

Chromatography Products

How to Condition a New Capillary GC Column

by Alan Sensue, Restek Technical Services Chemist

So, you've just purchased a new capillary column and you need to install it into your GC?

While many analysts have a routine that they perform, there are many new users who might not know the proper procedure. Here, we summarize proper column installation and conditioning. Note that detailed information about installing a capillary GC column is available in the article *Restek Capillary Column Installation Guide* on this website.

To begin, cool your GC and remove the old column. Don't discard it just yet – you might need it to troubleshoot the system if you encounter problems after installing the new column. Cap the ends of the old column with capillary column caps, or with pieces of used injection port septum, and put it aside.

Cut off the flame-sealed ends of the new column, work a fitting and ferrule 6-8 inches onto the injection port end of the column (the end at the front of the column cage as you are reading the column tag) and remove an additional 4 inches of column. This will remove any remaining stationary phase that had been heat-damaged during the flame sealing process, and eliminate ferrule fragments introduced into the column while you were installing the ferrule. Carefully insert the appropriate length of column into the injection port, as instructed in your instrument manual, tighten the fitting, turn on the carrier gas, and adjust the flow rate to the desired value (see Table 1). Confirm that there is flow through the column by submerging approximately 1 inch of the free end of the column in methanol, or other solvent compatible with the stationary phase in the column, and verify that a steady stream of bubbles is produced. Remove the end of the column from the solvent, then purge the column for 10-40 minutes at the appropriate carrier gas flow rate. When using helium or hydrogen as the carrier gas, purge as recommended in Table 1.

Table 1 Flow rates for purging capillary GC columns with helium or hydrogen carrier gas

Minimum Flow Rate (mL/ min.)	Minimum Purge Time (min.)
5.0	10
1.5	20
1.0	25
0.8	30
0.5	40
	min.) 5.0 1.5 1.0 0.8

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Purging will remove all traces of air (oxygen) from the injection port and column, which must be done before you heat the column. At elevated temperatures, even trace levels of oxygen will quickly cause irreversible damage to the stationary phase. During this purge, CHECK FOR LEAKS using an <u>electronic leak detector</u>. To prevent oxygen from entering the system and damaging the column, the system must be completely leak free. In addition, the carrier gas must be passed through a <u>high-quality oxygen trap</u>.

After purging, the column is ready to be conditioned. Do NOT connect the capillary column to the detector at this time. Conditioning time and temperature depend on several factors, including stationary phase chemistry, stationary phase thickness, the intended application for the column, and the type of detector you will be using. The following instructions should enable you to properly condition your new column. Note that the column conditioning times in Table 2 are **approximate**. The general rule is to condition the column only long enough to achieve a stable baseline and an acceptable signal-to-noise ratio for the compound peaks anticipated in the analysis. If you have questions, please call <u>Restek Technical Service</u> at 800-356-1688, ext. 4. We will help you to determine the best conditioning procedure for your new column – even if it isn't a Restek column.

Overnight conditioning is only recommended in a few situations. When a column will be used at its maximum operating temperature limit for extended periods of time (such as with simulated distillation analysis),

or when a thick-film column will be coupled to a very high sensitivity detector (such as an electron capture detector or mass spectrometer), overnight conditioning might be necessary to achieve a stable baseline. Note, however, that operating capillary columns at their maximum temperatures over a long period of time will shorten the lifetime of the column.

In most situations, we recommend that a new column be installed first thing in the morning, purged and leak checked as described above, and conditioned as follows:

- 1. Set your GC oven temperature to 40°C, and set a temperature ramp rate of 10°C/minute.
- 2. Program the oven either to 20°C above the final temperature called for in the analysis or to the column's maximum ISOTHERMAL temperature whichever is lower.
- 3. While the oven temperature begins its ramp, heat the injection port to the appropriate temperature.
- 4. After the oven temperature reaches the set point, hold this temperature for the time listed in Table 2.
- 5. With carrier gas still flowing, cool the oven, install a fitting and ferrule onto the detector end of the column as outlined above, connect the column to the detector, and repeat steps 1-4.

Your column should now be conditioned. As mentioned above, if you are using a high sensitivity detector, such as a mass spectrometer or an electron capture detector, the column might need additional conditioning to ensure a stable baseline. Consult the instrument manual for information.

