5 Columns and Traps

Capillary columns

Column hanger

- Procedure: Preparing capillary columns
- Procedure: Installing capillary columns in the split/splitless inlet
- Procedure: Installing capillary columns in the cool on-column inlet
- Procedure: Installing capillary columns in the purged packed inlet
- Procedure: Installing capillary columns in the PTV inlet and Volatiles Interface
- Procedure: Installing capillary columns in NPD and FID detectors
- Procedure: Installing capillary columns in the TCD
- Procedure: Installing capillary columns in the μ-ECD
- Procedure: Installing capillary columns in the FPD

Ferrules for capillary columns

Graphite and graphitized-Vespel ferrules Vespel ferrules

Packed metal columns

Overview: installing packed metal columns

Fittings

Preparing packed metal columns

Procedure: Making a spacer from Teflon tubing

- Procedure: Installing ferrules on a metal column
- Procedure: Installing an adapter in a detector fitting
- Procedure: Installing packed metal columns

Ferrules for packed metal columns

Packed glass columns

Overview: Installing glass packed columns

Procedure: Installing glass packed columns

Ferrules and O-rings for glass packed columns

Conditioning columns

Procedure: Preliminary column conditioning steps

Procedure: Conditioning a capillary column

Procedure: Conditioning packed columns

Conditioning chemical traps

Calibrating your capillary column (optional)

Calibration modes

Column calibration procedures

Procedure: Estimate the actual column length or diameter from an elution time

Procedure: Estimate the actual column length or diameter from the measured flow rate

Procedure: Estimate the actual column length and diameter

Columns and Traps

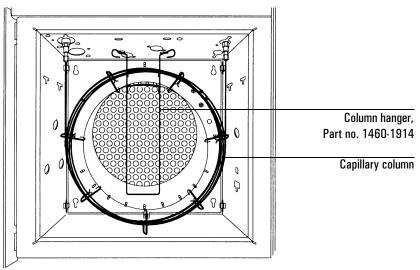
Capillary columns

This section contains information on preparing and installing capillary columns in inlets and detectors. See page <u>141</u> for packed metal columns and page <u>150</u> for packed glass columns.

Column hanger

Agilent capillary columns are wound on wire frames that mount on a hanger connected to the top of the oven interior.

You can connect the column hanger in two positions. Use the position that best centers the column in the oven. Column ends should make smooth curves to the inlet and detector fittings. Do not let any section of the column come in contact with the oven surfaces.



Procedure: Preparing capillary columns

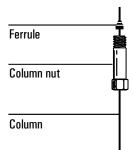
You must prepare your capillary column before installation. Proper preparation assures that the column end has no burrs or jagged edges and is not contaminated with graphite or other material.

WARNINGWear safety glasses to protect your eyes from flying particles while handling,
cutting, or installing glass or fused silica capillary columns. Use care in handling
these columns to prevent puncture wounds.

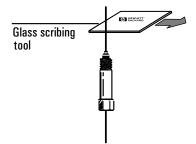
Materials required

Column nut and ferrule Capillary column Column cutter Magnifying loop Isopropanol Tissue

1. Place a capillary column nut and ferrule on the column.



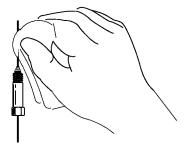
2. Score the column using a glass scribing tool. The score must be square to ensure a clean break.



3. Break off the column end by supporting it against the column cutter opposite the scribe. Inspect the end with a magnifying glass to make certain there are no burrs or jagged edges.



4. Wipe the column walls with a tissue dampened with isopropanol to remove fingerprints and dust.



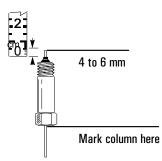
Procedure: Installing capillary columns in the split/splitless inlet

Before installing the column, be sure you have the correct glass liner installed. Instructions on choosing and installing liners are in <u>"Liners"</u>.

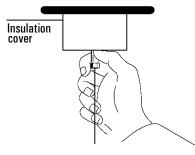
Materials required

Column nut and ferrule Column cutter Typewriter correction fluid 1/4-inch wrench Metric ruler

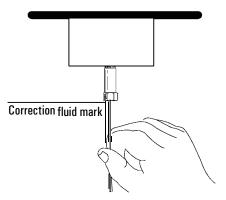
- 1. Prepare the column. See page <u>121</u> for instructions.
- 2. Position the column so it extends 4 to 6 mm above the end of the ferrule. Mark the column with typewriter correction fluid at a point even with the column nut.



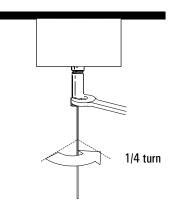
3. Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut until it starts to grab the column.



4. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut.



5. Tighten the column nut an additional 1/4 to 1/2 turn so that the column cannot be pulled from the fitting with gentle pressure.



6. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperature. Allow them to cool, and then retighten the fittings.

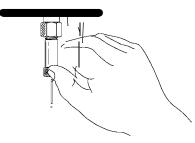
Procedure: Installing capillary columns in the cool on-column inlet

Before installing the column, be certain you have the correct hardware installed for the column and type of injection you are doing. See <u>"Hardware for the cool on-column inlet"</u> for detailed information.

Materials required

Column nut and ferrule Column cutter 1/4-inch wrench

- 1. Prepare the column. See page <u>121</u> for instructions.
- 2. Gently insert the column into the inlet until it bottoms. Insert the column nut into the inlet fitting and tighten the nut finger tight.



