
14	Absolute column outlet (detector) pressure (psia)	17.696
15	Column average linear velocity (cm/sec)	35.25
16	Column flow (mL/min)	1.77
17	Column holdup time (min)	1.42

Figure 9 Original method properties 2

Note that the column average linear velocity has dropped from 40.7 cm/s to 35.25 cm/s. This would result in longer retention times.

2 To determine the inlet pressure required to restore the column average linear velocity to 40.7 cm/s, use the Excel **Goal Seek** function as follows:

a In Excel, select **Tools / Goal Seek**.

b Fill in **B15** (the **Column average linear velocity**) as the set cell, **40.7** as the value, and **B11** (**Inlet pressure**) as the cell to be changed (Figure 10).

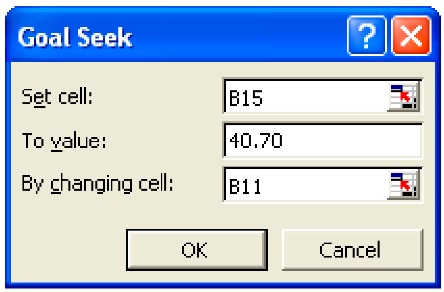


Figure 10 Goal Seek setup

- Click **OK**. The function finds the inlet pressure to restore the column average linear velocity to 40.7 cm/s (Figure 11).

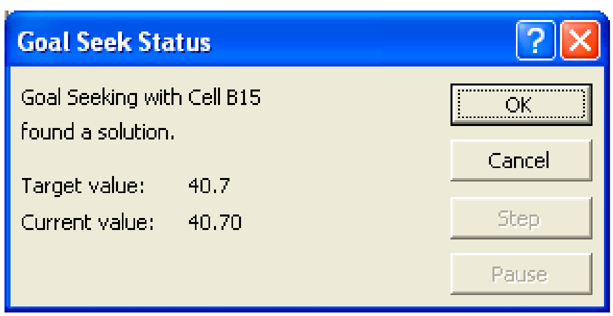


Figure 11 Goal Seek status

- Click **OK** again to accept the value (Figure 12).

	A	B
1		
2	Agilent Technologies	
3		
4		
5	Original GC Method	
6		
7		
8		INPUTS
9	Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
10	Initial Oven Temp (C)	50
11	Inlet Pressure (psig)	22.775
12	Length GC Column (m)	30
13	Diameter GC Column (mm)	0.25
14	Absolute column outlet (detector) pressure (psia)	17.696
15	Column average linear velocity (cm/sec)	40.70
16	Column flow (mL/min)	2.16
17	Column holdup time (min)	1.23

Figure 12 Original method properties 3

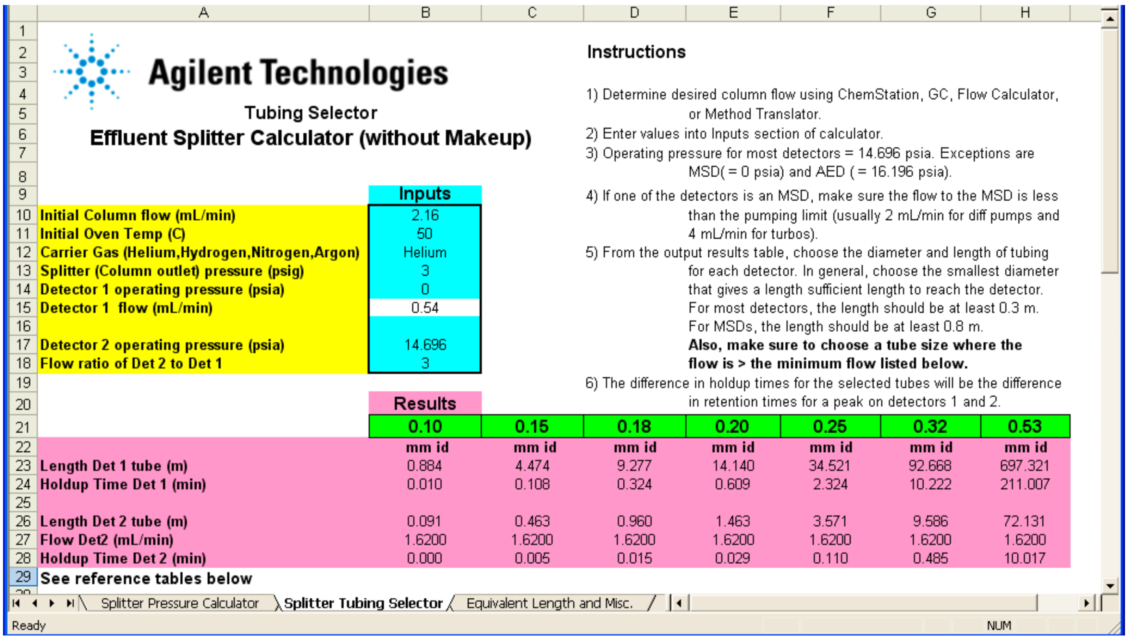
The new inlet pressure is 22.775 psig and the new column flow is 2.16 mL/min.

