Agilent
Splitter Calculator

Agilent Technologies

Notices

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Acknowledgement

In this Guide. . .

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 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.

3 Operation

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

Introduction

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 create a custom one using the splitter calculator.
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 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)
  XCD (Chemiluminescence 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 a spreadsheet calculator that is included for calculating tubing dimensions for special situations.

Figure 1 shows the plumbing configuration for the typical splitter.

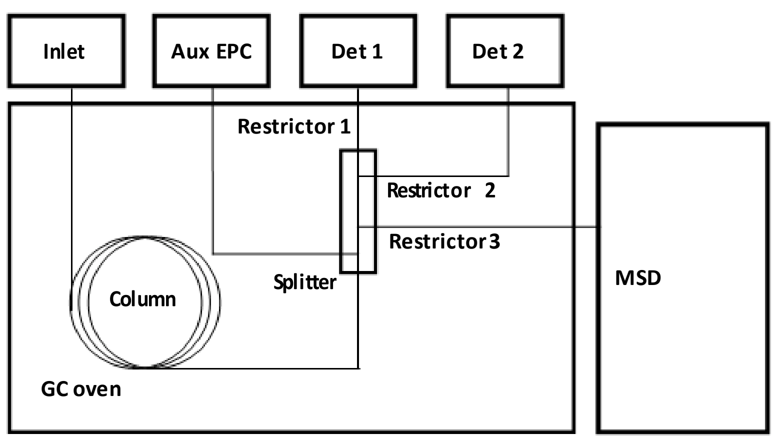


Figure 1 Splitter plumbing

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.

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.

Capillary Flow Technology

The splitting hardware is based on Capillary Flow Technology (CFT). 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.

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.

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 Splitter Configurations

The combination of restrictor diameters and lengths determines how the column effluent is divided (the split ratio) among each detector.

- Create a custom configuration. The CFT Accessories CD shipped with the splitter kit provides tools for the necessary calculations.

Typical Configurations

The important parameters when setting up a splitter are the lengths and diameters of the restrictor tubes that go to each detector. 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 each detector.
- The flow rate requirements of each detector.

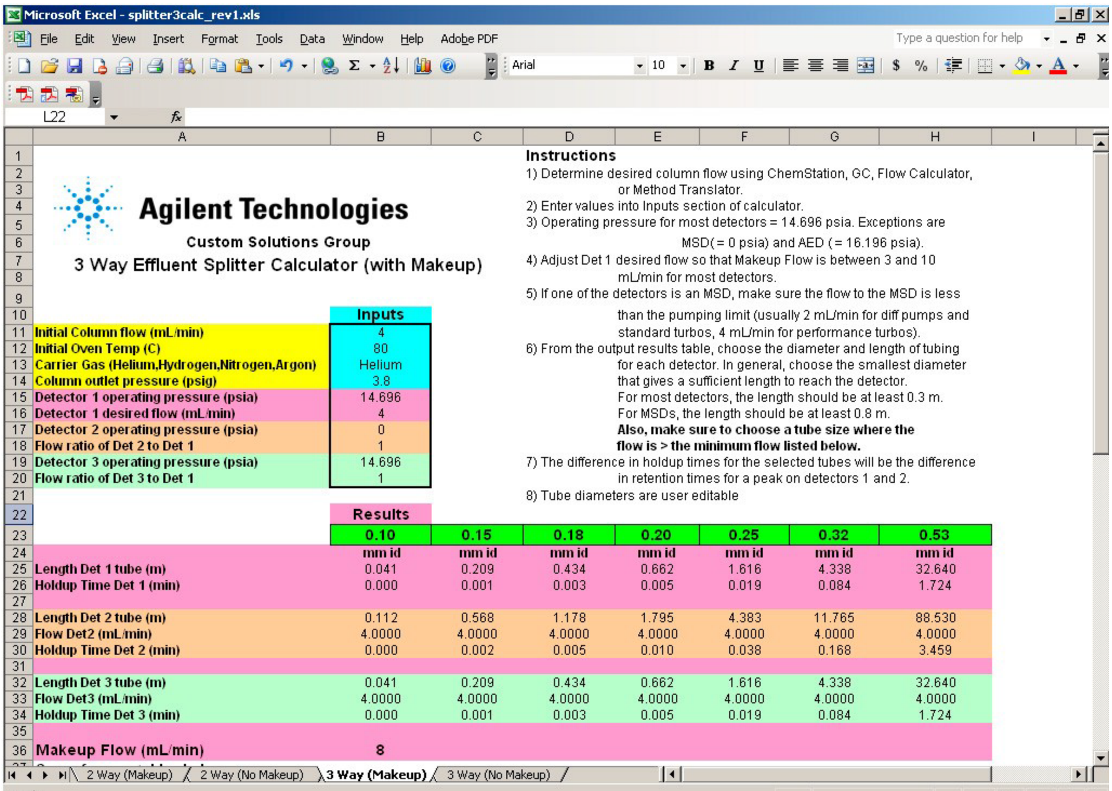
Splitting to an MSD

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.

