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Agilent G2855B Deans Switching System

Installation and Operation Guide

Agilent Technologies

Notices

Manual Part Number

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Introduction

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The Deans Switching System kit enables you to perform 'heart' cutting of samples from one column to another. A secondary column is used to separate components that are unresolved on the primary column.

How It Works

WARNING **Hydrogen gas is flammable and potentially explosive when accumulated inside the oven. For this reason, Hydrogen should not be used with this product due to possibility of tube breakage.**

Figure 1 shows the basic layout.

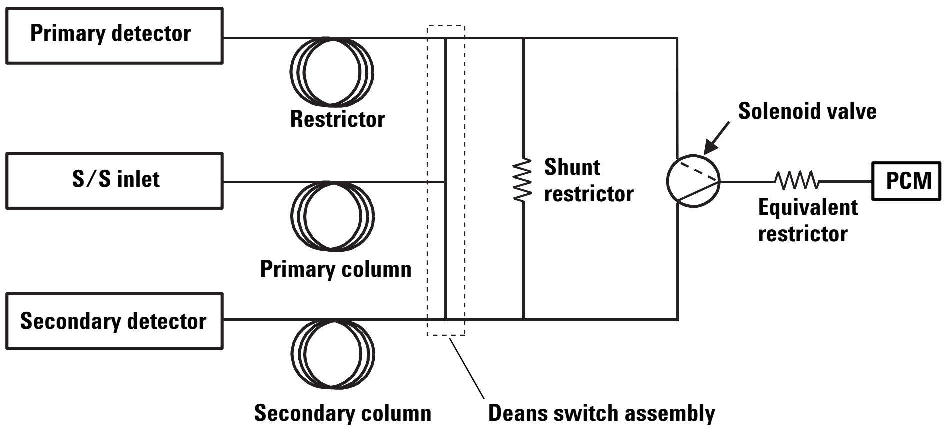

Figure 1 Gas flows

The sample is injected onto the **Primary** column. The effluent from this column normally goes to the **Primary Detector** via the **Restrictor**, a length of deactivated tubing.

When the **solenoid valve** is actuated, gas pressure from the Pneumatics Control Module (**PCM**) switches to the other side of the Deans switch assembly, forcing the effluent of the primary column to go to the **Secondary** column. This column has different separation properties, so that compounds that do not separate on the primary column may separate on the secondary column.

Very short switch times can be used because of the low internal volume of the Deans switch assembly.

GC Requirements

The system uses an Agilent 7890A Series gas chromatograph with:

- Two detectors
- Split/Splitless capillary inlet
- Pneumatics control module (PCM)

The top of the oven must be clear, except for inlets and detectors.

The PCM is the preferred switching gas source, as it automatically compensates for changes in atmospheric pressure. Even small changes in the weather can affect the retention times and hence the cut times on the system.

If you are using an Agilent 6890 Series gas chromatograph the above requirements apply. You will also need the Deans Switch 6890 additional parts kit listed on page 52.

Other Requirements

You will need the materials listed in Table 1.

Table 1 Tools and materials

Torx T-20 screwdriver
Precision tubing cutter, p/n 5190-1442
Side (diagonal) cutter, heavy duty
Two ¼-inch wrenches
The two capillary columns you will be using
Column nuts and ferrules to connect the columns to the inlets and detectors
Capillary column cutter
Paper clip or thin wire
Ruler with metric markings
Restrictor for 7890 GC, p/n G3430-80061
Restrictor for 6890 GC, p/n 19231-60610

Software is provided to calculate the length of the **Restrictor** tubing that you will be installing. It requires a personal computer with a CD-ROM drive.

Parts Supplied

The parts in the Deans switch accessory kit are listed in Table 2. The parts in the Deans switch switching valve kit are listed in Table 3. The parts in the Deans switch supplies and spares kit are listed in Table 4 on page 10.

Table 2 G2855-64010 Deans switch accessory kit

Description	Quantity
Oven wall bracket for CFT plate devices	1
Capillary column spring clip	4
Compact Deans switch assembly, inert	1
Deans switch switching valve kit (Table 3)	1
Deans switch supplies and spares kit (Table 4)	1
Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVDs	1

Table 3 G2855-60100 Deans switch switching valve kit

Description	Quantity
Screw, skt-hd-cap, M3 x 0.5 x 10 mm, lg	2
Valve bracket	1
Valve driver cable	1
Valve jumper cable	1
3-way valve assembly with glued fittings	1
Ferrule, Vespel 1/16-inch, 10/pk	1
Cross-over assembly 0.010-inch id x 15 cm (G2855-80580) • 1/16-inch od x 0.040-inch id x 5 cm tube, 316 SST • 1/16-inch od x 0.040-inch id x 10 cm tube, 316 SST • Modified cap • Tee, low mass braze, 1/16-inch • Tube, precise 0.010-inch id x 1/16-inch od x 15 cm	1 3 1 2 2 1

Table 4 G2855-60150 Deans switch supplies and spares kit

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

Parts Identification

Most of the parts in the kit are easily recognized. However, several of them are unique to this kit. They are identified in Figure 2.

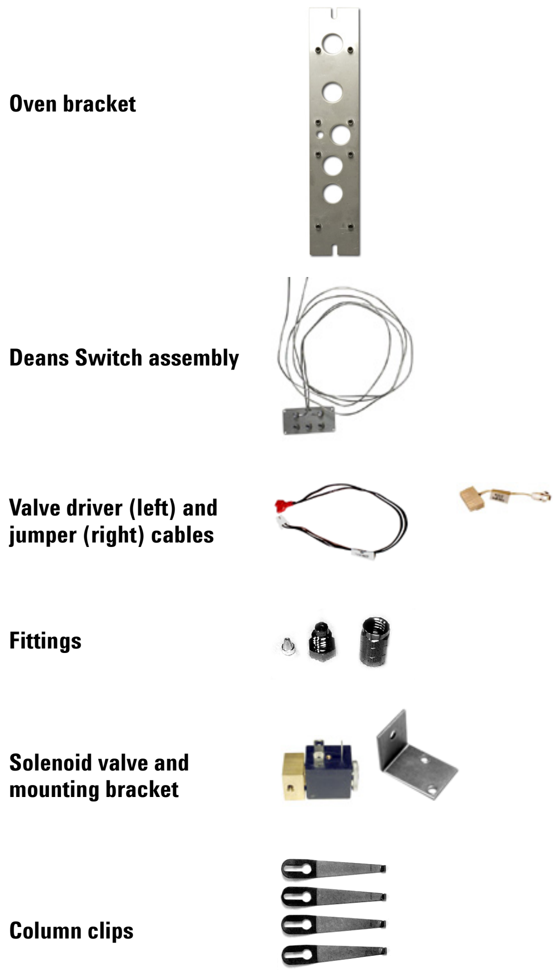

Figure 2 Unique parts

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This chapter contains step-by-step instructions for installing a Deans Switching System on an Agilent 7890A Series GC. For information on installing a Deans Switching System on an Agilent 6890 Series GC, see "6890 Series GC Supplemental Guide" on page 51.

Safety

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

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

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

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

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

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

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

CAUTION It is important to wear safety glasses at all times.

Installing the Solenoid Valve

If you are using a 6890 Series GC see "Installing the Solenoid Valve in a 6890 GC" on page 53.

1 Mount the solenoid valve on the valve bracket with two screws.

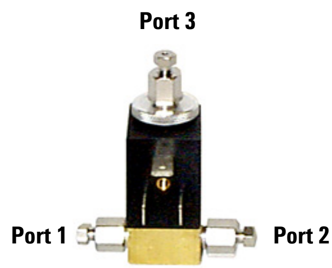

Figure 3 Solenoid valve with adapters

2 Mount the valve and bracket on the oven top as shown in Figure 4. Note that this secures the back of the panel.

3 Connect the valve driver cable to the valve (Figure 4).

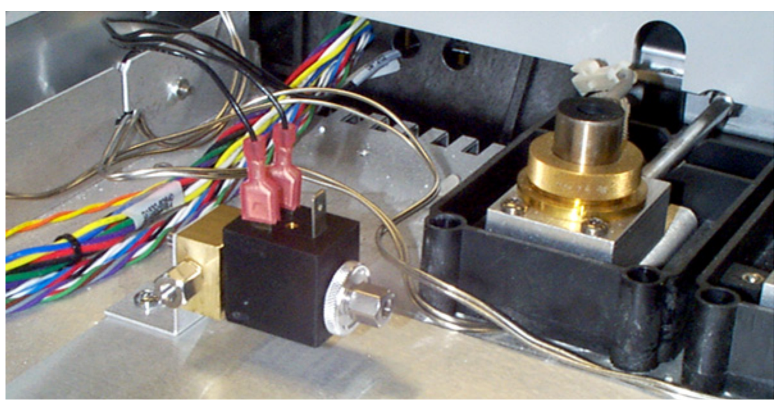

Figure 4 Mount valve and connect driver cable

4 Remove the right side panel. The main board will resemble either Figure 5 (no valve bracket) or Figure 6 (valve bracket present), except that the valve cables will not be present.