- 3. Tighten an additional 1/4-turn with a wrench or until the column does not move.
- 4. If you are using an automatic injection system with 250 μ m or 320 μ m columns, verify the installation by pushing the syringe manually into the inlet.
- 5. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

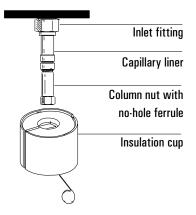
Procedure: Installing capillary columns in the purged packed inlet

Before installing a column in this inlet, be sure you have a capillary liner and glass insert installed. Instructions on choosing and installing this hardware are in <u>"Liners and inserts"</u>. If your insulation cup is not installed, begin with Step 1. Otherwise, begin with Step 4.

Materials required

Column nut and ferrule Column cutter Typewriter correction fluid 1/4-inch wrench Metric ruler Insulation cup No-hole ferrule to use as a plug when installing the insulation cup

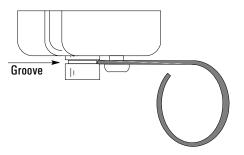
1. Install a plug in the inlet fitting.



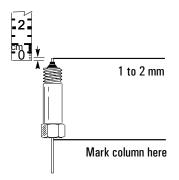
2. Install the insulation cup, if needed. Push the cup spring to the right. Slide the cup over the inlet fitting so that the insulation at the top of the cup is flush against the oven roof.



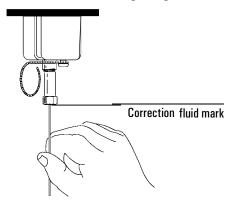
3. Place the spring into the groove in the inlet liner. Remove the column nut and put the no-hole ferrule aside.



- 4. Prepare the column. See page <u>121</u> for instructions.
- 5. Position the column so it extends above the end of the column nut by 1 to 2 mm. Mark the column with typewriter correction fluid at a point even with the column nut.



6. Push the column up 1 cm and guide it into the inlet liner. Slide the nut and ferrule up the column to the inlet liner. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Finger tighten the column nut until it starts to grab the column.



7. Tighten the column nut an additional 1/4 to 1/2 turn so that the column cannot be pulled from the fitting when gentle pressure is applied.



8. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow these to cool, and then retighten the fittings.

Procedure: Installing capillary columns in the PTV inlet and Volatiles Interface

The column installation procedures for these two inlets are unique to them. Details are in <u>"Procedure: Installing columns"</u> and <u>"Procedure: Installing columns"</u>.

Procedure: Installing capillary columns in NPD and FID detectors

Be sure you have the correct jet installed in your detector before installing a column. Details about choosing and installing detector jets are later in this chapter.

There are two types of NPD/FID detector fittings:

- Adaptable- for use with both packed and capillary columns
- *Capillary optimized* for use with capillary columns only. If your adaptable fitting does not have a capillary adapter installed, begin with step 1. If you have a capillary optimized fitting or if the capillary adapter is already installed in your adaptable fitting, begin with step 5.

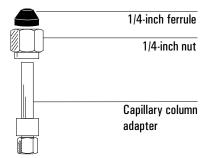
Materials required

Both fitting types:

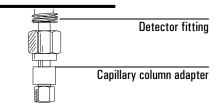
Column nut and ferrule Column cutter 1/4-inch wrench Metric ruler Typewriter correction fluid

For adaptable fitting only:

1/4-inch nut and ferrule Capillary column adapter 9/16-inch wrench 1. Assemble a brass nut and graphite/Vespel ferrule onto the adapter.

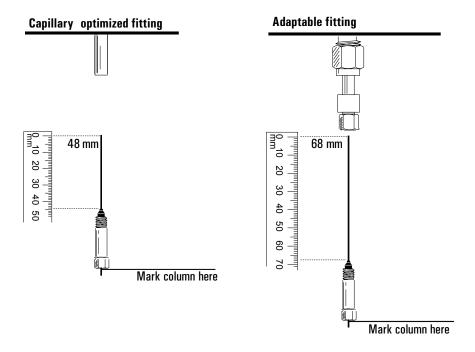


2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.

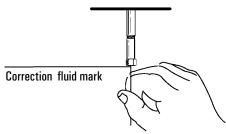


- 3. Prepare the column (see page <u>121</u> for instructions). If the column id is greater than 100 μ m, follow steps 7 to 9. If the column id is less than 100 μ m, follow steps 8 and 9.
- 4. If your column inside diameter is less than 100 μm:Position the column so it extends above the ferrule by 48 mm (*capillary*)

optimized fitting) or 68 mm (*adaptable* fitting). Mark the column with typewriter correction fluid even with the column nut.



- 5. Insert the column in the detector. Slide the nut and ferrule up the column to the detector base. Finger tighten the column nut until it starts to grab the column.
- 6. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Proceed to step 8.



7. Gently insert the column into the detector until it bottoms; do not attempt to force it further.

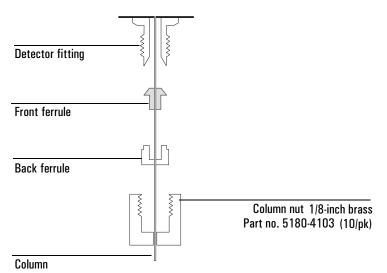
- 8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
- 9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

Procedure: Installing capillary columns in the TCD

Materials required

Capillary column adapter Column nut and ferrule set Column cutter Wrenches

1. Assemble the ferrules and a 1/8-inch brass nut on the column as shown.



See <u>Table 11</u> for the proper ferrules. Trim off a short piece of column to remove any ferrule fragments inside the column.

