Agilent G3180B Two-Way Splitter Kit

With Makeup Gas

Installation and Operation Guide

Agilent Technologies

Notices

Manual Part Number

G3180-90120

Supercedes G3180-90110

Edition

First edition, April 2006

Printed in USA

Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610 USA

Acknowledgement

Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

Safety Notices

CAUTION

A **CAUTION** 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 damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

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 G3180 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 between the two detectors) is governed by two 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 G3180B effluent splitter with makeup gas 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 between two 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 G3180B splitter.

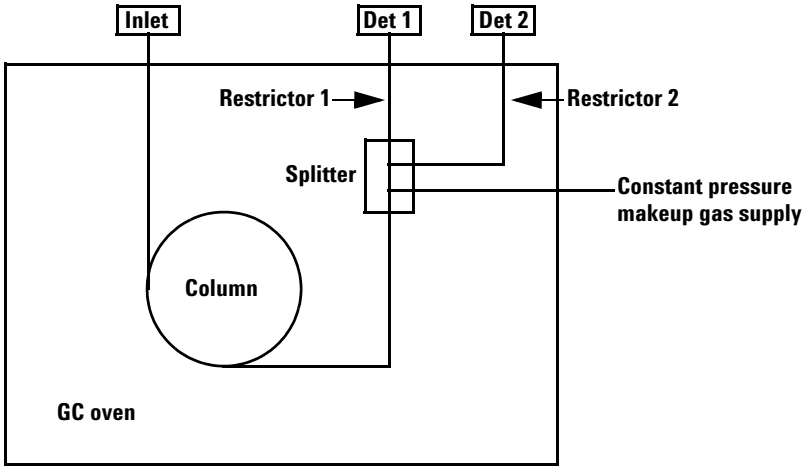

Figure 1 Splitter plumbing

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 flow through each restrictor changes with oven temperature, the *ratio* of the two flows at any temperature is the same.

Details

The G3180B kit addresses several limitations of previous approaches to splitting column effluent between two 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 two 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 by 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 two 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.

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.

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 205, 301, or 308) or a Pneumatics Control Module (PCM).

Other Requirements

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

Parts Supplied

The G3180B kit contains the following parts (Table 1).

Table 1 Parts supplied

Part number	Description	Quantity
0100-0124	Union, stainless steel, 1/16-inch tubing	2
0100-0241	Union, stainless steel, 1/8 to 1/16-inch reducing	1
G1580-00130	Valve box blanking plate	1
G1530-01340	Capillary column spring clips	4
0515-0374	Screw, M3 × 10 mm	7
G2855-60140	Oven bracket assembly	1
G2855-60560	T-screw oven bracket retainer	2
G2855-80022	Manual and calculator CD	1
G3180-90120	Manual, G3180B	
0100-2354	Tubing, stainless steel, 1/16-inch od × 0.01-inch id, 1 m	1
G3180-61500	Compact splitter with makeup gas assembly	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.

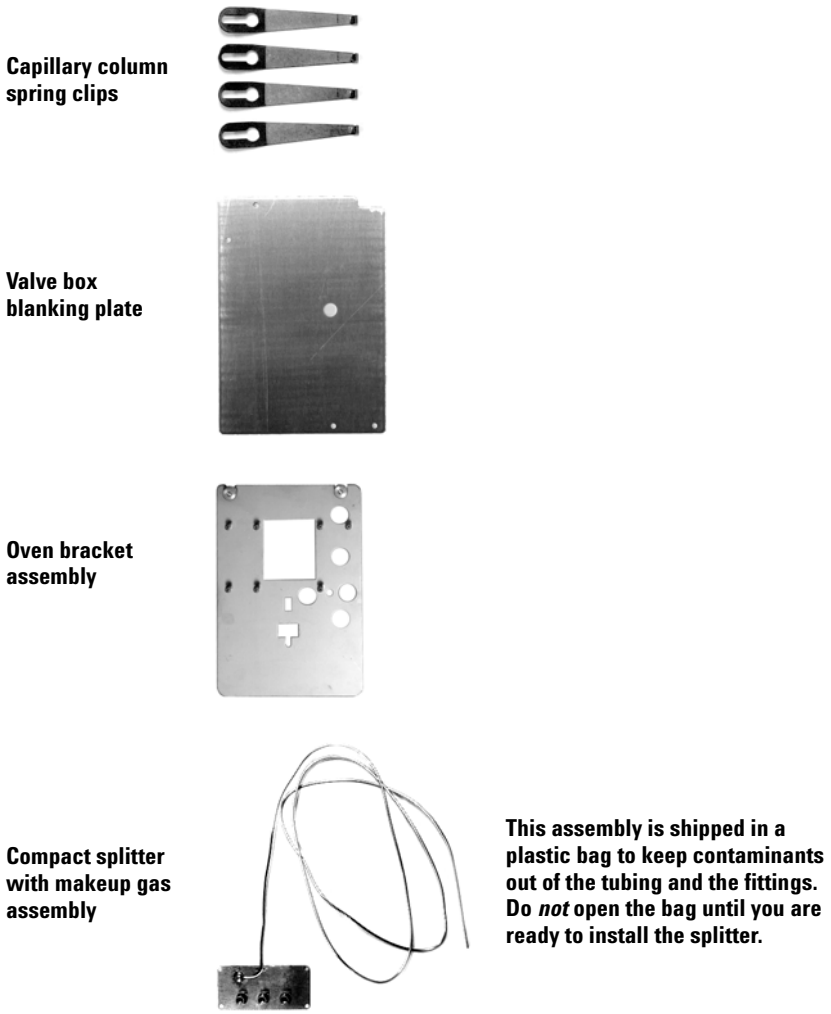

Figure 2 Part identification

Parts Not Supplied

Brown-dot frit (19231-60610)

Tools Required

Side cutter, large

Open-end wrenches

1 Introduction

2
Hardware 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 top.

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.