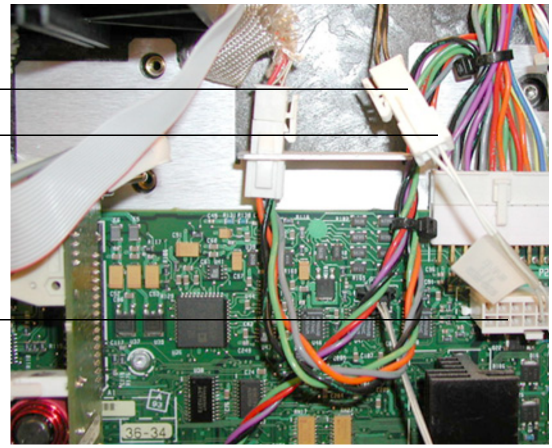

Figure 5 Main board without valve bracket

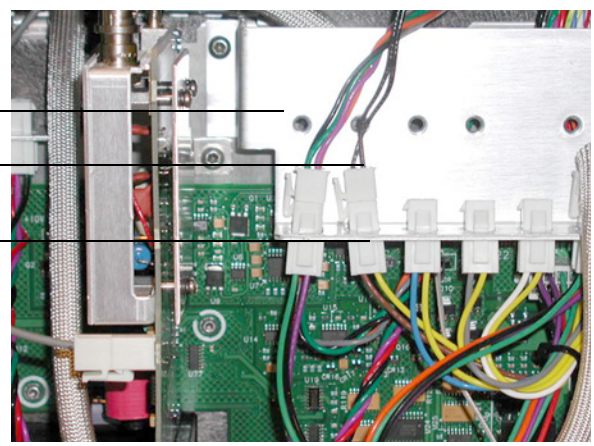

Figure 6 Main board with valve bracket

5 Is a valve bracket installed on the main board?

- **No** (Figure 5 on page 16): Connect the free end of the valve driver cable to the valve jumper cable. Connect the other end of the valve jumper cable to connector P22 at the top of the main board.
- **Yes** (Figure 6 on page 16): Connect the free end of the valve driver cable to socket V1 on the valve bracket.

NOTE You can use any of sockets V1 through V4 to control the valve, but must configure the selected valve as a *switching valve* and must refer to it when entering the cut time commands. This discussion assumes V1.

6 Connect the switching gas supply.

- **To supply the switching gas from a PCM**: Connect one of the 1-m coiled lengths of tubing to port **2** of the solenoid valve (Figure 7). Connect the stainless steel union to the other end.

  Cut the line from the PCM so that is about 15 cm (6 inches) long. Connect it to the stainless steel union.

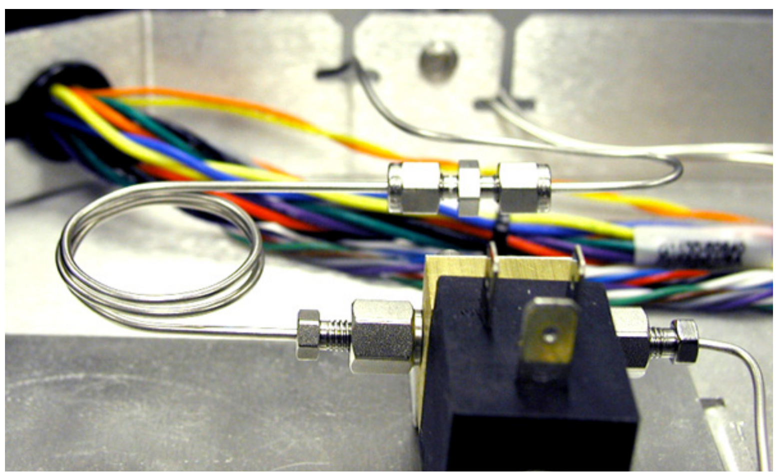

Figure 7 Pneumatics control module connection

- **To supply the switching gas from an Auxiliary Pressure controller** (Figure 8): Ensure that the restrictor with one brown ring (p/n G3430-80061, 6890 p/n 19231-60610) is installed in the Aux 3 channel outlet fitting.

  Remove the 1/8-inch end of the tubing using cutters or a file. Connect the tube to port **2** of the solenoid valve.

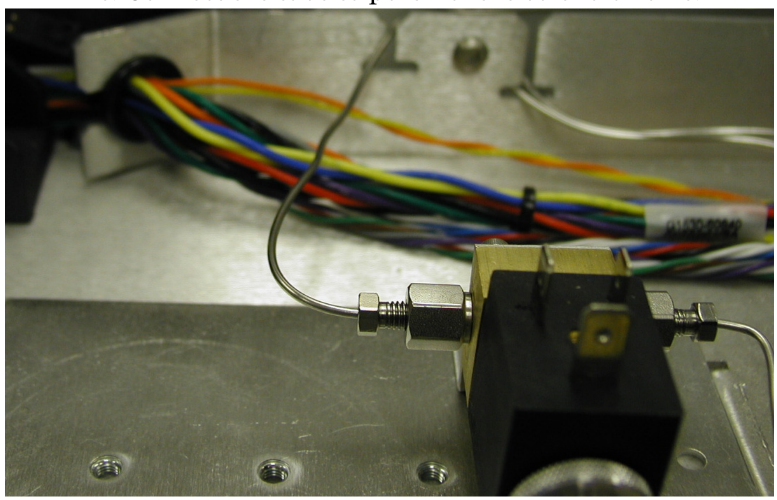

Figure 8 Auxiliary pressure controller connection

Installing the Column Clips

Install the four column clips on the oven shroud (Figure 9

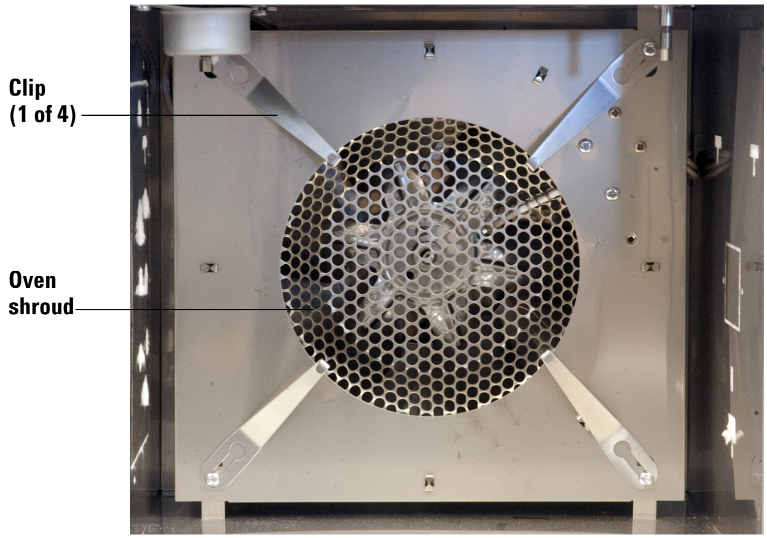

Figure 9 Column clips

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

Installing the Oven Bracket

Choose which side of the oven will be most convenient for most users in your laboratory. Usually the Deans switch assembly is installed on the left side of the oven. Consider ease of access to detectors, inlets, and accessories. Also, consider the ergonomics of the user holding the 1/4-inch wrench used on the fitting nuts.

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

2 Use the T-shaped thumbscrew to fasten the bracket to the T-slot in the oven wall (Figure 10). Pull the thumb screw sleeve back to facilitate slipping it into the slot on the oven wall.

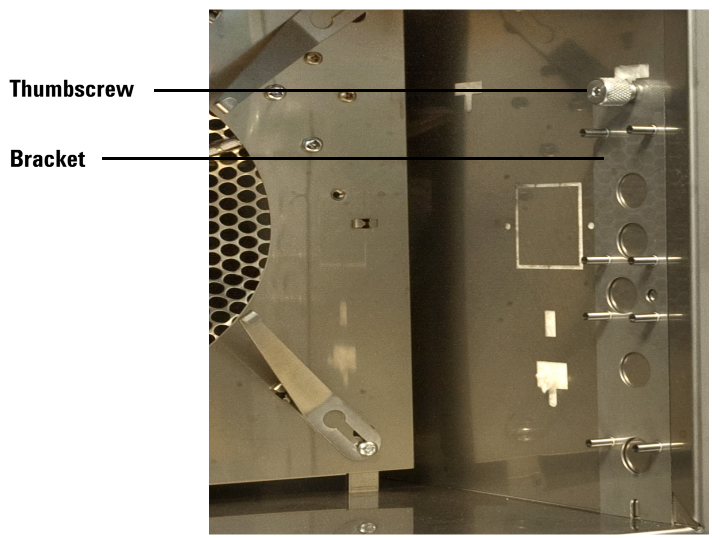

Figure 10 Installing the oven bracket

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

Installing the Deans Switch Assembly

Before fastening the Deans switch assembly to the oven bracket, prebend the tubes to cleanly fit against the oven walls (Figure 11). This will make installation easier and will also keep the inside of the oven clutter-free for ease of future maintenance.

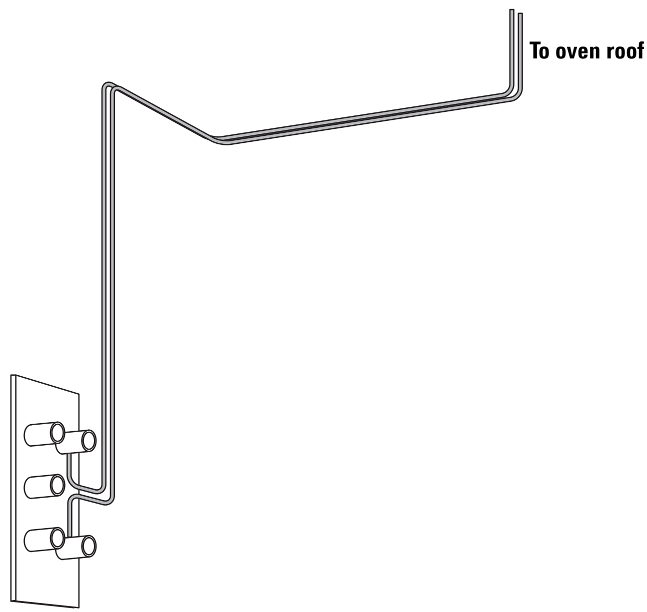

Figure 11 Preshape tubes to fit oven walls for easy installation

NOTE Bend the tubes over an object such as your thumb to avoid kinking.