- 2. Insert the column into the detector until it bottoms. Do not attempt to force it.
- 3. Slide the column nut and ferrule up the column to the detector and tighten the nut finger tight.
- 4. Pull the column out 1 mm. Use a wrench to tighten the nut an additional 1/4 turn. The column should not move.

Column outside diameter	Back ferrule	Front ferrule
0.8 mm	G1530-80400	G1530-80410
0.53 mm	G1530-80400	G1530-80420
0.45 mm	G1530-80400	G1530-80430
No-hole ferrule	G1530-80400	G1530-80440

Table 11Ferrules for the TCD detector

Procedure: Installing capillary columns in the $\mu\text{-ECD}$

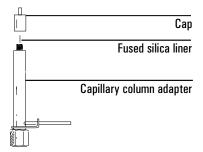
The detector is shipped with a capillary column adapter installed. If it has been removed, you must replace it before installing a capillary column.

The μ -ECD requires the indented liner, which is necked down near one end and is clear.

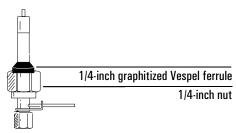
Materials required

Capillary column adapter Fused silica liner, indented 1/4-inch nut and 1/4-inch graphitized Vespel ferrule Column nut and ferrule Column cutter 1/4-inch and 9/16-inch wrenches

1. Remove the adapter cap and check the liner. Replace it if it is broken and reinstall the cap. The indentation must be at the cap end of the adapter.



2. Install a 1/4-inch nut and graphitized-Vespel ferrule on the adapter.

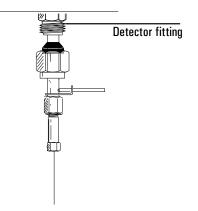


- 3. Prepare the column. See page <u>121</u> for instructions.
- 4. If the column id is $200 \ \mu m$ or more, push the column into the adapter until it stops at the indentation. Pull it back 1 to 2 mm and tighten the column nut firmly.

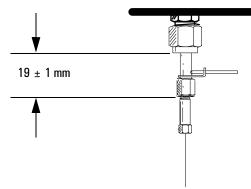
If the id is less than 200 μ m, mark the column 70±1 mm from the end. Insert column and nut into the adapter with the mark at the rear of the column nut, and tighten the column nut firmly.



5. Slowly install the adapter straight into the detector fitting. Make sure that the adapter is seated all the way into the detector fitting—jiggle it if necessary. Be careful not to break the column end.



If the adapter is properly installed, the distance between the 1/4-inch nut and the bottom of the adapter will be 19 ± 1 mm. If it is 22–23 mm, reinstall the adapter into the detector fitting.

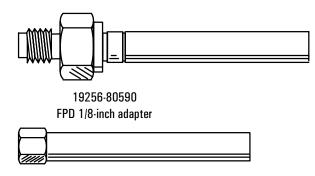


- 6. Slide the nut and ferrule up to the detector fitting and tighten the nut finger tight. Use a 9/16-inch wrench to tighten the nut an additional 1/4 turn.
- 7. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

Procedure: Installing capillary columns in the FPD

The FPD uses an adaptable fitting that can use both packed and capillary columns. If your adaptable fitting does not have a capillary adapter installed, begin with step 1. If the capillary adapter is already installed in your adaptable fitting, begin with step 5.

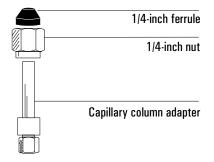
The FPD uses a special adapter for capillary columns. The FPD Capillary Adapter, part number 19256-80570, allows fused silica columns as large as 530 μ m ID to be run right to the base of the FPD flame, minimizing sample tailing or loss of chemically active sites.



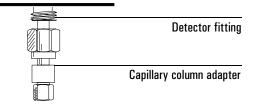
19256-80570 FPD Capillary Adapter

Materials required

Column nut and ferrule FPD Capillary column adapter 1/4-inch nut and ferrule Column cutter 1/4-inch wrench 9/16-inch wrench Metric ruler Typewriter correction fluid 1. Assemble a brass nut and graphite/Vespel ferrule onto the adapter.



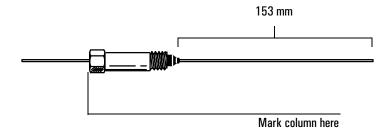
2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight. Use a wrench to tighten the nut an additional 1/4 turn.



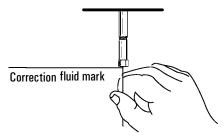
- 3. Install a column nut (part no. 18740-20870) and graphite ferrule (1.0 mm ID, part no. 5080-8773 or 0.5 mm ID, part no. 5080-8853) on the column.
- 4. After installing the nut and ferrule, prepare a fresh column end by cutting off a short piece of the column. See <u>page 121</u> for instructions.
- 5. Position the ferrule about 153 mm from the end of the column.

Optimum height depends on sample type and gas flow rates. If it is too high, the column end will be exposed to the flame. If too low, the sample may be exposed to hot stainless steel, causing slight tailing.

Mark the column at a point even with the bottom of the nut. Typewriter correction fluid works well.



- 6. Insert the column in the detector. Slide the nut and ferrule up the column to the detector base. Finger tighten the column nut until it starts to grab the column.
- 7. Adjust the column position so that the correction fluid mark on the column is even with the bottom of the column nut. Proceed to step 8.