Custom Configurations

Effluent Splitter Calculator (with Makeup)

Calculates dimensions (length and inside diameter) of restrictors to obtain a desired split ratio.



	Inputs
Initial Column flow (mL/min)	4
Initial Oven Temp (C)	80
Carrier Gas (Helium,Hydrogen,Nitrogen,Argon)	Helium
Column outlet pressure (psig)	3.8
Detector 1 operating pressure (psia)	14.696
Detector 1 desired flow (mL/min)	4
Detector 2 operating pressure (psia)	0
Flow ratio of Det 2 to Det 1	1
Detector 3 operating pressure (psia)	14.696
Flow ratio of Det 3 to Det 1	1

Results	0.10	0.15	0.18	0.20	0.25	0.32	0.53
	mm id	mm id	mm id	mm id	mm id	mm id	mm id
Length Det 1 tube (m)	0.041	0.209	0.434	0.662	1.616	4.338	32.640
Holdup Time Det 1 (min)	0.000	0.001	0.003	0.005	0.019	0.084	1.724
Length Det 2 tube (m)	0.112	0.568	1.178	1.795	4.383	11.765	88.530
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	0.002	0.005	0.010	0.038	0.168	3.459
Length Det 3 tube (m)	0.041	0.209	0.434	0.662	1.616	4.338	32.640
Flow Det3 (mL/min)	4.0000	4.0000	4.0000	4.0000	4.0000	4.0000	4.0000
Holdup Time Det 3 (min)	0.000	0.001	0.003	0.005	0.019	0.084	1.724
Makeup Flow (mL/min)	8						

Figure 2. Effluent Splitter Calculator

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.
- **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 1 and 15 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 1. Restrictors that have less flow will still work, but peak broadening and/or tailing may result.

Table 1 Suggested minimum restrictor flows

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

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

Column outlet pressure

The GC needs to know the pressure at the end of the column to be able to calculate column flows. Use either the GC keyboard or the ChemStation to set the outlet pressure for the column to 3.8 psig. The ChemStation screen where the column outlet pressure is set is shown in Figure 3.

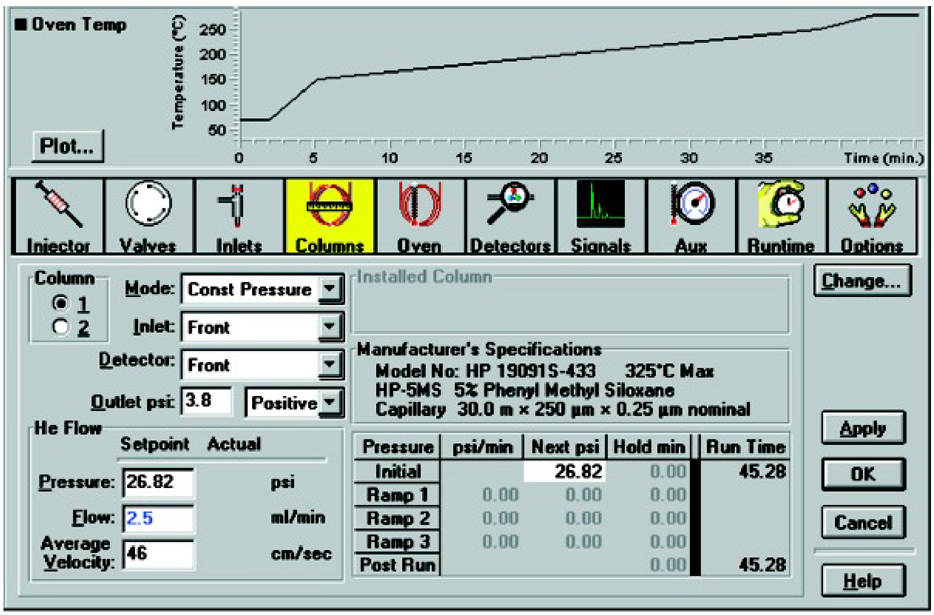


Figure 3 Column outlet pressure screen

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.