1 To avoid damage during installation, cap the ends of the tubes.

2 Run the tubes through the oven ceiling. There will be an excess amount of tubing through the roof of the oven. The tubes can be trimmed, but we recommend that you leave enough slack for any future uses. Coil the excess to avoid kinking.

3 Secure the Deans switch assembly to the oven bracket with four screws (Figure 12). Ensure the ejector holes on the fittings are facing outward. For installations on the right-hand side of the oven the Deans switch assembly must be mounted upside-down (compared to a left-hand installation) in order to ensure that the ejector holes face outward.

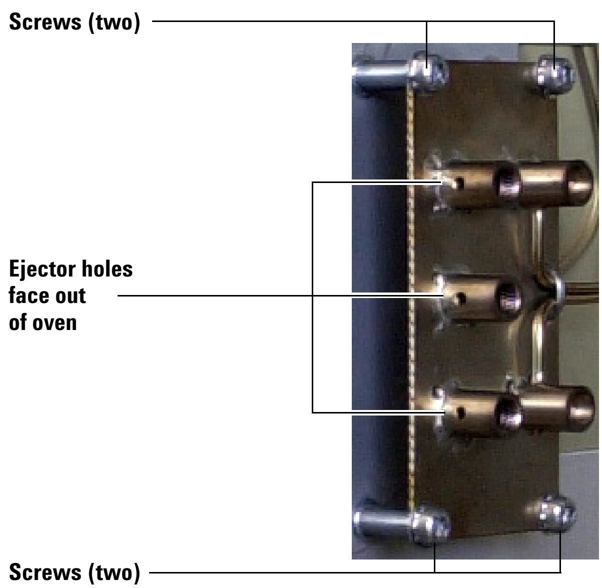

Figure 12 Attaching the Deans switch assembly

4 For installations on the left side of the oven, locate the tubing that is connected to the bottom side of the Deans switch assembly. For installations on the right side of the oven, locate the tubing that is connected to the top side of the Deans switch assembly. Connect the free end of the tubing to one side of a 1/16-inch brass tee. Connect a 5-cm piece of tubing to the opposite side of the tee. Connect the free end of the tubing to port **3** of the valve as shown in Figure 13.

5 For installations on the left side of the oven, locate the tubing that is connected to the top side of the Deans switch assembly. For installations on the right side of the oven, locate the tubing that is connected to the bottom side of the Deans switch assembly. Connect the free end of the tubing to one side of a 1/16-inch brass tee. Connect a 10-cm length of tubing to the opposite side of the tee. Connect the free end of the tubing to port **1** of the valve as shown in Figure 13.

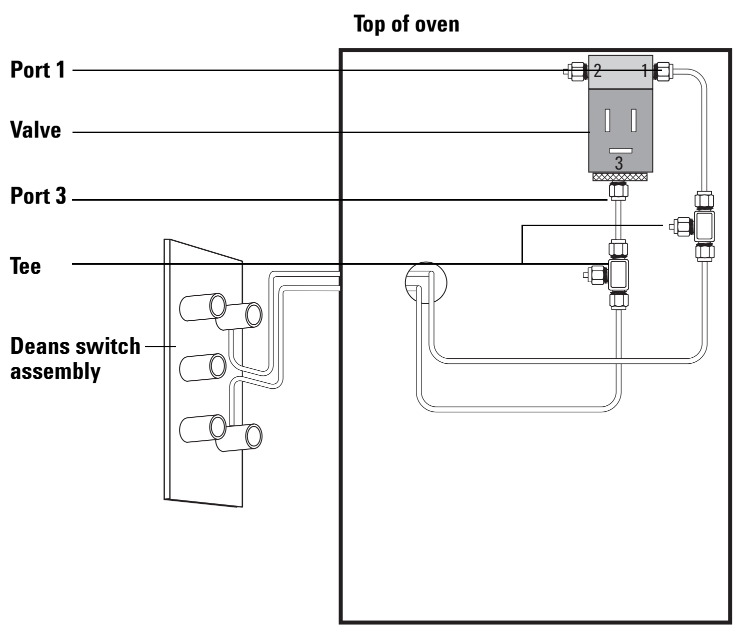

Figure 13 Connect tubing (shown for left hand oven mounting)

Installing the Columns

1 Clip the baskets of the two capillary columns together using the hooks on the column baskets.

2 Install the column assembly onto the capillary column clips (Figure 9). Center the assembly in the oven. Adjust and tighten the clips so that the column assembly is tightly held to the oven shroud.

We will be using the terms **primary** column and **secondary** column. *The primary column is the column into which the sample will be injected.*

3 Connect one end of the **primary** column to the capillary inlet. See the appropriate sections of your GC user documentation for correct installation of a capillary column to the inlet.

4 Preswage an internal nut and a SilTite ferrule to the exit end of the column. See “Swaging SilTite Ferrules” on the Instrument Utilities disk for detailed instructions.

5 Connect the exit end of the **primary** column to the middle fitting in the Deans switch assembly (Figure 14).

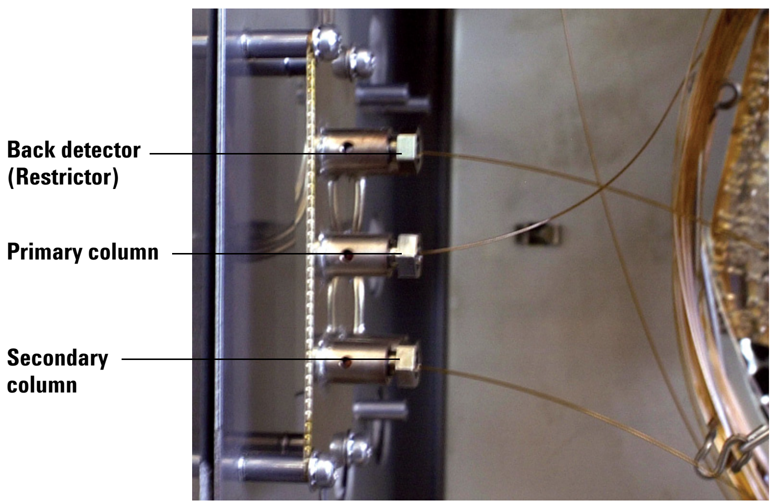

Figure 14 Completed Deans switching assembly

6 Finger-tighten the nut, then further tighten the fitting by turning the wrench only 15 to 20 degrees (Figure 15).

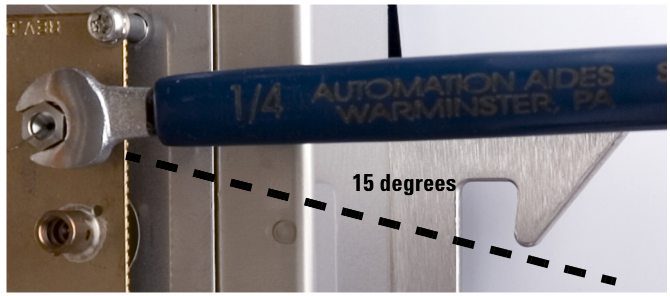

Figure 15 Tighten the fitting

7 Connect an end of the **secondary** column on the bottom fitting on the Deans switch assembly. Follow the instructions in step 6.

8 Connect the other end of the **secondary** column to the secondary detector (Figure 16).

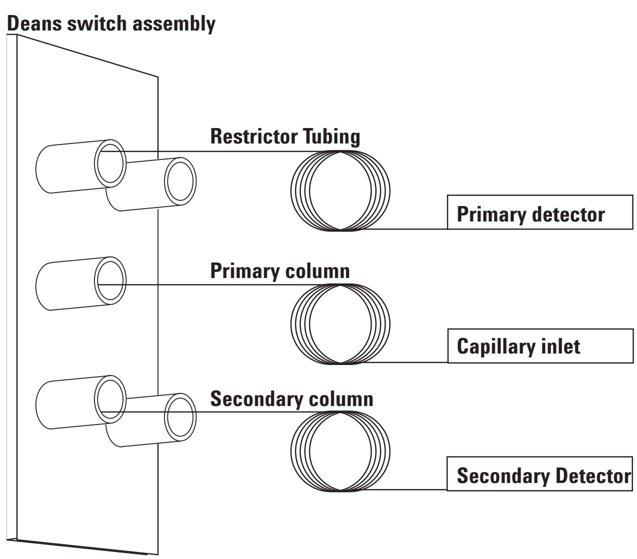

Figure 16 Connect columns

Installing the Restrictor Tubing

The next hardware installation step is to connect the **Restrictor** tubing from the Deans switch assembly to the primary detector. This tube must have the same pneumatic resistance as the secondary column, but the transit (holdup) time must be much shorter. This is done using a short length of narrow bore uncoated deactivated fused silica tubing.

For a given tubing diameter, the length is calculated based on the dimensions of the secondary column and the operating pressures of the two detectors. If you are developing a new method, decide what your method conditions will be before installing the restrictor.

First calculate the restrictor dimensions needed, then create and install the restrictor tubing.

Calculating the Restrictor Dimensions

Choose the smallest diameter that gives a length of at least 0.3 m for conventional detectors or 0.5 m for Mass Selective Detectors (MSDs). For most applications using 0.25-mm id columns and conventional detectors, 0.1-mm id tubing (supplied with the kit) is used. Systems with larger diameter columns or with MSDs may require larger diameter tubes (0.18-, 0.2-, or 0.25-mm id).

The Deans switch calculator software takes the desired method parameters (column dimensions, temperature, flows, etc.) and calculates the required restrictor length and pressure setpoints to operate the system. Before the calculator can be used, the method parameters must be chosen. As an example, we will use the method parameters shown in Table 5 on page 27.