- 8. Tighten the column nut finger tight, then withdraw the column about 1 mm. Use a wrench to tighten the nut an additional 1/4 turn.
- 9. After the column is installed at both inlet and detector, establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool and then retighten the fittings.

Ferrules for capillary columns

<u>Table 12</u> lists some of the ferrules used with capillary columns and inlet and detector liners/adapters. See the Agilent catalog for consumables and supplies for a more complete listing.

Graphite and graphitized-Vespel ferrules

Place some ferrules in a petri dish in the GC oven at 250 to 300°C for 30 minutes to remove compounds absorbed by the graphite. Leave a dish of assorted ferrules in the oven to ensure a clean supply.

The ferrule should slide onto the column but not fall off from its own weight. If it fits properly, 1/4 turn from finger tight will make a good seal. If it is loose, the column nut must compress the ferrule around the column. This is not a problem with soft graphite ferrules, but hard ferrules may require so much force that the inlet fitting may, the nut, or the ferrule may be damaged. With hard ferrules, it is best to start with an undersize hole and drill it to fit the column.

Vespel ferrules

These ferrules can be more leak-tight than graphite but have a lower temperature limit. Retighten after a few oven temperature cycles.

Typical use	Part no.
Inlet/detector liner/adapters	5080-8774
Capillary columns	5080-8773
Capillary columns	5080-8853
Connect column to inlet or detector	5181-8831
Cutting capillary columns	5181-8836
	Inlet/detector liner/adapters Capillary columns Capillary columns Connect column to inlet or detector

Table 12 Hardware Used with Capillary Columns

* Ferrule and O-ring ids

Packed metal columns

Overview: installing packed metal columns

There are two sizes of packed metal columns, 1/4-inch and 1/8-inch, in common use. This general procedure applies to both sizes of columns, as well as PTFE columns used with the FPD.

- 1. Prepare your packed column (page <u>143</u>).
- 2. See <u>Table 13</u> or <u>Table 14</u> for fittings required.
- 3. Install the adapter, if needed (page <u>147</u>).
- 4. Install the column (page <u>148</u>).
- 5. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

Fittings

Table 13 Fittings for 1/4-inch and 1/8-inch Packed Metal Columns

Inlet or 1/4-inch packed metal column		netal column	1/8-inch packed metal column	
detector	Where to install	Comments	Where to install	Comments
Purged-packed inlet	1/4-inch liner	See <u>"Procedure: Installing</u> <u>liners"</u> for instructions on installing liner.	1/8-inch liner	See " <u>Procedure: Installing</u> <u>liners"</u> for instructions on installing liner.
Adaptable NPD*, FID or FPD	1/4-inch adapter (Part no. 19231- 80530)	Remove or install adapter, as desired. See page <u>147</u> for instructions on installing an adapter.	1/8-inch adapter (Part no. 19231- 80520)	See page <u>147</u> for instructions on installing an adapter.
ECD	Detector fitting	Remove adapter, if necessary.	1/8-inch adapter. (Part no. 19301- 80530)	See page <u>147</u> for instructions on installing an adapter.
TCD	1/4-inch adapter (Part no. G1532- 20710)	See page <u>147</u> for instructions on installing an adapter.	Detector fitting	Remove adapter, if necessary.

* Do not remove the plugs from your NPD until you are ready to connect the column and operate the detector. Failure to observe this simple procedure may reduce the collector's effectiveness or slow down the bead's stabilization period the first time the detector is used.

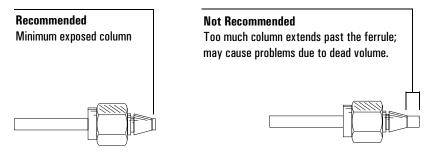
The FPD 1/8-inch OD Adapter, part number 19256-80590, allows installation of PTFE columns concentrically around the FPD fused silica liner, eliminating exposed hot stainless steel.

Table 14Fittings for FPD with a PTFE Column

1/4-in PTFE column		1/8-in PTFE column	
Where to install	Comments	Where to install	Comments
1/4-inch adapter (Part no. 19231-80530)	Remove or install adapter, as desired. See page <u>147</u> for instructions on installing an adapter.	1/8-inch adapter (Part no. 19256-80590)	See page <u>147</u> for instructions on installing an adapter.

Preparing packed metal columns

Before installing these columns, a ferrule should be locked on the column end so that it is flush with the end of the column. This prevents problems caused by dead volume in the fitting.



Use the following instructions to install new SWAGELOK nuts and ferrules onto 1/8-inch or 1/4-inch metal columns. If your column already has ferrules installed, proceed to the instructions on installing adapters (page <u>147</u>) or installing packed metal columns (page <u>148</u>).

Procedure: Making a spacer from Teflon tubing

Materials required

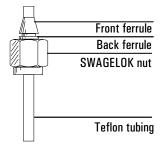
1/4-inch or 1/8-inch Teflon tubing 1/4-inch or 1/8-inch nut and ferrule set Bench vise Male SWAGELOK fitting 9/16-inch or 7/16-inch wrench Razor or sharp knife

1. Secure a new male SWAGELOK fitting in a bench vise.

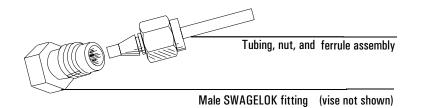


Male SWAGELOK fitting

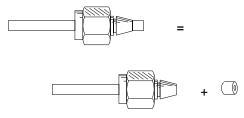
2. Slide a SWAGELOK nut, back ferrule, and front ferrule onto a piece of Teflon tubing. If the end of the tubing is not cut straight, use a razor or sharp knife to make a flat, smooth end.