Calculate acceptable restrictor tube dimensions

Given the new flow, the next step is to choose restrictor tubes.

1 Select the **Splitter Tubing Selector** tab.

2 Following the instructions on the worksheet, fill in cells **B10 – B18** as shown in Figure 13.



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Tubing Selector

Effluent Splitter Calculator (without Makeup)

Instructions

1) Determine desired column flow using ChemStation, GC, Flow Calculator, or Method Translator.
2) Enter values into Inputs section of calculator.
3) Operating pressure for most detectors = 14.696 psia. Exceptions are MSD(= 0 psia) and AED (= 16.196 psia).
4) If one of the detectors is an MSD, make sure the flow to the MSD is less than the pumping limit (usually 2 mL/min for diff pumps and 4 mL/min for turbos).
5) From the output results table, choose the diameter and length of tubing for each detector. In general, choose the smallest diameter that gives a length sufficient length to reach the detector. For most detectors, the length should be at least 0.3 m. For MSDs, the length should be at least 0.8 m. **Also, make sure to choose a tube size where the flow is > the minimum flow listed below.**
6) The difference in holdup times for the selected tubes will be the difference in retention times for a peak on detectors 1 and 2.

	Inputs
Initial Column flow (mL/min)	2.16
Initial Oven Temp (C)	50
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Splitter (Column outlet) pressure (psig)	3
Detector 1 operating pressure (psia)	0
Detector 1 flow (mL/min)	0.54
Detector 2 operating pressure (psia)	14.696
Flow ratio of Det 2 to Det 1	3

Results	0.10 mm id	0.15 mm id	0.18 mm id	0.20 mm id	0.25 mm id	0.32 mm id	0.53 mm id
Length Det 1 tube (m)	0.884	4.474	9.277	14.140	34.521	92.668	697.321
Holdup Time Det 1 (min)	0.010	0.108	0.324	0.609	2.324	10.222	211.007
Length Det 2 tube (m)	0.091	0.463	0.960	1.463	3.571	9.586	72.131
Flow Det2 (mL/min)	1.6200	1.6200	1.6200	1.6200	1.6200	1.6200	1.6200
Holdup Time Det 2 (min)	0.000	0.005	0.015	0.029	0.110	0.485	10.017

See reference tables below

Figure 13 Splitter tubing selector

The columns under the headings **0.10, 0.15,...0.53** are the lengths of restrictor tubing required for the diameter given as the column heading.

NOTE

In this example, the MSD was set as detector 1.

If you have UCDFS tubing diameters that are not shown in the spreadsheet, the id values in a column heading can be edited and the other values in the column will be appropriately recalculated.

In general, it is preferable to choose tubes that are closest to the minimum lengths to reach from the splitter to the detectors (0.8 m for MSDs without the QuickSwap accessory, at least 0.5 m for FPDs, and at least 0.3 m for the rest).

Select the most appropriate restrictor tube dimensions from the calculator table

From the results, if 0.1-mm id UCDFS tubing is available, the length required, 0.884 m, would fit to the MSD.

The restrictor tube to the FID (detector 2) would be 0.463 m of 0.15-mm id UCDFS if available, or 0.960 m of 0.18-mm id UCDFS. Since 0.18-mm id tubing is included with the splitter consumables kit, it was chosen for this example.

Assemble the splitter configuration with 0.884 m of 0.1-mm id restrictor tubing to the MSD and 0.960 m of 0.18-mm id to the FID.

Since this is a configuration example, we will skip the remaining steps (step 6 and step 7 on page 27).

Other calculations

When splitting to an MSD other calculations may be useful. See “Changing Parameters for an Assembled Splitter Setup” on page 35 for instructions on how to determine other useful parameters, like the inlet minimum and maximum pressures, that can be used with this MSD setup.

Changing Parameters for an Assembled Splitter Setup

Once you have designed, cut, and assembled a restrictor tubing configuration, you may wish to calculate what effect changing parameters would have on the system. For example, if you want to change the inlet pressure (and thus column flow) with the MSD setup in Example 2, it would be useful to calculate the maximum and minimum acceptable inlet pressures.