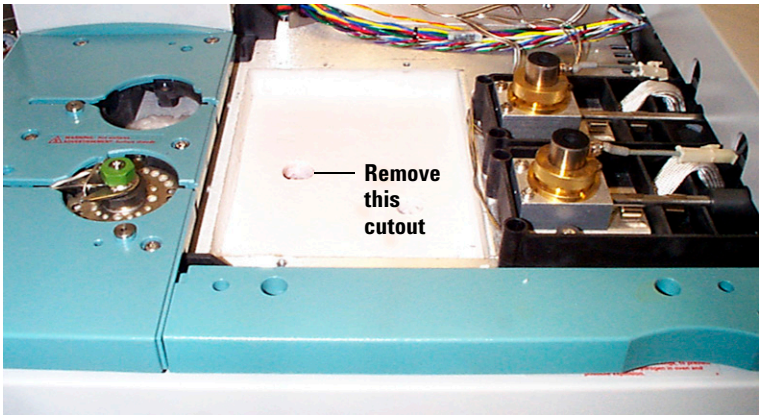

Figure 4 Remove the insulation cutout

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.

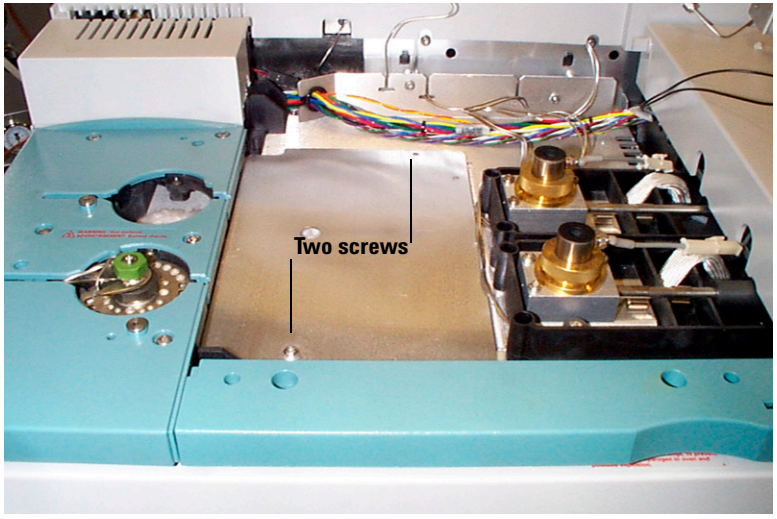

Figure 5 Install valve box blanking plate

Install the Column Clips

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

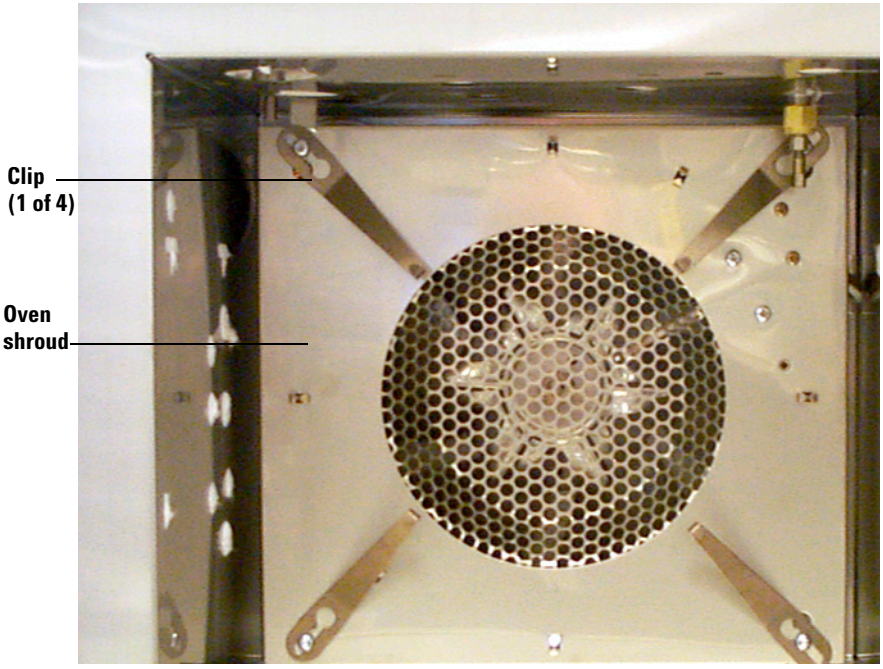

Figure 6 Oven clips

Install the Bracket and Splitter

The 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.

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).

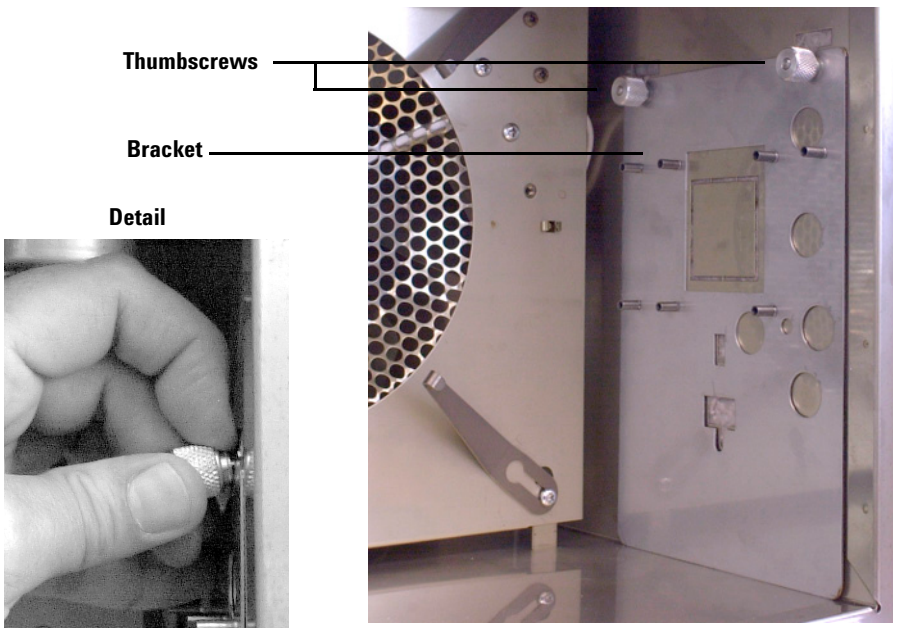

Figure 7 Installing the bracket

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.