3. Insert the Teflon tubing, ferrules, and nut into the vise-held SWAGELOK fitting. Tighten the nut 3/4 turn past finger tight to set the ferrules on the tubing.



- 4. Loosen the nut and remove the assembly from the male SWAGELOK fitting.
- 5. Cut off the end of the tubing extending beyond the ferrule with a razor or sharp knife. This piece of tubing is the spacer.



6. Insert the spacer into the vise-held SWAGELOK fitting.



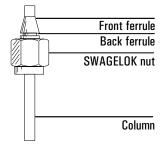
The male SWAGELOK fitting and spacer should be kept on hand to be used whenever new ferrules are being installed on a column.

Procedure: Installing ferrules on a metal column

Materials required

Male SWAGELOK fitting with Teflon tubing spacer SWAGELOK nut and ferrule set Wrenches

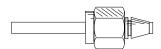
1. Install new SWAGELOK nut and ferrules on the column.



2. Install the Teflon tubing spacer in the male fitting. Fully insert the column with its nut and ferrules into the vise-held fitting. Tighten the nut finger tight.

Use a wrench to tighten the nut an additional 1-1/4 turn for 1/4-inch columns or 3/4 turn for 1/8-inch columns.

3. Unscrew the column nut from the vise-held fitting and remove the column. Ferrules should now be set in place on the column with the column correctly positioned.



Procedure: Installing an adapter in a detector fitting

This is a general procedure for installing many types of adapters onto detector fittings. See <u>Table 13</u> for adapter part numbers.

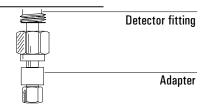
Materials required

7/16-inch or 9/16-inch wrench Graphitized Vespel ferrule Nut Adapter

1. Assemble a brass nut and a graphitized Vespel ferrule onto the adapter.

Graphitized Vespel ferrule	
Nut	
Adapter	

2. Insert the adapter straight into the detector base as far as possible. Hold the adapter in this position and tighten the nut finger tight.



1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.

3. Proceed to "Installing Packed Metal Columns" on page <u>148</u>.

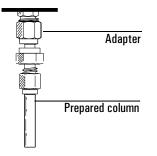
Procedure: Installing packed metal columns

Before following this procedure, make sure an adapter or liner is installed (page 147), if needed, and that your column is prepared (page 143.)

Materials required

Prepared metal column Column adapter, if needed Wrench

1. Insert the column into the adapter, detector, or inlet liner until it bottoms. Tighten the nut finger tight.



- If you are installing a column directly into the detector fitting:
 1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.
 1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.
- 3. If you are installing a column onto an adapter: Tighten the column nut using two wrenches in opposition, one on the column nut and the other on the liner or adapter body. This prevents the liner or adapter from rotating while you tighten the column nut.
 1/4-inch column, tighten an additional 3/4 turn with a 9/16-inch wrench.
 1/8-inch column, tighten an additional 1/4 turn with a 7/16-inch wrench.
- 4. Establish a flow of carrier gas through the inlet. Heat the oven, inlet, and detector to operating temperatures. Allow them to cool, and then retighten the fittings.

Ferrules for packed metal columns

<u>Table 15</u> lists some of the nuts and ferrules used with packed metal columns. Consult the Agilent catalog for consumables and supplies for a more complete listing.

Ferrules that are prepared improperly cause leaks and contamination. Here are some hints to avoid problems.

Graphite and graphitized-Vespel ferrules. Place these ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

Vespel ferrules. These ferrules can be more leaktight than graphite, but have a lower temperature limit. They should be retightened after a few oven temperature cycles to ensure a good seal. Be sure to use the correct ferrule for the size column you are using.

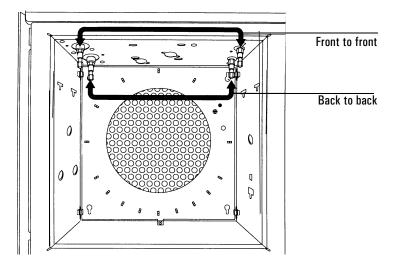
Table 15 N	Nuts and Ferrules	Used with Packed	Metal Columns
------------	-------------------	-------------------------	---------------

. *		- .
Item*	Typical use	Part no.
1/4-inch swage stainless steel, pkg of 20 (<i>nut, front ferrule, back ferrule</i>)	1/4-inch	5080-8753
1/8-inch swage stainless steel, pkg of 20 (<i>nut, front ferrule, back ferrule</i>)	1/8-inch	5080-8751
1/4-inch swage brass, pkg of 20 each (<i>nut, front ferrule, back ferrule</i>)	1/4-inch	5080-8752
1/8-inch swage brass, pkg of 20 each (<i>nut, front ferrule, back ferrule</i>)	1/8-inch	5080-8750
1/4-inch graphitized Vespel ferrule, pkg of 10	inlet/detector liner/adapters 1/4-inch columns	5080-8774
1/8-inch graphitized Vespel ferrule, pkg of 10	1/8-inch columns	0100-1107

* O-ring and ferrule ids

Packed glass columns

Glass packed columns must be installed simultaneously at the inlet and the detector and must be installed parallel to the oven door:



You can install glass packed columns directly in the purged-packed inlet, μ -ECD, and adaptable NPD, FID, and FPD fittings. The TCD requires an adapter.