Table 5 Example method parameters

	Primary column	Secondary column
Type	HP1-MS	INNOWax
Diameter (mm)	0.25	0.25
Length (m)	15	15
Film (µm)	0.25	0.25
Detector	FID	FID
Column flow (mL/min)	2	3
Initial oven temperature	40 °C	
Carrier gas	Helium	

Insert disk 1 of the Agilent Technologies GC and GC/MS Hardware User Information and Instrument Utilities Software DVD into the drive. If the disk does not autostart, select **Run** from the Start menu and type **X:\start.htm**, where **X** is the letter assigned to the CD drive.

1 Choose the topic **G2855B Capillary Flow Technology Deans Switching System** from the menu and select **Install Deans Switch Flow Calculator**. Follow the installation instructions.

2 Click **Start**, then select **Programs** and the **Deans Switch Calculator**. The calculator screen opens (Figure 17 on page 28).

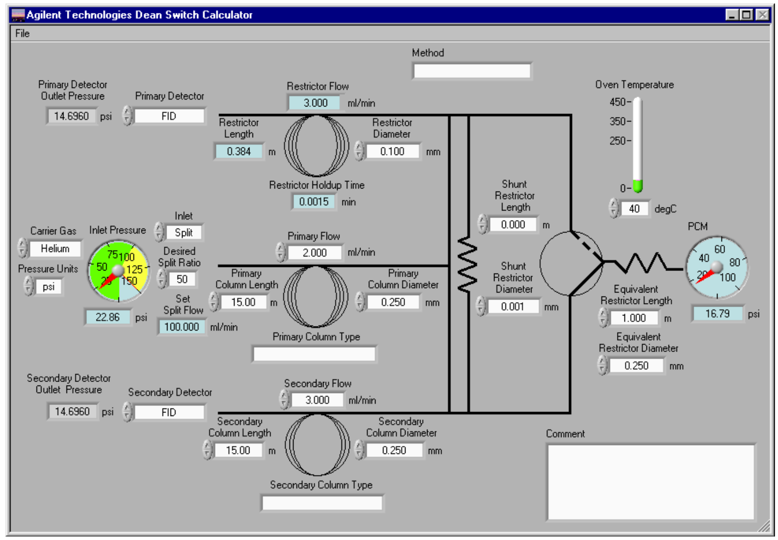

Figure 17 Calculating the **Restrictor** length

For this example, fill in the parameter shown in brackets [ ].

3 Define the **Carrier Gas**. [`Helium`]

4 Enter the **Pressure Units** you will use. [`psi`]

5 Enter the starting temperature (**degC**) of the analysis. [`40`]

6 Enter the **Primary Detector** type. If this is a non-Agilent detector, choose **Other** and set the **Primary Detector Outlet Pressure**. This *must* be in *absolute* units. [`FID`]

7 Enter the **Secondary Detector** type. If this is a non-Agilent detector, choose **Other** and set the **Secondary Detector Outlet Pressure**. This *must* be in *absolute* units. [`FID`]

8 Enter the **Primary Column Length** in meters. [`15`]

9 Enter the **Primary Column Diameter** (id) in mm. [`0.25`]

10 Enter the **Primary Flow** in mL/min. This should be chosen for maximum column efficiency. [`2.0`]

11 Enter the **Secondary Column Length** in meters. [`15`]

12 Enter the **Secondary Column Diameter** (id) in mm. [`0.25`]

13 Enter the **Secondary Flow** in mL/min. This should be about 50 percent greater than the **Primary Flow**. The difference between the primary and secondary flows must be at least 1 mL/min.

If the resulting flow is too high for the secondary detector (such as a mass spectrometer), set the secondary flow to a value that the detector can handle and then adjust the primary flow to keep the ratio of the flows correct (Secondary flow = Primary flow + 50%). [`3.0`]

14 Enter the **Restrictor Diameter** (id) in mm. The tubing supplied has an id of 0.1 mm. [`0.1`]

15 Read the **Restrictor Length** in meters. In this example, 0.384 m of 0.1-mm id restrictor tubing is required.

If a diameter larger than 0.1-mm id is required, these are the Agilent part numbers:

- 0.18 mm id × 10 m, 160-2615-10
- 0.20 mm id × 10 m, 160-2205-10
- 0.25 mm id × 10 m, 160-2255-10

Creating and Installing Restrictor Tubing

1 Cut the length of tubing (calculated in the section above) from the 5-meter length of restrictor tubing supplied in kit G2855-60150 (see Table 4 on page 10).

2 Preswage an internal nut and a SilTite ferrule to one end of the restrictor. See “Swaging SilTite Ferrules” on the Instrument Utilities disk for detailed instructions.

3 Connect the swaged end of this restrictor tube to the top inlet of the Deans switch assembly (see Figure 14 on page 24).

4 Connect the other end of the restrictor tube to the primary detector using the appropriate nut and ferrule.

NOTE

If the secondary column is ever changed in length or internal diameter, or if a detector of different operating pressure is used, you will need to recalculate the dimensions of the restrictor tube.

Removing Fittings

Disconnect tubing from the Deans switch assembly

Loosen and remove the nut from the Deans switch assembly 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 ejector hole in the side of the fitting (Figure 18). Press firmly. The ferrule will click when it breaks free.

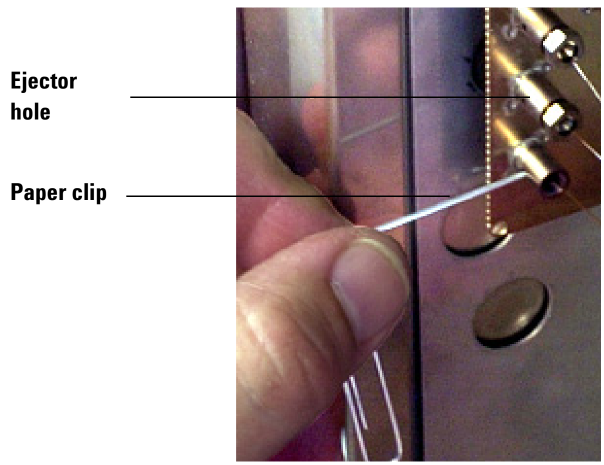

Figure 18 Releasing a ferrule

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, cap the end of the tubes. Tighten to finger-tight plus 15 degrees.

Protect the Deans switch assembly

Seal the ports of the Deans switch assembly with plugs when it 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.

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This chapter discusses selecting the method parameters for a Deans switch method and using the calculator software to set up the analysis. An example method will be developed for the analysis of methyl tertiary-butyl ether (MTBE), benzene, and toluene in gasoline.

Column Considerations

The primary and secondary column phases are chosen to have different selectivity. The analyte(s) of interest are separated on the first column from at least the bulk of the sample matrix. The analyte(s) and any co-eluting matrix interferences are then cut onto the second column. Because the phase of the second column has a different selectivity, the analyte(s) are (hopefully) separated from the matrix interferences.

Some phase combinations that have been used effectively are:

- DB-1 (non-polar) with HP-INNOWax (polar). This is a good choice for general analytical separations. It is limited by the upper temperature limit of INNOWax (260 °C).
- DB-5 (low polarity) with DB-17MS (medium polarity). This is a good choice for separations that require higher temperatures (up to 320 °C). It works well for cutting organophosphorus pesticides from difficult matrices like essential oils.
- DB-5 (low polarity) with Cyclosil (chiral). This pair is useful for resolving enantiomers that are overlapped with matrix interferences in, for example, flavors and fragrances.
- HP-INNOWax (polar) with HP-Plot Q. This combination works well for cutting trace thiophene from benzene.

Which column should be the primary column? Two useful rules are:

- Choose the phase which separates the analytes from the largest matrix interferences.
- Choose the more robust phase, since the primary column receives the bulk of the sample loading.

For the example gasoline method, the DB-1MS will be the primary column. This is a nonpolar, rugged, low-bleed phase that spreads out the components of gasoline approximately in boiling point order.

Since most of the matrix interferences will be hydrocarbons and the three analytes are polar, HP-INNOWax will be used as the secondary column. The HP-INNOWax will retain the polar analytes relative to the coeluting hydrocarbons.

Column dimensions are selected with the same considerations used in any gas chromatographic method, where tradeoffs between speed, resolution and capacity are made.

In the gasoline method, the analytes are present in percent concentrations. The method should have just enough resolution to separate the three analytes and do so in the fastest way possible. A good starting point is to select columns with a relatively small diameter, thin film, and short length. Since the Deans switch has a small but finite dead volume, it is suggested that columns of diameter smaller than 0.2-mm id be avoided.

The primary column will be a 15-m × 0.25-mm id × 0.25-µm film thickness DB-1MS and the secondary column will be a 15-m × 0.25-mm id × 0.25-µm HP-INNOWax.

Inlet Considerations

There are no inherent limitations on the inlets that can be used for Deans switch analyses. Choose the inlet type using the same considerations as with conventional GC methods.

In the gasoline example, the analytes are at high concentrations. To prevent peak overloading, we will use a split injection. The split ratio must be high because of the chosen column dimensions, we will use 150:1. A small (0.2 µL) injection will reduce column loading and save carrier gas.

Detector Considerations

The detectors are selected based on the needs of detection limit, dynamic range, selectivity, ease of use, etc. As long as the detector can accept the flow rate of carrier gas from the column or restrictor to which it is connected, it can be used. Since Deans switch methods are run in constant pressure mode, the detector must also be compatible with column flows changing during temperature programmed runs. If MSDs are used, the column sizes and carrier flow rates must be chosen to match the pumping capacity of the system.

The flame ionization detector (FID) is a good starting point. Due to the high resolving power of 2-D GC methods, analytes are often separated completely. If so, the FID is the easiest and most cost effective detector. If the matrix is very complex, selective detectors can be used to measure the analytes.

FIDs will be used for the gasoline example method.