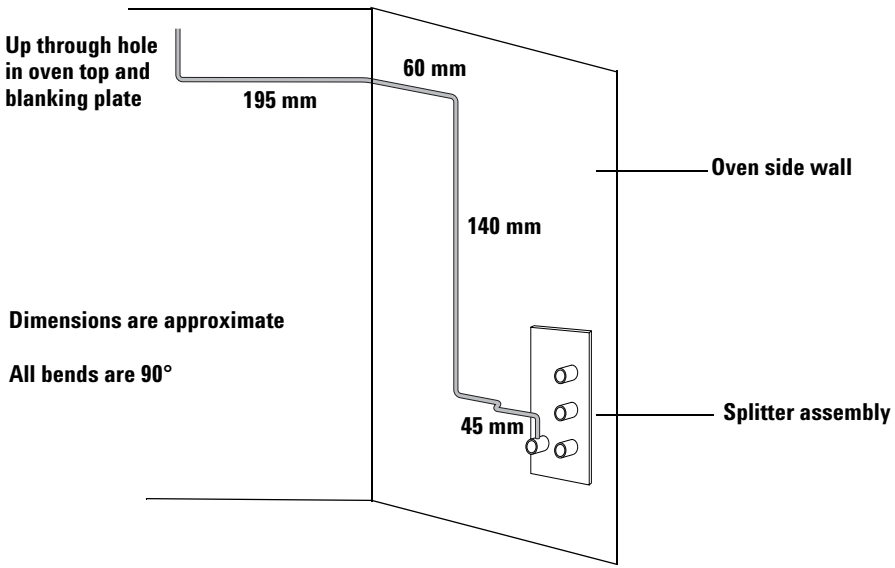

Figure 8 Prebending the splitter tubing

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.

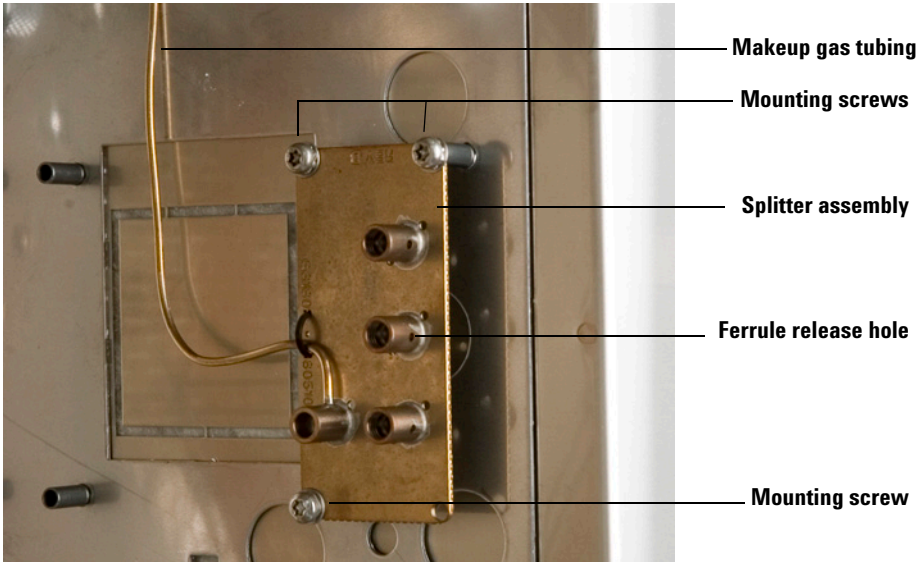

Figure 9 Installing the splitter assembly

Connect the Makeup Gas Supply

Connect the makeup gas source to the PCM or Auxiliary Pressure controller.

To supply the makeup gas froma PCM

1. Connect the tubing from the PCM to the 1 meter 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.

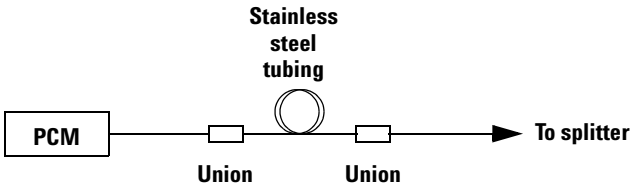

Figure 10 Plumbing a PCM makeup supply

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) between the two 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 two detectors. The dimensions of the restrictors are chosen to give the desired 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 two detectors
- The flow rate requirements of the two detectors

Table 2 lists typical splitting configurations. Table 3 shows the resulting gas flows. All calculations assume helium as the carrier gas.

Table 2 Restrictor configurations

Configuration	Det 1	Det 2	Split ratio, Det 2/Det 1	Diam R1, mm id	Length R1, m	Diam R2, mm id	Length R2, m
1	atm*	atm	1	0.25	0.544	0.25	0.544
2	atm	atm	5	0.18	0.418	0.25	0.311
3	atm	MSD,D**	1	0.18	1.060	0.18	2.890
4	atm	MSD, T***	1	0.18	0.530	0.18	1.440
5	atm	MSD, D	2	0.18	2.130	0.18	2.890
6	atm	MSD, T	2	0.18	1.064	0.18	1.443
7	atm	MSD, D	5	0.10	0.507	0.18	2.890
8	atm	MSD, T	5	0.18	2.660	0.18	1.443

* atm Atmospheric pressure detectors such as FID, TCD, ECD, FPD and NPD
** MSD, D MSD with diffusion pump or standard turbo pump (2 mL/min flow capability)
*** MSD, T MSD with performance turbo pump (4 mL/min flow capability); makeup pressure supply is set to 3.8 psig

Table 3 Splitter flows

	40 °C		200 °C		300 °C		400 °C	
Configuration	**Flow R1, mL/min**	**Flow R2, mL/min**	**Flow R1, mL/min**	**Flow R2, mL/min**	**Flow R1, mL/min**	**Flow R2, mL/min**	**Flow R1, mL/min**	**Flow R2, mL/min**
1	14.7	14.7	7.3	7.3	5.2	5.2	3.9	3.9
2	5.1	25.6	2.5	12.7	1.8	9.1	1.4	6.8
3	2	2	1	1	0.7	0.7	0.54	0.54
4	4	4	2	2	1.4	1.4	1.1	1.1
5	1	2	0.5	1	0.36	0.72	0.27	0.54
6	2	4	1	2	0.71	1.4	0.53	1.06
7	0.4	2	0.2	1	0.14	0.7	0.1	0.5
8	0.8	4	0.4	2	0.28	1.4	0.21	1.1

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). To plumb this system, 0.544-m lengths of 0.25-mm id uncoated deactivated fused silica tubing are connected as restrictors from the splitter to the two detectors.