There are three types of glass packed columns available. You must make certain that your column is compatible with the inlet fitting and detector used. <u>Table 16</u> summarizes the inlet and detector fittings required and the appropriate column configuration.

Overview: Installing glass packed columns

- 1. See <u>Table 16</u> for information on fittings and column configuration required.
- 2. Remove or install an adapter, if necessary (see page <u>147</u>).
- 3. Follow the general procedure for installing glass columns on page <u>152</u>.

Inlet or detector	Where to install	Column configuration	Comments
Purged-packed inlet	Inlet fitting (no liner installed) <i>or</i> 1/4-inch liner*	A or B, depending on the detector C (works with all detectors)	Allow at least 50 mm of empty column to prevent an inserted syringe needle from contacting either the glass wool plug or column packing.
<i>Adaptable</i> NPD, FID, or FPD Cannot use with <i>capillary optimized</i> detector	Detector fitting	A	Remove adapter, if installed. There must be at least 40 mm of empty column to prevent the bottom end of the jet from touching either column packing or the glass wool plug.
μ -ECD	Detector fitting	Α	Remove capillary column adapter, if installed.
TCD	1/4-inch adapter (Part no. G1532-20710)	В	Instructions for installing adapters are on page <u>147</u> .

Table 16 Installing Glass Packed Columns

* See the "Procedure: Installing liners" for instructions on installing liners

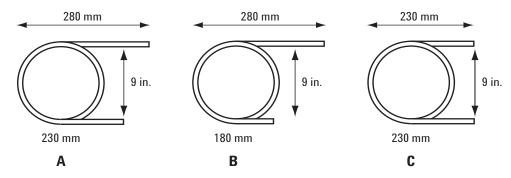


Figure 19. Column Configurations

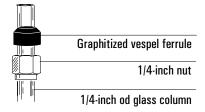
Procedure: Installing glass packed columns

Materials required

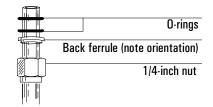
Recommended:	Alternative:
Two 1/4-inch graphitized Vespel ferrules	Four O-rings
Two 1/4-inch nuts	Two back ferrules
9/16-inch wrench	Two 1/4-inch nuts
	9/16-inch wrench

1. Assemble a brass nut and graphitized Vespel ferrule on each end of the column. Alternative method: Install a 1/4-inch nut, back ferrule, and two O-rings on each end of the column. An extra O-ring below the nut keeps the nut from dropping into the coiled portion of the column.

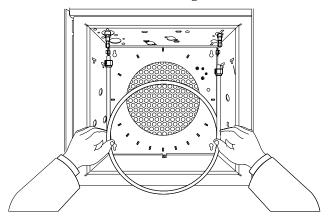
Recommended



Alternative installation method



2. Insert the column into the inlet until it bottoms. Insert the column into the detector fitting but *do not* force it. It may be necessary to start the long end of the column in the inlet at an angle to clear the oven floor.



- 3. Withdraw the column 1 to 2 mm from both the inlet and detector. Tighten both column nuts finger tight.
- 4. Tighten both column nuts 1/4 turn with a wrench. If you use graphitized Vespel ferrules, proceed to step 5. If you use O-rings, proceed to step 6.
- **Caution** Overtightening the column nut or forcing it to bottom in both the inlet and detector may shatter the column.
 - 5. Set flow through the column and raise the inlet, detector, and oven to operating temperature. Then set the oven to ambient and allow it to cool.
 - 6. Use the wrench to tighten the nut an additional 1/2 turn. Tighten further, as necessary, to prevent leakage.

Ferrules and O-rings for glass packed columns

<u>Table 17</u> lists ferrules and O-rings used with glass packed columns. Consult the Agilent Catalog for consumables and supplies for a more complete listing.

Ferrules that are prepared improperly cause leaks and contamination. To avoid problems, place graphitized Vespel ferrules in a petri dish and bake in the GC oven at 250 to 300°C for 30 minutes before use to remove organic compounds absorbed by the graphite. Leave a petri dish of assorted ferrules in the GC oven to ensure a clean supply.

ltem*	Typical use	Part no.
1/4-inch graphitized Vespel ferrule, pkg of 10	Inlet/detector liners, 1/4-inch glass packed columns	5080-8774
Silicone O-ring, 6.0-mm	1/4-inch glass packed columns	0905-0322

Table 17 Glass Packed Columns Consumables

* O-ring and ferrules ids

Conditioning columns

Conditioning involves establishing a flow of carrier gas through a column and then heating it for one-half hour for capillary columns and overnight for packed columns. This drives off contaminants and makes the column fit for analytical use.

New packed columns should be conditioned, since they often contain volatile contaminants from the coating process. It may also be necessary to condition a used column that has been stored for some time without end caps or plugs.

Conditioning is not as important with capillary columns since they contain a minimal amount of stationary phase.

The following procedures include preliminary steps and the actual conditioning procedure, which differs for packed and capillary columns.