Column Flow Rates

In a Deans switch method, the flow from the primary column plus a switching flow go through the secondary column. The primary column flow is usually chosen to be at or near the optimum for that column. This prevents the flow through the secondary column from exceeding the optimum to such an extent that there is a significant loss in resolution.

Table 6 gives suggested flows for different column diameters and carrier gases. Two flows are listed for each combination. **Best efficiency** gives approximately the optimum resolution. **Fast analysis** yields a faster chromatogram with only a small reduction in chromatographic resolution.

Table 6 Suggested column flows (mL/min)

Column	Helium		Hydrogen		Nitrogen		Argon	
diameter (mm)	Best efficiency	Fast analysis	Best efficiency	Fast analysis	Best efficiency	Fast analysis	Best efficiency	Fast analysis
0.10	0.40	0.80	0.50	1.00	0.13	0.25	0.11	0.22
0.18	0.72	1.44	0.90	1.80	0.23	0.45	0.20	0.40
0.20	0.80	1.60	1.00	2.00	0.25	0.50	0.22	0.44
0.25	1.00	2.00	1.25	2.50	0.31	0.63	0.28	0.55
0.32	1.28	2.56	1.60	3.20	.040	0.80	0.35	0.70
0.45	1.80	3.60	2.25	4.50	0.56	1.13	.050	0.99
0.53	2.12	4.24	2.65	5.30	0.66	1.33	0.58	1.17

For the example gasoline method, the **Fast analysis** flow will be chosen. The primary column flow will be set to 2.0 mL/min of helium (at the initial oven temperature).

The flow for the secondary column is determined by taking the flow from the primary column and increasing it by about 50%, with a minimum increase of 1.0 mL/min. The difference between the two flows reflects the switching flow. It must be large enough to prevent tailing in the switch.

In the gasoline example, the primary column flow is 2.0 mL/min. Increasing this by 50% gives 3.0 mL/min, which meets the 1.0 mL/min difference requirement. The flow for the HP-INNOWax column will be set to 3.0 mL/min.

Deans switch methods are run in constant pressure mode. Since the Deans switch must be operated at constant pressure, by definition the secondary column will be in constant pressure mode.

A second consequence of the firmware or software using the incorrect outlet pressure for the primary column is that for split injections, the split ratio calculation will also be incorrect. If a split injection is being used, the split flow must be calculated separately to give the desired split ratio. The Deans switch software calculates the correct split flow to enter to obtain the desired split ratio.

Oven Temperature Program

The oven temperature program is chosen in the normal way. For the gasoline example method, the oven will be programmed at 40 °C for 1 minute, 10 °C/min to 250 °C, and held for 5 minutes.

The low initial temperature is needed to separate the MTBE and the high final temperature is required to elute all of the highest boiling gasoline components. To determine the temperature program rate, a useful guideline is to program at approximately 10 °C/holdup time of the column. For the primary column, the holdup time is close to 1 minute, thus it will be programmed at 10 °C/min.

Note that the holdup time for the primary column cannot be calculated until after the Deans Switch Calculator is used to determine all the pressure setpoints, as shown in the next section.

The holdup times for each column are printed when the calculator results are printed.

Pressure Setpoints for Primary and Secondary Columns

It it now time to calculate the pressure setpoints for the primary column inlet and for the PCM (or Aux EPC) modules to obtain the desired flows.

Run the calculator program (Figure 19).

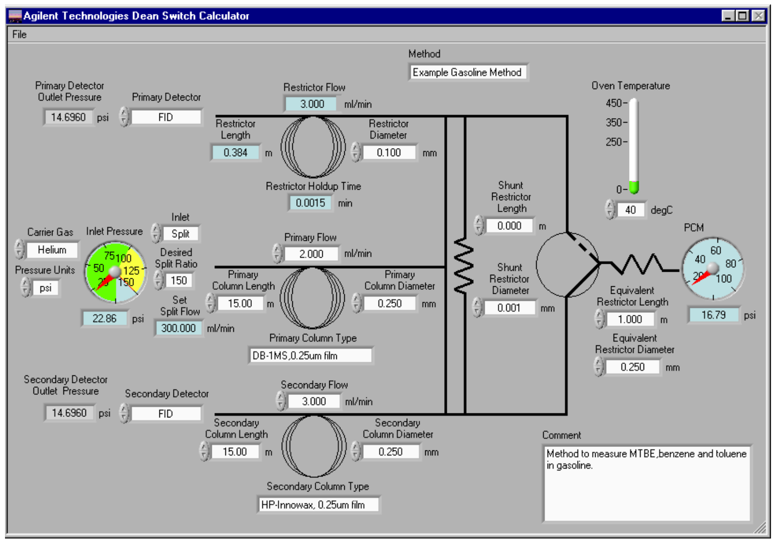

Figure 19 Calculating pressures

Enter the parameters for the example gasoline method, shown in square brackets.

1 Define the **Carrier Gas**. [He]

2 Enter the **Pressure Units** that you will use. [psi]

3 Enter the starting temperature (**degC**) of the analysis. [40]

4 Enter the **Primary Detector Type**. If this is a non-Agilent detector, choose **Other** and set the **Primary Detector Outlet Pressure**. This *must* be in *absolute* units. [FID]

5 Enter the **Secondary Detector Type**. If this is a non-Agilent detector, choose **Other** and set the **Secondary Detector Outlet Pressure**. This *must* be in *absolute* units. [FID]

6 Enter the **Primary Column Length** in meters. [15]

7 Enter the **Primary Column Diameter** (id) in mm. [0.25]

8 Enter the **Primary Flow** in mL/min. This should be chosen for maximum column efficiency. [2.0]

9 Enter the **Secondary Column Length** in meters. [15]

10 Enter the **Secondary Column Diameter** (id) in mm. [0.25]

11 Enter the **Secondary Flow** in mL/min. This should be about 50% greater than the Primary flow, with at least 1 mL/min difference between the primary and secondary flows.

If the resulting flow is too high for the secondary detector (such as a mass spectrometer), set the secondary flow to a value that the detector can handle and then adjust the primary flow to keep the ratio of the flows correct (Secondary flow = Primary flow + 50%). [3.0]

12 Enter the **Restrictor Diameter** (id), in mm. The tubing supplied has an internal diameter of 0.1 mm. [0.1]

13 Enter the **Equivalent Restrictor Length** in meters. This restrictor is a length of tubing or a frit used to dampen pressure oscillations from the Aux EPC or PCM module. It will normally be a 1.0-m length of 0.25-mm id stainless steel tubing. If it is the FID air frit, enter 1.16 m for the length and 0.32-mm id for the diameter. The calculator will account for this restriction when calculating the pressures. [1.0]

14 Enter the **Equivalent Restrictor Diameter** in mm (see previous item). This will normally be 0.25 mm.[0.25]

15 Read the pressure (16.79 psi) for the switching gas under the PCM dial. Set the PCM or Aux EPC pressure to this value.

16 Read the pressure (22.86 psi) for the inlet under the **Inlet Pressure** dial. Set the inlet pressure to this value.

17 You can enter the dimensions of the Shunt restrictor into the boxes labeled **Shunt Restrictor Diameter** and **Shunt Restrictor Length**. These entries are optional, since they are not used in the calculations. They are provided for record keeping purposes.

18 If the **Split** item in the **Inlet** field is selected, two fields appear labelled **Desired Split Ratio** and **Set Split Flow**. As mentioned previously, the split ratio calculation in the GC will not be correct.

If the method calls for a split injection, enter the **Desired Split Ratio**. Read the calculated **Set Split Flow** value and enter it into the GC setpoints to obtain the **Desired Split Ratio**.

Enter `[150]` for the **Desired Split Ratio.** Read **300 mL/min** at the **Set Split Flow** window and enter it into the GC.

19 There is a field for optional **Comment**.

20 To save the calculations, type a name into the box labeled **Method** and select **Save method** from the **File** menu. Use **Load method** to reload the method at a later date. To print the current calculation results, select **Print current method** in the **File** menu.

21 Ensure that the valve you are using, usually Valve 1, on the GC is configured as a Switching Valve.

Determining the Switching Times

Prepare a calibration sample that contains the compounds that you wish to switch. The analytes should be at the maximum concentration that will be encountered in real samples. This is important with high concentration analytes, because if they overload on the primary column, the peak width will increase and the switching time range will need to be widened. If the analyte peaks are too overloaded, the split ratio or the amount injected should be adjusted to improve the peak shape.

For the example gasoline method, the calibration standard consists of 12% (v/v) ethanol, 20% MTBE, 6% benzene, and 20% toluene in iso-octane. Before running the calibration sample, you must know the order of elution from the primary column. For the example, the elution order is: ethanol, MTBE, benzene, iso-octane, and toluene.

Make sure that Valve 1 is turned OFF and that there are no entries in the time table for Valve 1 (so that no switching will take place).

Inject the calibration sample and start the run. All of the peaks will elute to the primary detector. The chromatogram is shown in Figure 20.

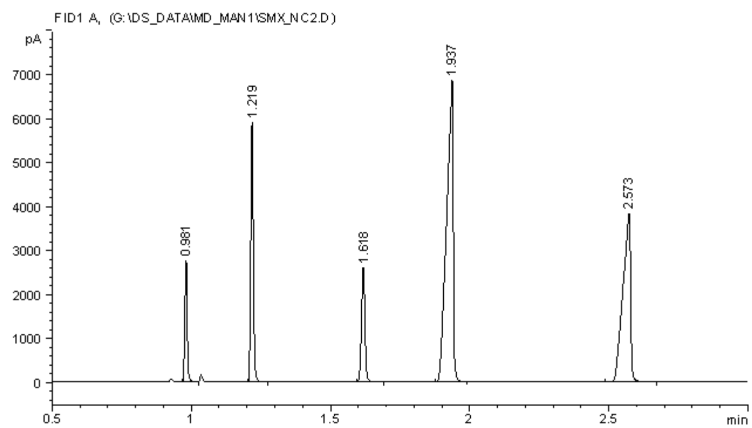

Figure 20 Calibration chromatogram

The retention times are: ethanol (0.981 min), MTBE (1.219 min), benzene (1.618 min), iso-octane (1.937 min), and toluene (2.573 min).