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 psi. 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 7.3 mL/min. The total flow will be 14.6 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 13.6 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 400 °C, the column flow should not exceed 6.8 mL/min ([3.9 + 3.9] –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 psig 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 9.4 mL/min ([5.2 + 5.2] – 1 = 9.4) is 56.3 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.

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 should be expected. They are shown in the configuration tables more as a caution than as a recommendation.

In practice, the column flow can be set to within 0.5 mL/min of the total flow if necessary. For example, the 1:1 split to an MSD with a performance turbo pump running a method programmed to 300 °C should have a column flow of no more than 2.3 mL/min ([1.4 + 1.4] – 0.5) at 300 °C.

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).

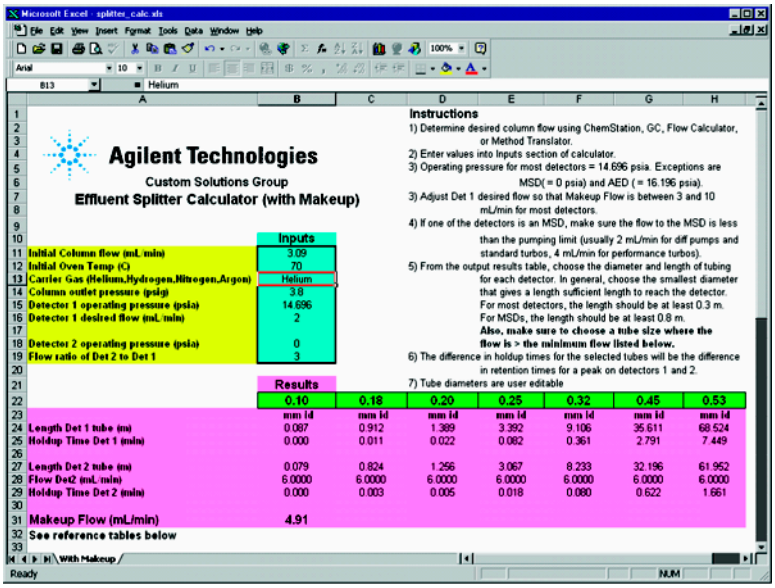

Figure 11 Effluent Splitter Calculator

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

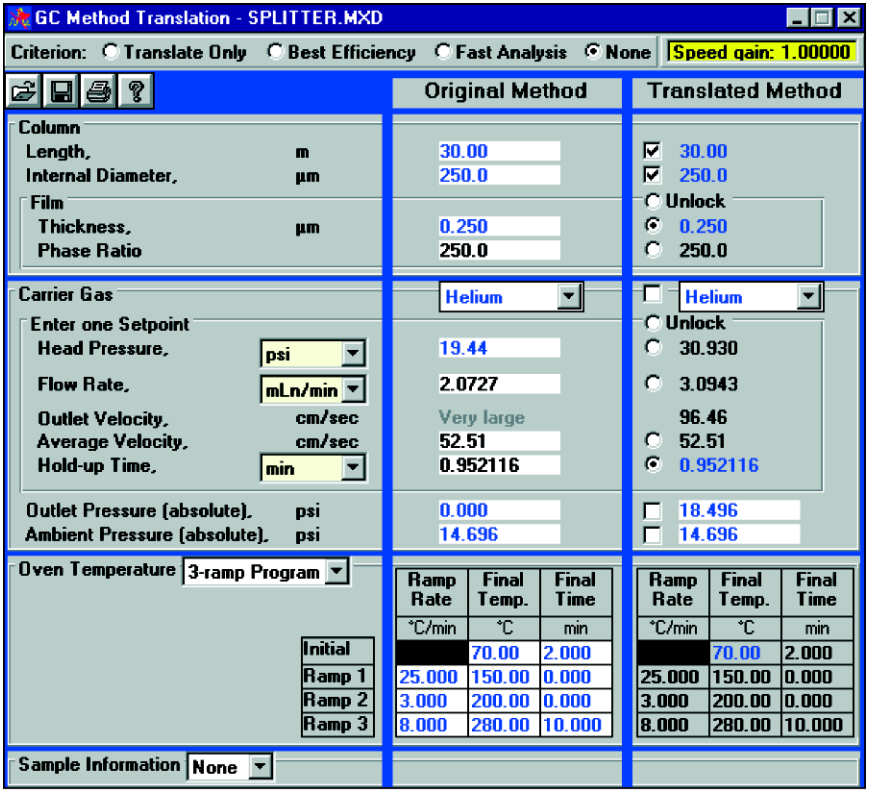

Figure 12 GC Method Translation

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

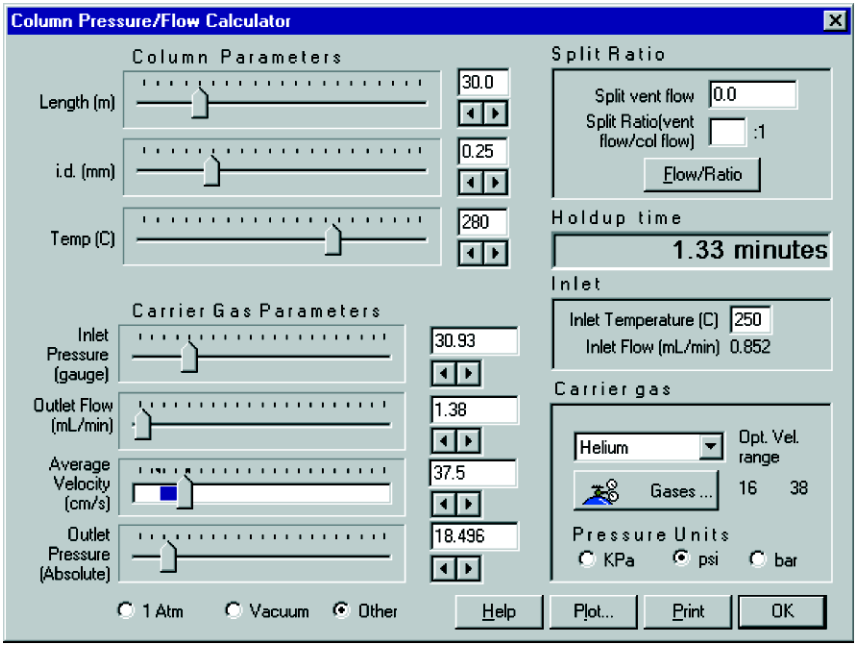

Figure 13 Column Flow/Pressure Calculator