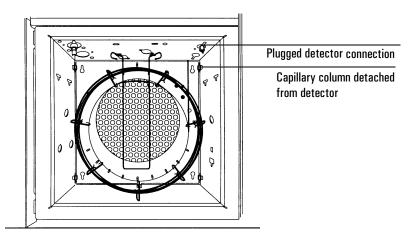
Procedure: Preliminary column conditioning steps

Materials required

Two 7/16-inch wrenches

No-hole ferrule and capillary nut for detector connection

- 1. Turn off the detectors. Shut off the detector support gases. It is especially important to shut off hydrogen!
- 2. If the column to be conditioned is not already installed, connect one end to an available inlet. If you are not sure how to install a column, see the instructions earlier in this chapter. DO NOT connect the remaining end to a detector!
- 3. If you plan to condition a capillary column in a split/splitless inlet, install the proper liner and attach the column in the normal manner, making sure about 5 to 7 mm of column extends above (in front of) the column ferrule.
- 4. Cap the detector(s) fittings with the no-hole ferrule and column nut.



Procedure: Conditioning a capillary column

WARNING Do not use hydrogen as the carrier for conditioning! It could vent into the oven and present an explosion hazard.

1. Select an appropriate column pressure—given as **psi(kPa)**—from this table.

	Inside diameter					
Length, m	0.10 mm	0.20 mm	0.25 mm	0.32 mm	0.53 mm	
10	25 (170)	6 (40)	3.7 (26)	2.3 (16)	0.9 (6.4)	
15	39 (270)	9 (61)	5.6 (39)	3.4 (24)	1.4 (9.7)	
25	68 (470)	15 (104)	9.5 (65)	5.7 (40)	2.3 (16)	
30	83 (570)	18 (126)	12 (80)	7 (48)	2.8 (19)	
50		32 (220)	20 (135)	12 (81)	4.7 (32)	
60		39 (267)	24 (164)	14 (98)	5.6 (39)	

- 2. Enter the selected pressure. Let gas flow through the column at room temperature for 15 to 30 minutes to remove air.
- 3. Program the oven temperature from room temperature to the maximum temperature for the column. Increase the temperature at a rate of 10 to 15°C/min and hold at the maximum temperature for 30 minutes.
- 4. If you will not be using the conditioned column immediately, remove it from the oven. Cap both ends to prevent air, moisture, and other contaminants from entering the column.

Procedure: Conditioning packed columns

WARNING Do not use hydrogen as the carrier for conditioning! It could vent into the oven and present an explosion hazard.

- 1. Press [Col 1] or [Col 2] to open the column control table.
- 2. Enter an appropriate column flow:
 - 20 to 30 mL/min for 2 mm ID glass or 1/8 inch OD metal columns.
 - 50 to 60 mL/min for 4 mm ID glass or 1/4 inch OD metal columns.
- 3. The conditioning temperature is never greater than the maximum temperature limit for the column; 30°C less than the maximum is usually sufficient. Slowly raise oven temperature to the conditioning temperature for the column.

	OVEN		
Гemp		45	50
Init temp			50
[nit time			5
ate 1		1	5.00
inal temp	1		250
inal time	1	72	0.00
lode: Const	ant f	LOW	

4. Continue conditioning overnight at the final temperature. If you will not be using the conditioned column immediately, remove it from the oven. After removing the column, cap both ends to prevent air, moisture, or other contaminants from entering the column.

Conditioning chemical traps

If your traps are preconditioned, you will not need to perform a conditioning procedure before using them. However, all traps need regeneration periodically, for example after using one to four cylinders of gas, or if gases of the highest purity were not used. You can recondition Agilent moisture and activated charcoal traps. Agilent oxygen traps cannot be reconditioned; you must replace them if they become contaminated. Follow the manufacturer's instructions for reconditioning traps.

The molecular sieve and activated charcoal traps can also be repacked. Instructions for repacking traps are shipped with each trap.

Item	Part no.
Moisture trap (packed with Molecular Sieve 5A, 45/60 mesh)	5060-9077
Conditioned moisture trap (packed with preconditioned Molecular Sieve 5A, 45/60 mesh)	5060-9084
Activated charcoal trap	5060-9096
Molecular Sieve 5A (100 grams, 45/60 mesh)	5080-6759
Activated charcoal (100 grams, 30/60 mesh)	5080-6751
Cap for ends of traps, 1/8-inch, 6 per package	5180-4124
Reducer trap fittings	5062-3502

Table 18 Ordering Information for Agilent Traps

Calibrating your capillary column (optional)

Preparation

As you use a capillary column, you may occasionally trim off portions, changing the column length. If measuring the actual length is impractical, and if you are using EPC with a defined column, you can use an internal calibration routine to estimate the actual column length. Similarly, if you do not know the column internal diameter or believe it is inaccurate, you can estimate the diameter from related measurements.

Before you can calibrate the column, make sure that:

- You are using a capillary column
- The column is defined
- There are no oven ramps
- The column gas source (usually the inlet) is On and non-zero

Also note that column calibration fails if the calculated column length correction is ≥ 5 m, or if the calculated diameter correction is ≥ 20 µm.

Calibration modes

There are three ways to calibrate the column length and/or diameter:

- Calibrate using an actual measured column flow rate
- Calibrate using an unretained peak time (elution time)
- Calibrate both length and diameter using flow rate and elution time

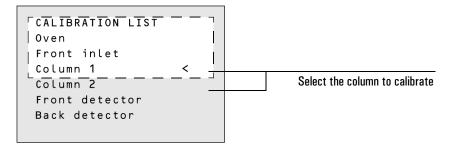
CautionWhen you measure the column flow rate, be sure to convert the measurement
to normal temperature and pressure if your measurement device does not report
data at NTP. If you enter uncorrected data, the calibration will be wrong. See
"Measuring flow rates with a bubble meter" for details.