The minimum inlet pressure, excluding chromatographic effects, is the inlet pressure at which the splitter pressure drops to near atmospheric pressure, about 0.5 psig (15.196 psia). At atmospheric pressure, the MSD will start sucking gases from the FID and the splitter will no longer work.

The upper limit on inlet pressure will be that which results in the maximum flow through the tube to the MSD, either 2 mL/min or 4 mL/min, depending on the MSD’s vacuum pump.

The **Splitter GC Method** calculator on the **Splitter Pressure Calculator** worksheet can answer questions of this nature. After inputting the dimensions and parameters of a given splitter setup, you can alter the input values to determine the effects of changes.

To determine the inlet pressure operating range for Example 2, select the **Splitter Pressure Calculator** worksheet and input the parameters determined in the example into the **Splitter GC Method** calculator. See Figure 14.

Splitter GC Method

	INPUTS
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Initial Oven Temp (C)	50
Inlet Pressure (psig)	22.775
Length GC Column (m)	30
Diameter GC Column (mm)	0.25
Length Detector 1 tube (m)	0.884
Diameter Detector 1 tube (mm)	0.1
Absolute Detector 1 operating pressure (psia)	0
Length Detector 2 tube (m)	0.96
Diameter Detector 2 tube (mm)	0.18
Absolute Detector 2 operating pressure (psia)	14.696

	RESULTS
Column average linear velocity (cm/sec)	40.69
Column flow (mL/min)	2.16
Column hold-up time (min)	1.23
Splitter Pressure (psig)	3.008
Splitter Pressure (psia)	17.704
Flow Detector 1 (mL/min)	0.54
Hold-up time Detector 1 (min)	0.0095
Flow Detector 2 (mL/min)	1.62
Hold-up time Detector 2 (min)	0.0153
Flow ratio of Det 2 to Det 1	3.01
Percent flow to detector 1	24.97%

Figure 14 Example initial parameters

Note that some of the calculated output parameters vary slightly from those calculated above due to rounding errors, but that the overall agreement is good.

To determine the inlet pressure that produces 0.5 psig in the splitter, use the Excel **Goal Seek** function.

1 Select **Tools / Goal Seek**.

2 Fill in **E25** (the splitter pressure in psig) as the set cell, **0.5** as the value, and **E11** as the cell to be changed (Figure 15).

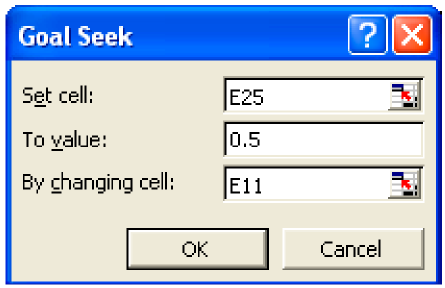

Figure 15 Changing the column length

3 Click **OK.** The function finds the inlet pressure that produces 0.5 psig splitter pressure (Figure 16).

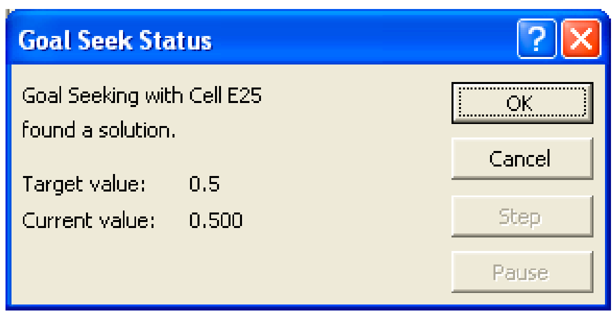

Figure 16 Goal Seek Status

4 Click **OK** again to accept the value. See Figure 17.

Splitter GC Method

	INPUTS
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Initial Oven Temp (C)	50
Inlet Pressure (psig)	8.907
Length GC Column (m)	30
Diameter GC Column (mm)	0.25
Length Detector 1 tube (m)	0.884
Diameter Detector 1 tube (mm)	0.1
Absolute Detector 1 operating pressure (psia)	0
Length Detector 2 tube (m)	0.96
Diameter Detector 2 tube (mm)	0.18
Absolute Detector 2 operating pressure (psia)	14.696

	RESULTS
Column average linear velocity (cm/sec)	17.77
Column flow (mL/min)	0.65
Column hold-up time (min)	2.81
Splitter Pressure (psig)	0.500
Splitter Pressure (psia)	15.196
Flow Detector 1 (mL/min)	0.40
Hold-up time Detector 1 (min)	0.0111
Flow Detector 2 (mL/min)	0.25
Hold-up time Detector 2 (min)	0.0920
Flow ratio of Det 2 to Det 1	0.63
Percent flow to detector 1	61.51%

Figure 17 Example minimum inlet pressure calculation

The result shows that the inlet pressure should not be set below 8.9 psig. Note that other ramifications would be bad chromatography from the low average linear velocity and low flows sweeping the splitter connections. Note also that the split ratio between detectors has changed.