To set the switching times for MTBE, determine the times just before and just after the peak. Figure 21 shows these for MTBE.

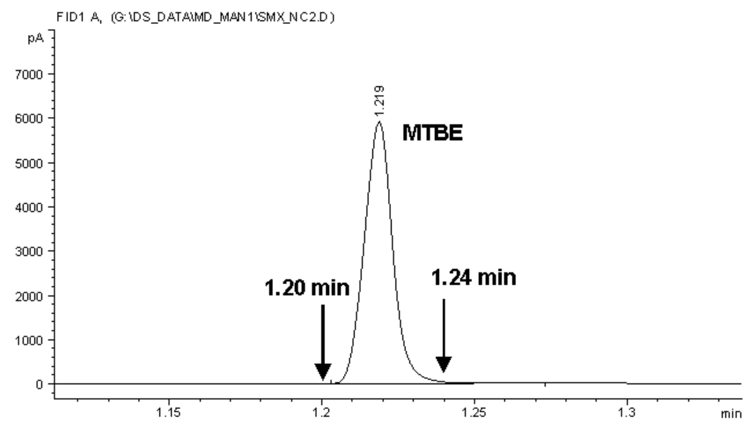

Figure 21 MTBE times

To switch the MTBE peak, insert entries into the GC time table to turn Valve 1, which controls the Deans switch, ON at 1.20 min and OFF at 1.24 min. This will cut the MTBE onto the INNOWax column.

Make a second injection, now switching the MTBE peak to the INNOWax column. Figure 22 shows the result.

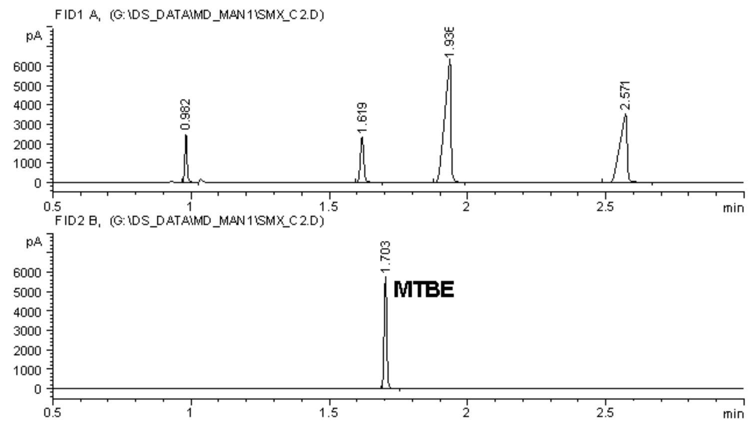

Figure 22 MTBE cut to INNOWax column

Note that the MTBE peak is now absent from the primary column chromatogram (top) and appears in the secondary column chromatogram (bottom).

Once the method is determined to work with the calibration standard, it should be checked with a sample that has the same matrix interferences as the real samples for which the method is intended.

Figure 23 shows the uncut chromatogram of RFA, a reference gasoline. Note that there is a serious overlap of an interference with the MTBE peak.

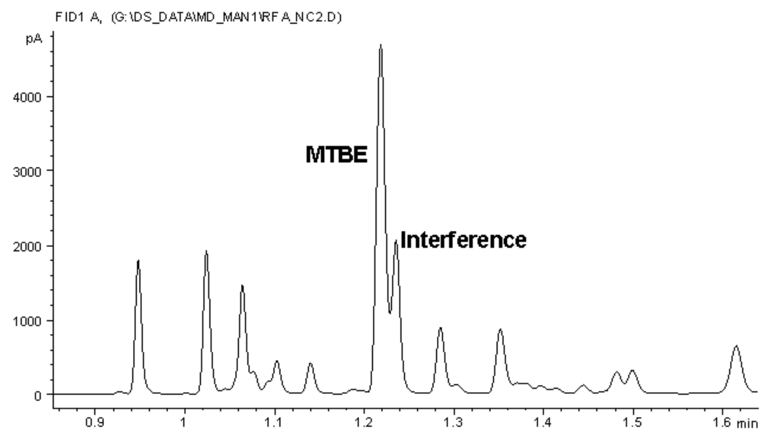

Figure 23 Uncut RFA chromatogram

Figure 24 shows the separation with the MTBE cut to the INNOWax column.

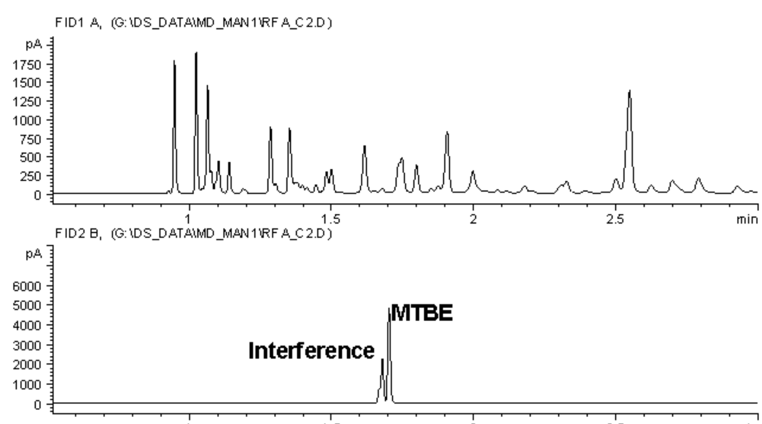

Figure 24 RFA with MTBE cut to INNOWax

The separation is much better with the 2-D method. There still remains some overlap. If this is unacceptable, using longer columns would improve the separation.

Cut times for benzene and toluene were selected using the same procedure as for MTBE. Benzene was cut from 1.60 to 1.65 minutes and toluene from 2.51 to 2.62 min.

Figure 25 shows the test mix run with MTBE, benzene, and toluene cut to the INNOWax column.

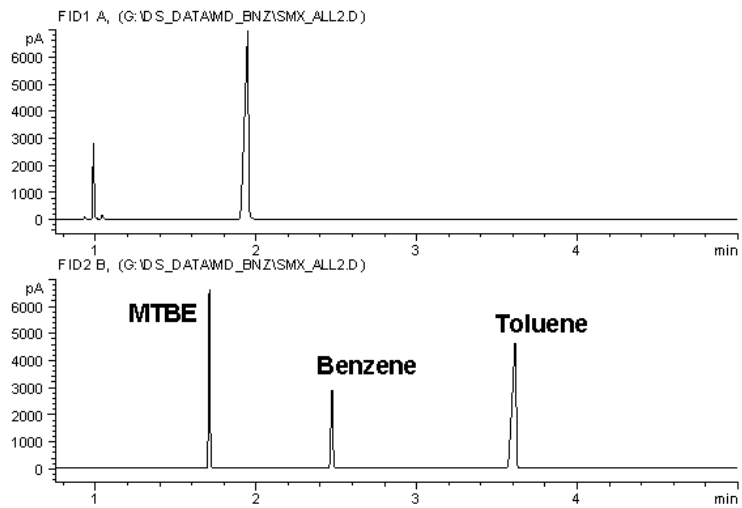

Figure 25 Multiple cuts

To check for matrix interference problems, a sample of local pump gasoline was run (Figure 26).

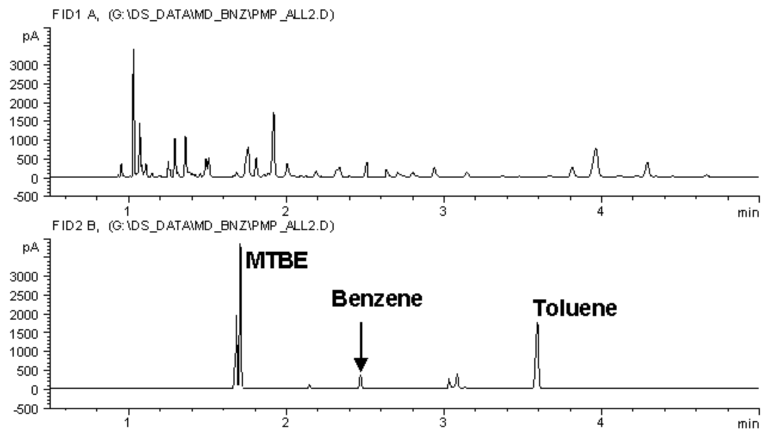

Figure 26 Local pump gasoline

In all three cases, the interfering compounds elute before the analyte. Benzene and toluene are completely separated from interferences.

Ensure that compounds from one cut do not interfere with those from another. If there is interference between two cuts, then run two methods on the sample, cutting only one compound per method. In the current example, there are no such interferences, so one method will suffice.

Backflushing to Save Time

If using a 6890 Series GC see "Backflushing to Save Time" on page 60.

For the example gasoline method, the three analytes are completely eluted from both columns by 4 minutes. However, the method must be run approximately 22 minutes to flush all of the gasoline components from the column (Figure 27).

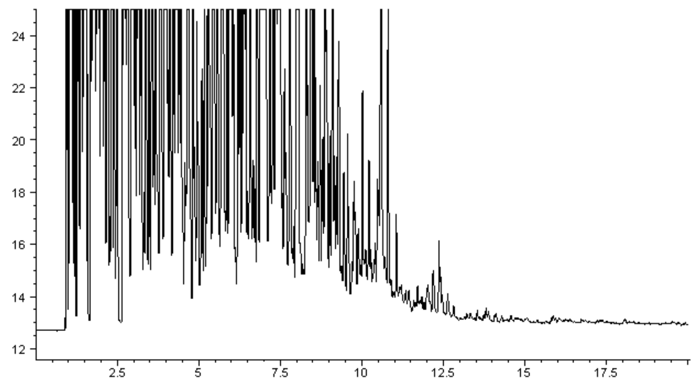

Figure 27 Eluting gasoline

To shorten the analysis time, the temperature or pressure could be ramped after 4 minutes to push the remaining gasoline components out of the column more rapidly.