Column calibration procedures

These procedures are described below using Column 1 as an example.

Procedure: Estimate the actual column length or diameter from an elution time

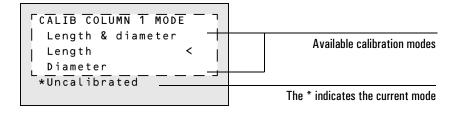
- 1. Set oven ramp 1 to 0.00, then verify that the column is defined. For more information, see <u>"Procedure: Setting up an isothermal run"</u> or <u>"Configure the column"</u>.
- 2. Perform a run using an unretained compound and record the elution time.
- 3. Press [Options]. Scroll to Calibration and press [Enter].
- 4. From the calibration list, select Column 1 or Column 2 and press [Enter].



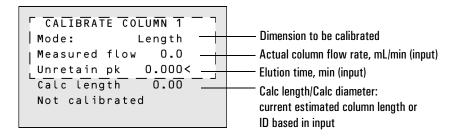
5. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



6. To recalibrate or to change calibration mode, press [Mode/Type] to see the column calibration mode menu.



7. Scroll to Length or Diameter and press [Enter]. The following menu appears:

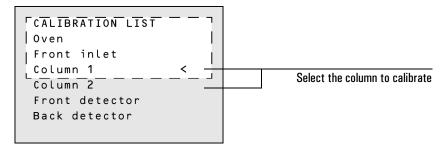


- 8. Scroll to Unretain pk and enter the actual elution time from the run performed above.
- 9. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

Procedure: Estimate the actual column length or diameter from the measured flow rate

- 1. Set oven ramp 1 to 0.00, then verify that the column is defined. For more information, see <u>"Procedure: Setting up an isothermal run"</u> or <u>"Configure the column"</u>.
- 2. Set the oven, inlet, and detectors temperatures to 35°C and allow them to cool to room temperature.
- 3. Remove the column from the detector.
- CautionWhen you measure the column flow rate, be sure to convert the measurement
to normal temperature and pressure if your measurement device does not report
data at NTP. If you enter uncorrected data, the calibration will be wrong. See

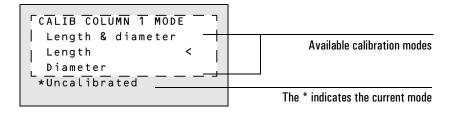
<u>"Measuring flow rates with a bubble meter"</u> for details.
 - 4. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
 - 5. Press [Options]. Scroll to Calibration and press [Enter].
 - 6. From the calibration list, select Column 1 or Column 2 and press [Enter].



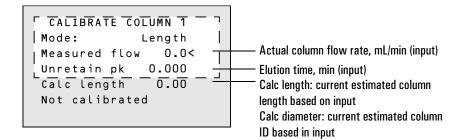
7. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



8. To recalibrate or change calibration mode, press [Mode/Type] to see the column calibration mode menu.



9. Scroll to Length or Diameter and press [Enter]. The following menu appears:



- 10. Scroll to Measured flow and enter the corrected column flow rate (in mL/min) from the run performed above.
- 11. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.

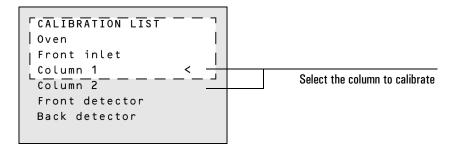
Procedure: Estimate the actual column length and diameter

- 1. Set oven ramp 1 to 0.00, then verify that the column is defined. For more information, see <u>"Procedure: Setting up an isothermal run"</u> or <u>"Configure the column"</u>.
- 2. Perform a run using an unretained compound and record the elution time.
- 3. Set the oven, inlet, and detectors temperatures to 35°C and allow them to cool to room temperature.
- 4. Remove the column from the detector.

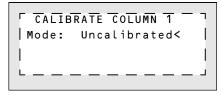
CautionWhen you measure the column flow rate, be sure to convert the measurement
to normal temperature and pressure if your measurement device does not report
data at NTP. If you enter uncorrected data, the calibration will be wrong. See

<u>"Measuring flow rates with a bubble meter"</u> for details.

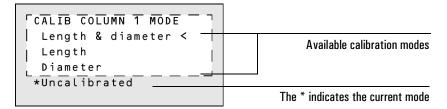
- 5. Measure the actual flow rate through the column using a bubble meter. Record the value. Reinstall the column.
- 6. Press [Options]. Scroll to Calibration and press [Enter].
- 7. From the calibration list, select Column 1 or Column 2 and press [Enter].



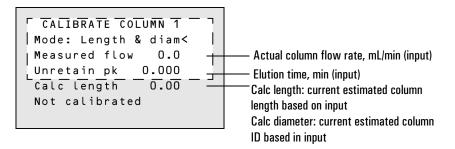
8. The GC displays the current calibration mode for the column. In this example, the column is uncalibrated.



9. To recalibrate or change calibration mode, press [Mode/Type] to see the column calibration mode menu.



10. Scroll to Length or Diameter and press [Enter]. The following menu appears:



- 11. Scroll to Measured flow and enter the corrected column flow rate (in mL/ min) from the run performed above.
- 12. Scroll to Unretain pk and enter the actual elution time from the run performed above.
- 13. When you press [Enter], the GC will estimate the column length or diameter based on the elution time input and will now use that data for all calculations.