To calculate the maximum inlet pressure (in this example, that pressure which sends 4 mL/min into the MSD), use the **Goal Seek** function again:

1 Select **Tools / Goal Seek**.

2 Fill in **E27** (Flow to Detector 1) as the set cell, **4.00** as the value, and **E11** as the cell to be changed (Figure 18).

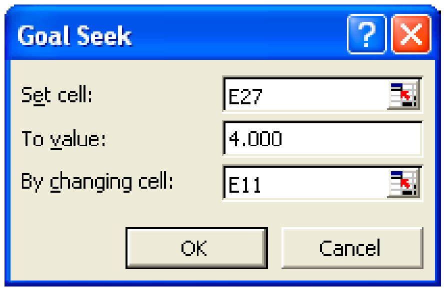

Figure 18 Goal Seek

3 Click **OK**. The function then finds the inlet pressure that sends 4 mL/min to the MSD (Figure 19).

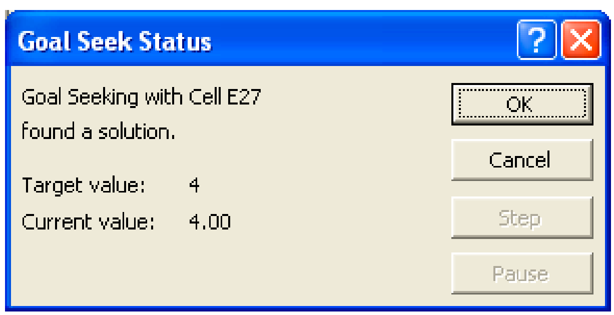

Figure 19 Goal Seek status

4 Click **OK** again to accept the value. See Figure 20.

Splitter GC Method	
	INPUTS
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Initial Oven Temp (C)	50
Inlet Pressure (psig)	133.654
Length GC Column (m)	30
Diameter GC Column (mm)	0.25
Length Detector 1 tube (m)	0.884
Diameter Detector 1 tube (mm)	0.1
Absolute Detector 1 operating pressure (psia)	0
Length Detector 2 tube (m)	0.96
Diameter Detector 2 tube (mm)	0.18
Absolute Detector 2 operating pressure (psia)	14.696
	RESULTS
Column average linear velocity (cm/sec)	197.88
Column flow (mL/min)	39.07
Column hold-up time (min)	0.25
Splitter Pressure (psig)	33.473
Splitter Pressure (psia)	48.169
Flow Detector 1 (mL/min)	4.00
Hold-up time Detector 1 (min)	0.0035
Flow Detector 2 (mL/min)	35.07
Hold-up time Detector 2 (min)	0.0015
Flow ratio of Det 2 to Det 1	8.77
Percent flow to detector 1	10.24%

Figure 20 Example maximum inlet pressure calculation

Thus, the operating range of inlet pressure is 8.9—133 psig, at least from the point of view of flow to the MSD. In practice, the inlet pressure should not cause the splitter pressure to rise above 4.0 psig (above 4.0 psig, the splitter flow calculations become inaccurate). Performing a Goal Seek with a target splitter pressure of 4.0 in cell E25 and using cell E11 as the cell to be changed provides a practical upper limit on inlet pressure of 27.435 psig.

4
Restrictor and Column Installation

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To Connect the Splitter 42
To Disconnect Tubing from the Splitter 45
Protect the column and restrictors 45
Protect the splitter 45

This chapter shows how to assemble and disassemble splitter hardware.

To Install the Column

NOTE

Restrictors and the column exit are connected to the splitter assembly using Agilent internal nuts and SilTite ferrules. See "Swaging SilTite Ferrules" on the CD for details.

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.

To Connect the Splitter

1. Connect the restrictor tubing to the connectors on the splitter (Figure 21). Finger-tighten until just snug, then tighten with a wrench an additional 15 degrees (Figure 22). Install the back detector's restrictor first.
2. Connect the column exit to the splitter. Tighten as you did the restrictors.

CAUTION

Arrange all tubes (restrictors and column) so that they do not touch the oven walls. This could create a cold spot.