Because column connections are a common source of leaks, if you plan to do dual-column analysis, you should install, condition, and test each column individually. Only after the performance of both columns has been proven to be acceptable should they be connected in common to a guard column, using a <u>SeCure Y</u>, or similar connector.

Proper column conditioning is essential for optimal column performance. Once you establish a conditioning procedure that works well for you, record this information in your laboratory notebook or equipment logbook for future reference. If you encounter problems during column installation or conditioning, or at any other point in your analysis, remember that the Restek Technical Service chemists are only a phone call or e-mail message away.

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Table 2 Conditioning times for capillary GC columns

For these columns:

MXT®-1, MXT®-1HT, MXT®-1SimDist, MXT®-500

Rtx @ -1, Rtx @ -1MS, Rtx @ -1PONA, Rtx @ -5, Rtx @ -5MS, Rtx @ -5SilMS, Rtx @ -5Amine, Rtx @ -440, Rtx @ -PCB, Rtx @ -2887, Rtx @ -627, Rtx @ -TNT, Rtx @ -TNT2

Rxi -1ms, Rxi -5ms

Stx -500

XTI®-5

Column Length (meters)	Film Thickness	Approx. Time	
	(µm)	(min.)	(hr.)
<u><</u> 15	0.1 - 0.25	15	0.25
	0.5 - 1.0	30	0.5
	1.0 - 1.5	60	1
	1.5 - 3.0	90	1.5
30	0.1 - 0.25	30	0.5
	0.5 - 1.0	45	0.75
	1.0 - 1.5	60	1
	1.5 - 3.0	90	1.5
<u>></u> 60	0.1 - 0.25	60	1
	0.5 - 1.0	90	1.5
	1.0 - 1.5	120	2
	1.5 - 3.0	150	2.5

For these columns:

MXT®-35, MXT®-50, MXT®-65, MXT®-65TG, MXT®-200, MXT®-624, MXT®-1301, MXT®-1701, MXT®-502.2, MXT®-Volatiles

Rtx®-17, Rtx®-20, Rtx®-35, Rtx®-35MS, Rtx®-35 Amine, Rtx®-50, Rtx®-65, Rtx®-65TG, Rtx®-200, Rtx®-200MS, Rtx®-624, Rtx®-1301, Rtx®-1701, Rtx®-BAC1, Rtx®-BAC2, Rtx®-CLPesticides, Rtx®-CLPesticides2, Rtx®-Dioxin, Rtx®-Dioxin2, Rt

 $\label{lem:condition} OPPesticides, \ Rtx @-OPPesticides2, \ Rtx @-G43, \ Rtx @-502.2, \ Rtx @-VMS, \ Rtx @-VGC, \ Rtx @-VRX, \ Rtx @-Volatiles$

Stx -CLPesticides, Stx -CLPesticides2

Column Length (meters)	Film Thickness	Approx. Time	
	(µm)	(min.)	(hr.)
<u><</u> 15	0.1 - 0.25	20	0.3
	0.5 - 1.0	40	0.7
	1.0 - 1.5	60	1
	1.5 - 3.0	80	1.3
30	0.1 - 0.25	40	0.7
	0.5 - 1.0	60	1
	1.0 - 1.5	80	1.3
	1.5 - 3.0	100	1.7
≥60	0.1 - 0.25	80	1.3
	0.5 - 1.0	120	2
	1.0 - 1.5	160	2.7
	1.5 - 3.0	200	3.3

For these columns:

DEX chiral phases

FAMEWAX

MXT®-WAX

Rt-CW20M F&F, Rt-2560, Rt-TCEP

Rtx®-225, Rtx®-2330, Rtx®-WAX

Stabilwax @, Stabilwax @-DA, Stabilwax @-DB

Column Length (meters)	Film Thickness (µm)	Approx. Time	
		(min.)	(hr.)
≤15	0.1 - 0.25	30	0.5
_	0.5 - 1.0	45	0.75
	1.0 - 1.5	60	1
	1.5 - 3.0	90	1.5
30	0.1 - 0.25	60	1
	0.5 - 1.0	90	1.5
	1.0 - 1.5	120	2
	1.5 - 3.0	150	2.5
>60	0.1 - 0.25	80	1.3
_	0.5 - 1.0	120	2
	1.0 - 1.5	160	2.7
	1.5 - 3.0	200	3.3