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, Hydrogen, Nitrogen, or Argon.
- **Detectors 1 and 2 operating pressure (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 Ratio of Detector 2 to Detector 1**. This is the desired split ratio between the two detectors. Usually this number is 1, meaning the effluent divides equally between 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.

Make sure that the flow through each restrictor tube is at least equal to the suggested minimum flow in Table 4. Restrictors that fail this test 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 31 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.

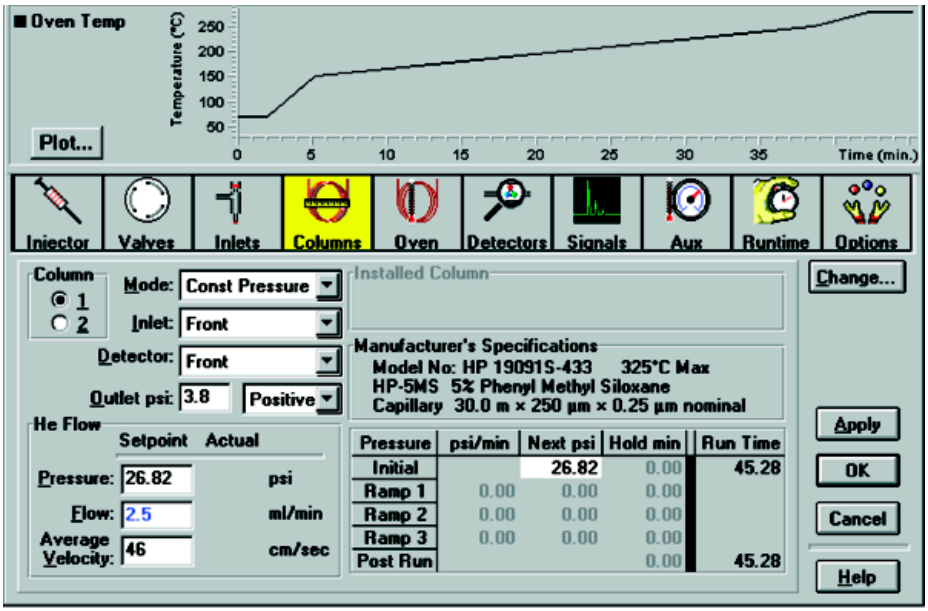

Figure 14 Column outlet pressure screen

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

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

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. Connect the restrictors to the connectors on the splitter (Figure 15). Finger-tighten until just snug, then tighten with a wrench an additional 15° (Figure 16). Install the back restrictor first.
2. Connect the restrictors to the appropriate detectors.
3. 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.

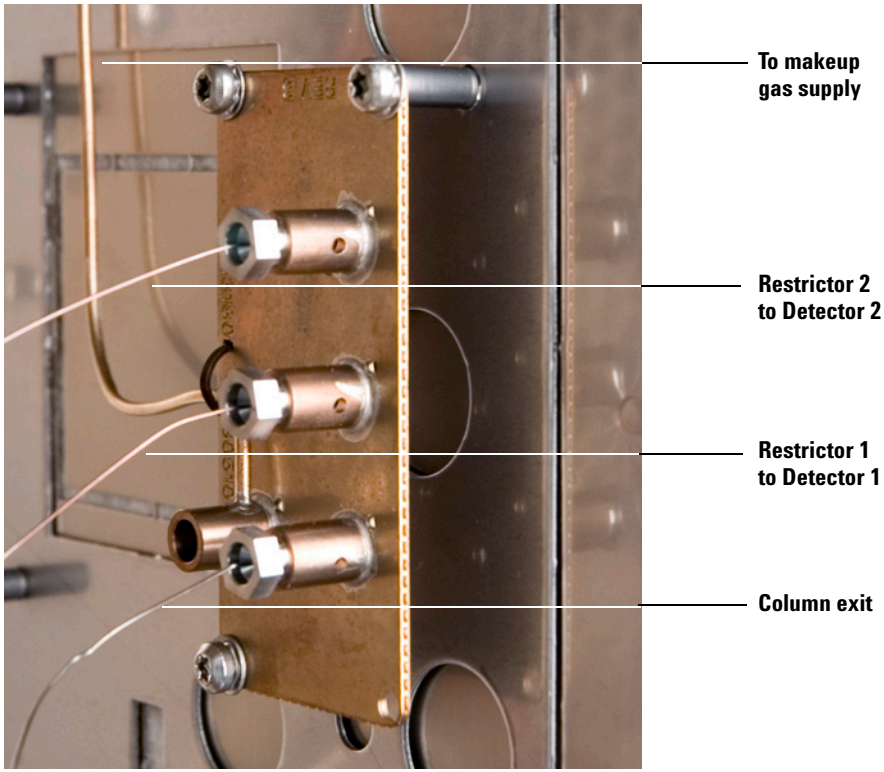

Figure 15 Restrictor and column connections

CAUTION Do not overtighten the fittings. The dashed line in Figure 16 (about 15° clockwise from finger-tight) is usually enough.