If a split/splitless inlet is used, backflushing is another alternative with the Deans switching system.

In the example gasoline method, the oven is programmed at 40 °C for 1 minute, ramped at 10 °C/min to 250 °C, and held for 5 minutes. Since all the analyte peaks are out by 4 minutes, which corresponds to an oven temperature of 70 °C, the column controls can be set to backflush beginning at 4 minutes.

NOTE If using a 7890A GC, you can also use the backflush wizard available in the ChemStation to perform backflush as a post-run program.

4
6890 Series GC Supplemental Guide

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Installing the Solenoid Valve in a 6890 GC 53
Selecting and Installing the Shunt Restrictor 59
Backflushing to Save Time 60

This chapter describes the procedures for installation and operation of the Deans Switch hardware on a 6890 Series GC.

Parts Required

Table 7 G2855-60102 Deans Switch - additional parts for 6890 GCs

Description	Quantity
6890/5890 Valve box blank plate	1
Restrictor, FID AIR (brown)	1
Union, SS 1/16-inch tubing	1
O-ring, 2-010, fluoroelastomer, 5/pk	1
Screw, T-20, M4 x 8 mm	2
Tube, 1/16-inch od x 0.010 id x 1 m tube, 316 SST	1

Installing the Solenoid Valve in a 6890 GC

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

CAUTION It is important to wear safety glasses at all times.

NOTE Follow our recommendations as closely as possible for ease of use and maintenance.

1 Raise the GC top cover to expose the oven top.
2 Remove the valve box cutout using a side cutter (Figure 28).

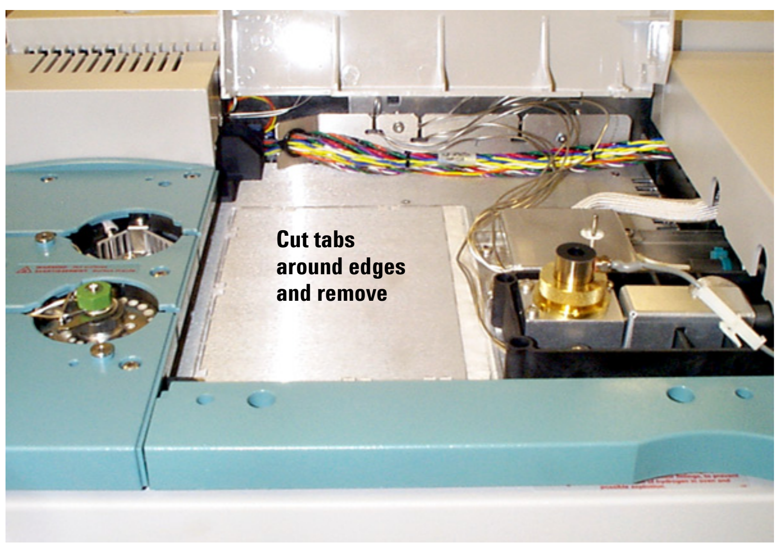

Figure 28 Remove the valve box cutout (6890N GC shown)

3 This exposes a layer of soft insulation. Remove it to expose the hard oven insulation. Remove the precut insulation piece at the location shown in Figure 29.

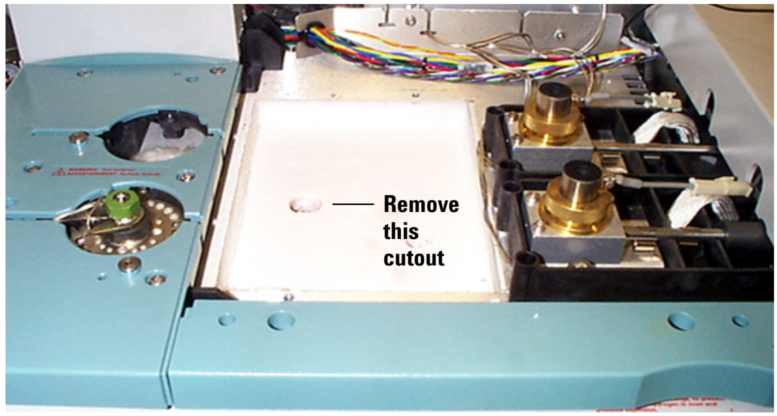

Figure 29 Remove the insulation cutout (6890N GC shown)

4 Replace the soft insulation. Install the valve box blanking plate, using one screw in the front position to secure it. The back end will be secured later. See Figure 30.

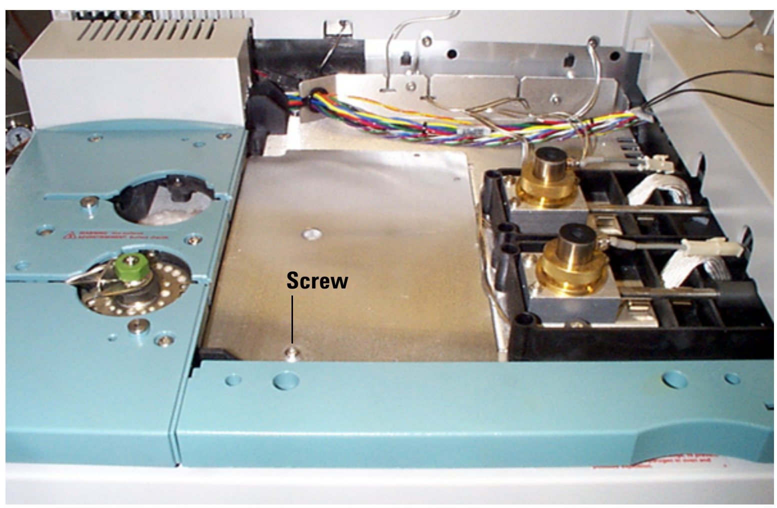

Figure 30 Install valve box blanking plate (6890N GC shown)

5 Mount the solenoid valve on the valve bracket with two screws.

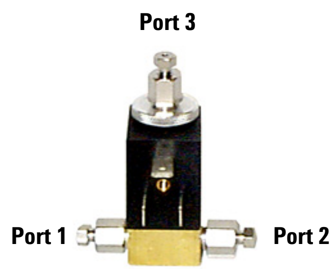

Figure 31 Valve with adapters

6 Mount the valve and bracket on the oven top as shown in Figure 32. Note that this secures the back of the panel.

7 Connect the valve driver cable to the valve (Figure 32).

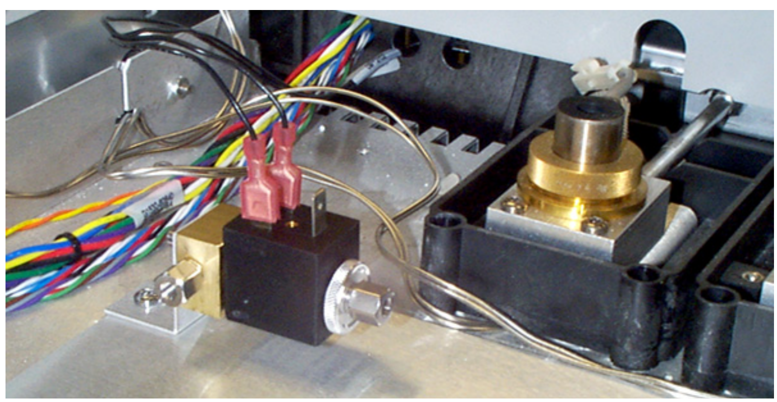

Figure 32 Mount valve and connect driver cable (6890N GC shown)

8 Remove the right side panel. The main board will resemble either Figure 33 (no valve bracket) or Figure 34 (valve bracket present), except that the valve cables will not be present.

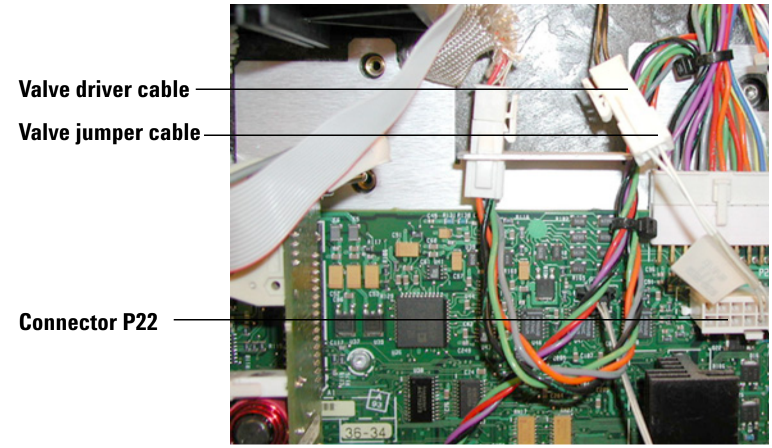

Figure 33 Main board without valve bracket (6890N GC shown)

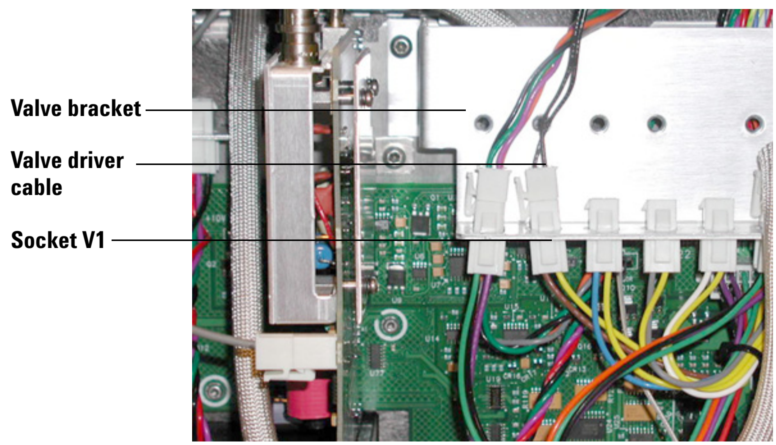

Figure 34 Main board with valve bracket (6890N GC shown)

9 Is a valve bracket installed on the main board?

- **No** (Figure 33 on page 56): Connect the free end of the valve driver cable to the valve jumper cable. Connect the other end of the valve jumper cable to connector P22 at the top of the main board.
- **Yes** (Figure 34 on page 56): Connect the free end of the valve driver cable to socket V1 on the valve bracket.