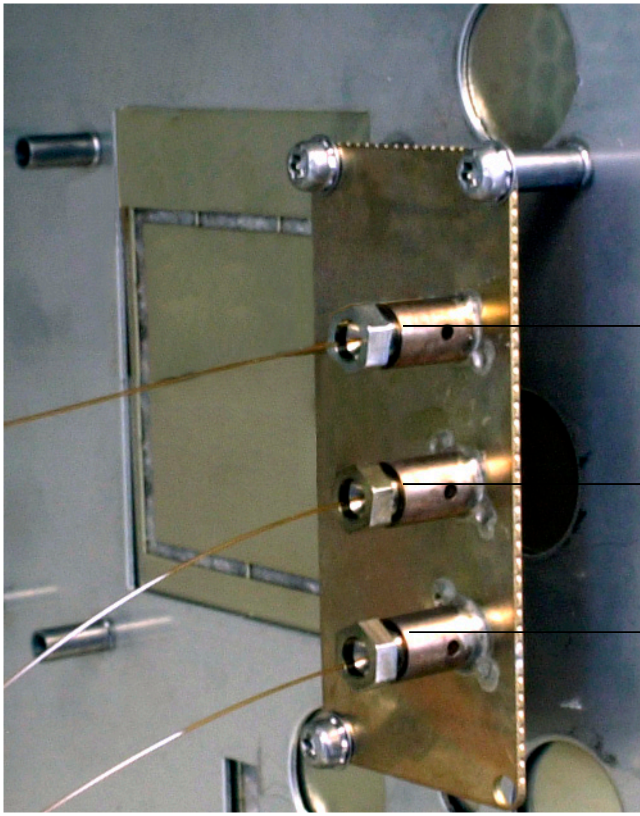

Figure 21 Restrictor and column connections

CAUTION

Do not overtighten the fittings. The dashed line in Figure 22 (about 15 degrees clockwise from finger-tight) is usually enough.

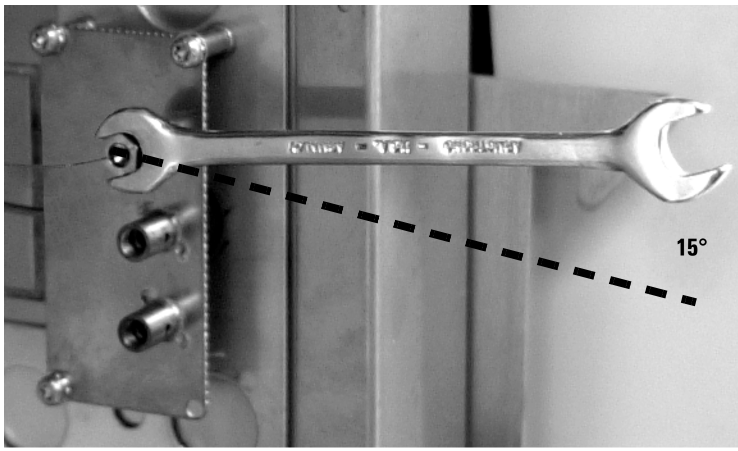

Figure 22 Tightening the connections

To 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, such as a pen or a paper clip, and insert it in the ferrule release hole in the side of the fitting (Figure 23). Press firmly. The ferrule will click when it breaks free.

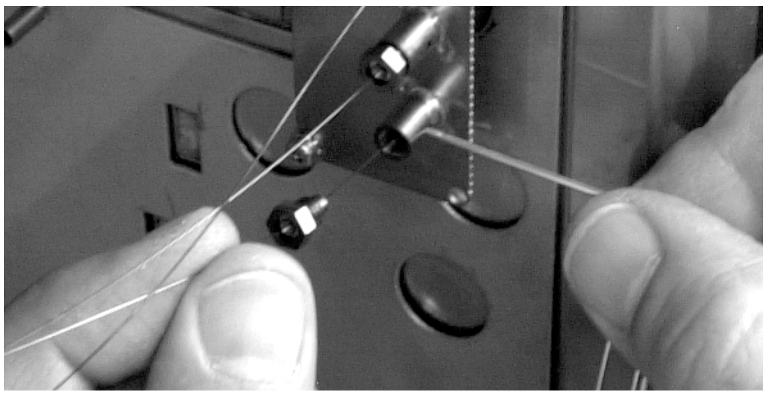

Figure 23 Releasing a ferrule

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

Because there is no inert gas purge, heat the oven ONLY when the splitter is connected to the column and the carrier gas is on. Heating the oven without column carrier gas flowing into the splitter damages the splitter's deactivation.

Always remove the splitter from the oven when not in use. Protect it from contamination by installing plugs on all fittings.

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