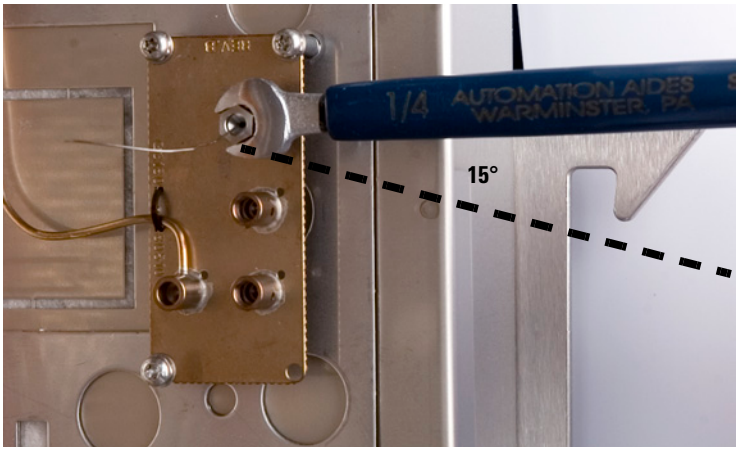

Figure 16 Tightening the connections

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 17). Press firmly. The ferrule will click when it breaks free.

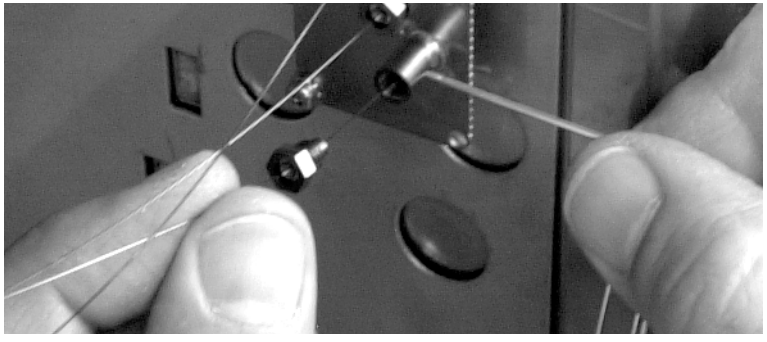

Figure 17 Releasing a ferrule

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 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.

Leave the excess wire on the other end to serve as a handle when removing the plug.

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. The initial oven temperature is 70 °C and is programmed to 280 °C. The method is run 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 1:3 between the ECD (detector 1) and an MSD (detector 2). 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 18) is useful for this. Use the **None** mode and check the button to make the hold-up times the same.

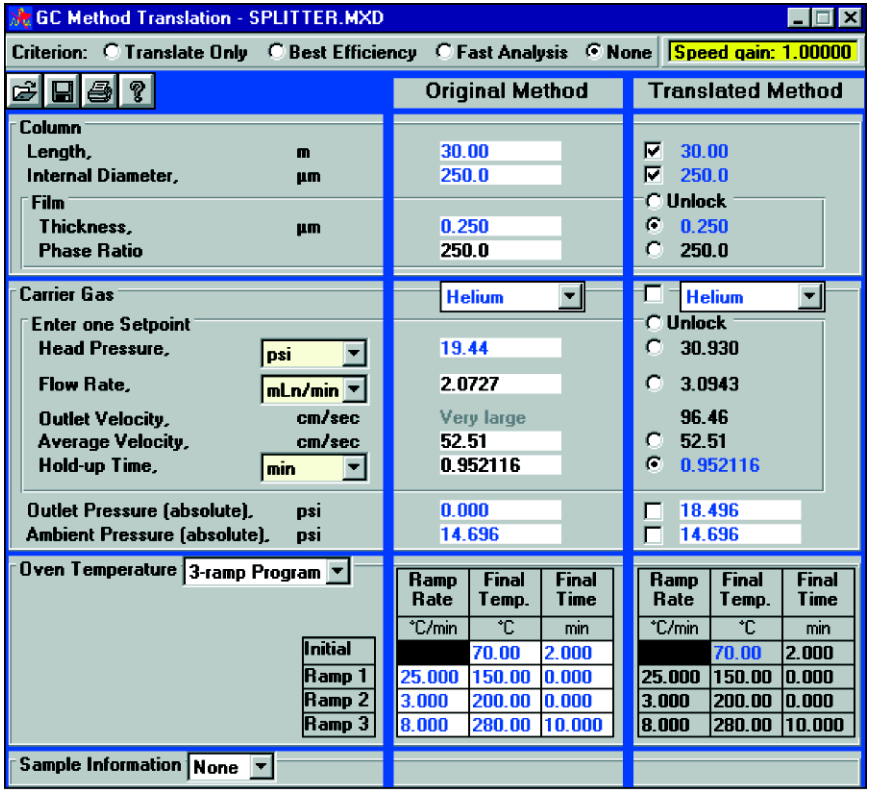

Figure 18 Calculating column flow

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 "splitter_calc.xls" in Excel. We will choose to have 2 mL/min go to the ECD initially. With a split ratio of 3, this will send 6 mL/min to the MSD. This flow is acceptable with a performance turbo system but will give somewhat degraded detection limits. Fill in the input column as shown (Figure 19) with the ECD assumed to be Detector 1 and the MSD as Detector 2.