NOTE

You can use any of sockets V1 through V4 to control the valve, but must configure the selected valve as a *switching valve* and must refer to it when entering the cut time commands. This discussion assumes V1.

---

10 Connect the switching gas supply.

- **To supply the switching gas from a PCM**: Connect one of the 1-m coiled lengths of tubing to port **2** of the solenoid valve (Figure 35). Connect the stainless steel union to the other end.

  Cut the line from the PCM so that is about 15 cm (6 inches) long. Connect it to the stainless steel union.

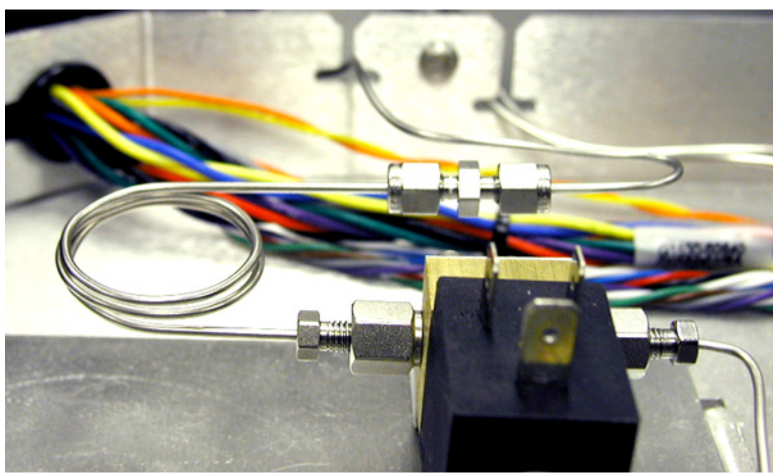

Figure 35 Pneumatics Control Module connection (6890N GC shown)

- **To supply the switching gas from an Auxiliary Pressure controller** (Figure 36): Ensure that the restrictor with the brown dot (6890 p/n 19231-60610, 7890A p/n G3430-80061) is installed in the Aux 3 channel outlet fitting.

  Remove the 1/8-inch end of the tubing using cutters or a file. Connect the tube to port **2** of the solenoid valve.

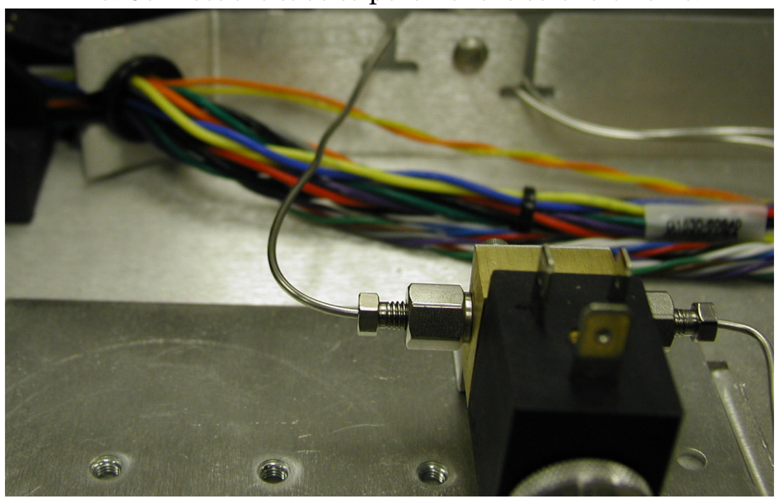

Figure 36 Auxiliary pressure controller connection (6890N GC shown)

Selecting and Installing the Shunt Restrictor

A Shunt Restrictor prevents sample diffusing into the unswitched control lines.
Use 0.010-inch id × 1/16-inch od × 15-cm restrictor tubing (p/n G2855-20700).

Install the selected coil between the free ends of the two tees (Figure 37).

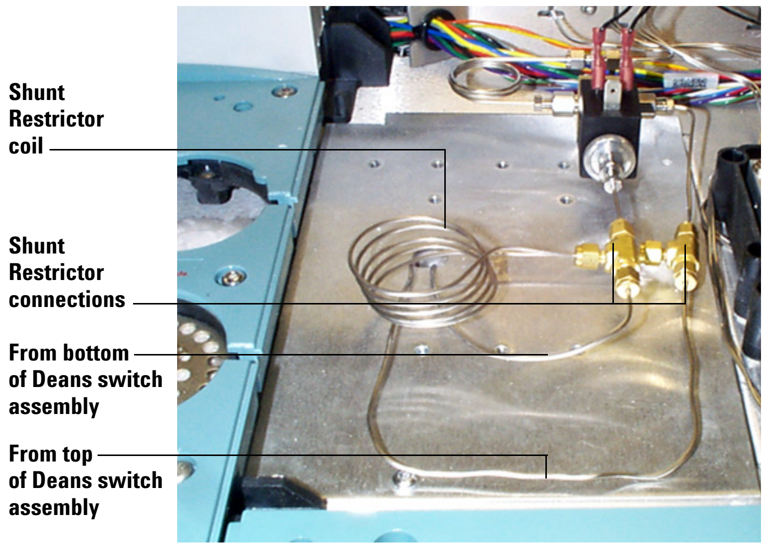

Figure 37 Install the Shunt Restrictor coil (6890N GC shown)

Backflushing to Save Time

For the example gasoline method, the three analytes are completely eluted from both columns by 4 minutes. However, the method must be run until about 22 minutes to flush all of the gasoline components from the column (Figure 38).

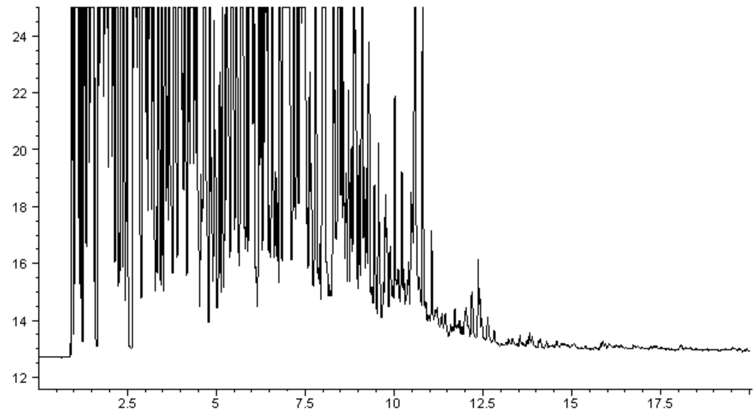

Figure 38 Eluting gasoline

To shorten the analysis time, the temperature or pressure could be ramped after 4 minutes to push the remaining gasoline components out of the column more rapidly.

If a split/splitless inlet is used, backflushing is another alternative with the Deans switching system.

In the example gasoline method, the oven is programmed at 40 °C for 1 minute, ramped at 10 °C/min to 250 °C, and held for 5 minutes. Since all the analyte peaks are out by 4 minutes, which corresponds to an oven temperature of 70 °C, the column controls can be set to backflush beginning at 4 minutes.

For 6890 GC

Starting at 4.00 minutes, program the split inlet pressure to decrease at 90 psi/min from 22.86 psi down to 0.5 psi. At the same time, program the PCM (or Aux EPC) module to increase in pressure at 90 psi/min from 16.26 psi up to 60 psi.

These pressure changes reverse the carrier flow in the primary column and increase the carrier flow in the secondary column. Heavy components in the primary column are backflushed out the split vent.

Backflushing removes heavier components rapidly. In this example, backflushing for only 2 minutes completely clears both columns. The run time is reduced to 6 minutes.

Figure 39 shows the chromatogram of the pump gasoline, but now with backflushing. The run is over in 6 minutes.

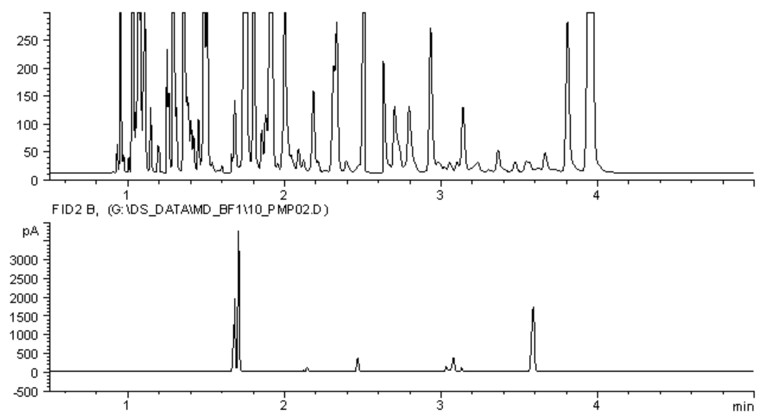

Figure 39 Gasoline with backflushing

Check to see if the backflushing time is sufficient by running a blank run (without backflushing) after running a backflush method. If the backflushing time is too short, heavy peaks will remain in the column and appear in the blank run. If they do, extend the backflushing time.

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