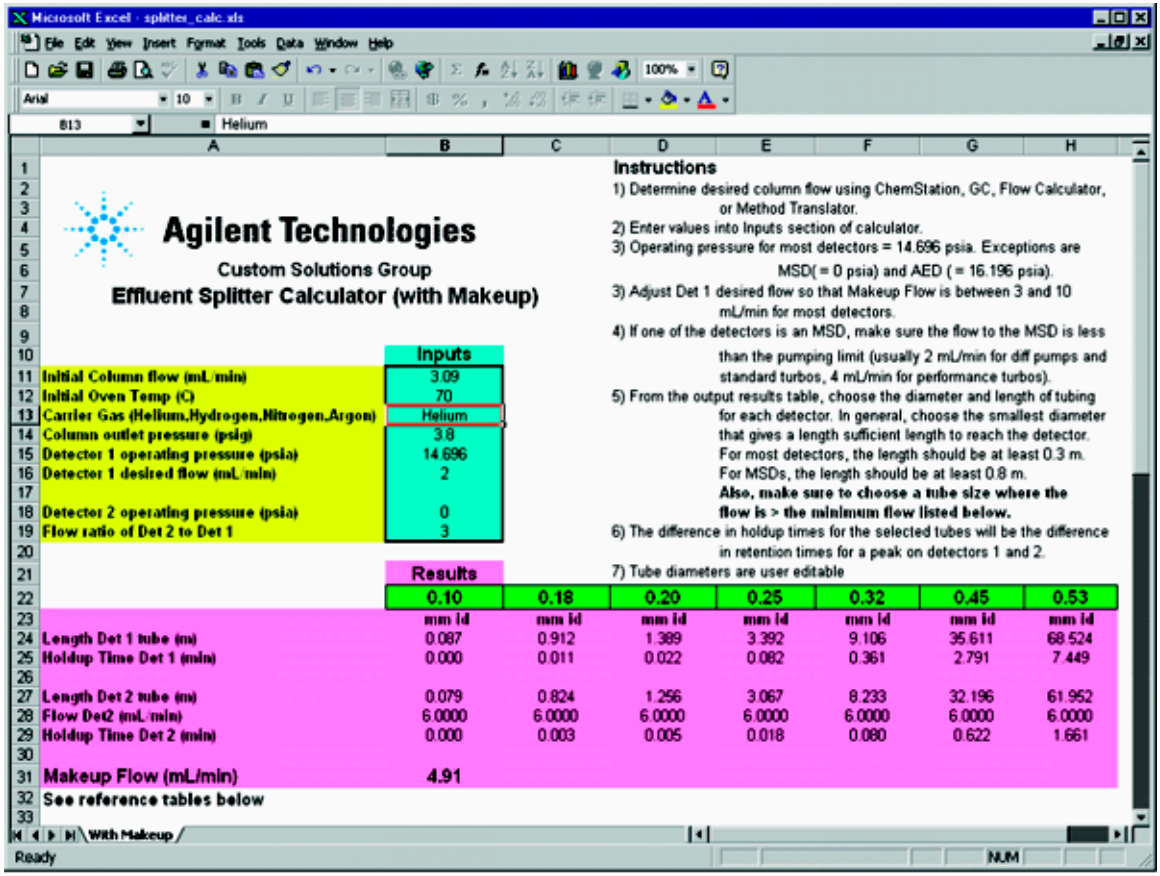

Figure 19 The Effluent Splitter calculator

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, requiring 0.912 m for the ECD restrictor and 0.824 m for the MSD restrictor.

Table 4 on page 33 shows that in both cases the flow is higher than the minimum 0.72 mL/min suggested for helium in 0.18-mm id tubing.

Calculate column flow

To find the makeup flow at 280 °C, first find the column flow at 280 °C. The Flow Calculator software (Figure 20) requires that the output pressure be entered in psia. Therefore 18.496 psia (3.8 psig) is entered.

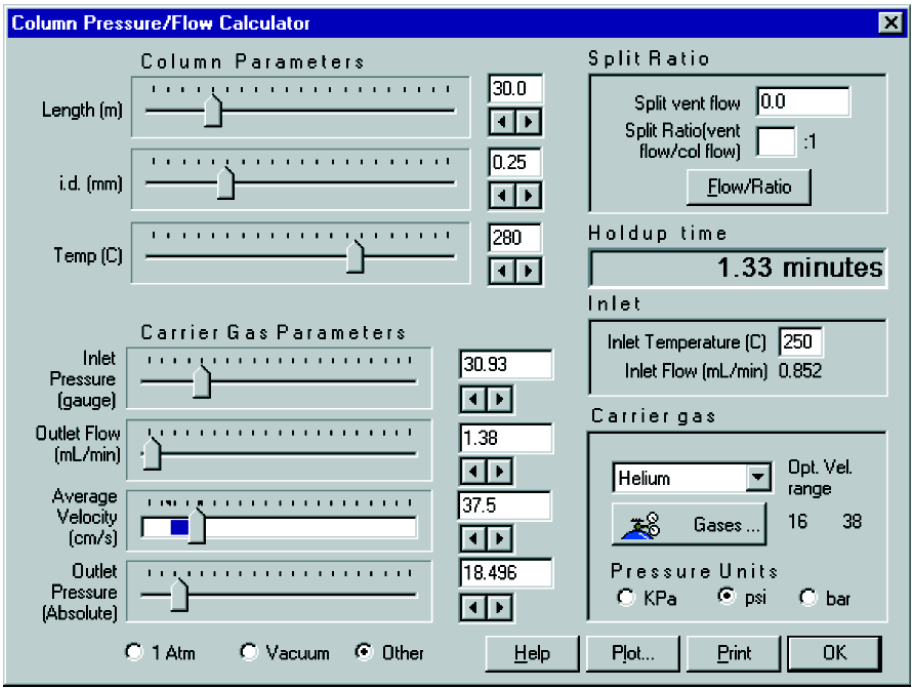

Figure 20 Column flow calculation

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

Calculate ECD restrictor flow

The flow through the ECD restrictor at 280 °C is calculated to be 0.88 mL/min (Figure 21).

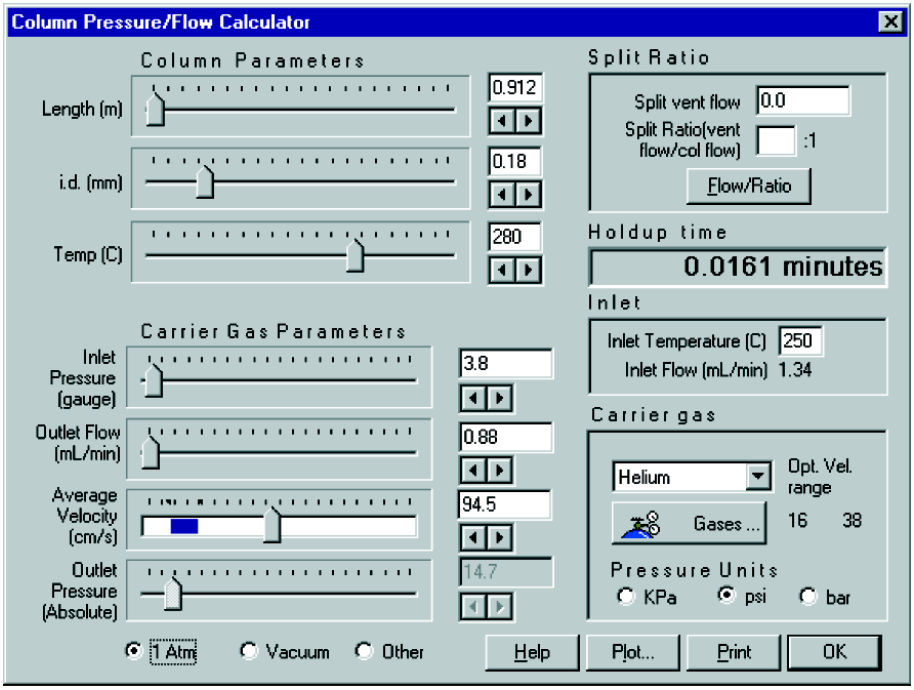

Figure 21 ECD restrictor flow calculation

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

Calculate MSD restrictor flow

The flow through the MSD restrictor (Figure 22) at 280 °C is:

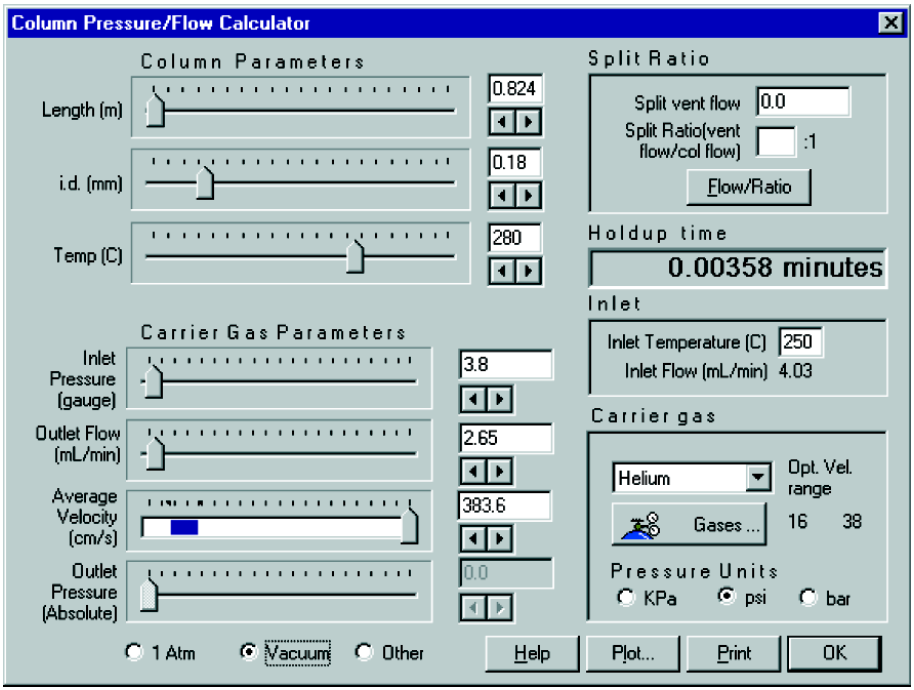

Figure 22 MSD restrictor flow calculation

The flow to the MSD at 280 °C is 2.65 mL/min. This flow is higher than the minimum 0.72 mL/min suggested for helium in 0.18-mm id tubing. The calculated makeup flow is then [0.88 + 2.65] – 1.38 = 2.15 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 purges air out of the fitting, preventing air from reaching the MSD.

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

1. Cool down 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 column in the inlet and turn on carrier gas to purge air from the column.
5. Preswage metal ferrule on the outlet end of the column.
6. Remove 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, the splitter makeup pressure is time-programmed to rise rapidly after elution of the last peak of interest while the inlet pressure decreases 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 from above, the MSD will limit the flow, and thus pressure, that can be used for backflushing. The flow allowed to go to the MSD (with a performance turbo) must be 8 mL/min or less. 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.

The restrictor to the MSD was 0.824 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 17.4 psig at 280 °C.

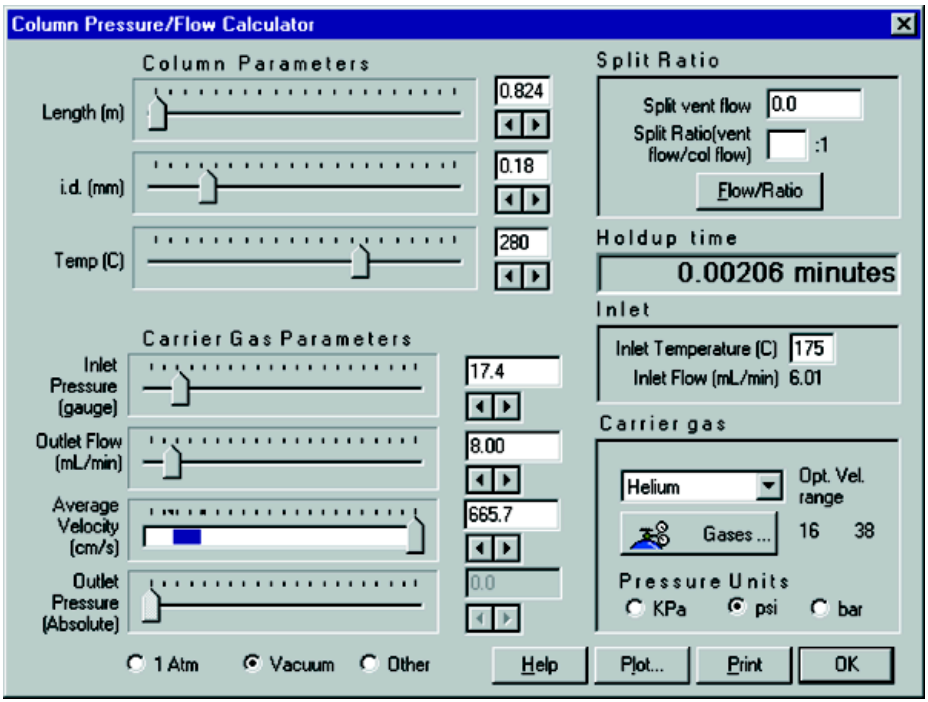

Figure 23 Column backflush flow calculation

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.

Index

Agilent Technologies

Printed in USA, April 2006

G3180-90120