





How to Condition a New Capillary GC Column

So, you've just purchased a new capillary column and you need to install it into your GC, but what's the best procedure? While many analysts have a routine that they perform, there are many new users who might not know the proper method. Here, we summarize proper column installation and conditioning. Note that detailed information about installing a capillary GC column is available in the article <u>Restek</u> <u>Capillary Column Installation Guide</u> on this website.

To begin, cool all heated zones in 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. Seal the ends of the old column with capillary column end caps, or with pieces of a used injection port septum, and then put the old column aside. You are now ready to install and condition your new column.

Carefully remove the new column from its box and take off the end caps or septa that came on it. Then work a fitting and ferrule 15-20 cm (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 or the side with the Integra-Guard[®] retention gap/guard column section) and remove approximately 10 cm (4 inches) of column using the smooth side of a <u>ceramic scoring wafer</u> (or similar tool). This will prevent ferrule fragments that entered the end of the tubing while you were installing the ferrule from being introduced into the analytical column. 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 I). Confirm that there is flow through the column by submerging approximately 2.5 cm (1 inch) of the free end of the column in methanol, or another 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, and 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 I.

*Caution: Hydrogen gas is explosive; do not allow the oven to fill with hydrogen and do not heat the column while hydrogen is flowing into the oven. Take all necessary safety precautions when using hydrogen as the carrier gas.

Column ID (mm)	Minimum Flow Rate (mL/min)	Minimum Purge Time (min)
0.45 / 0.53	5.0	10
0.32	1.5	20
0.25 / 0.28	1.0	25
0.15 / 0.18	0.8	30
0.10	0.5	40

Table I: Flow rates for purging capillary GC columns with helium or hydrogen* carrier gas.

Purging will remove all traces of oxygen and moisture from the injection port and column, which must be done before you heat the column. At elevated temperatures, even trace levels of oxygen or water will quickly cause irreversible damage to the stationary phase. During this purge, CHECK FOR LEAKS at the injection port using an <u>electronic leak detector</u>. To prevent oxygen and moisture 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> before entering the column.

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, 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 II 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 e-mail <u>Restek</u> <u>Technical Service</u> or call us at 800-356-1688, ext. 4. We will help you to determine the best conditioning procedure for your new column.

Note that overnight conditioning is only recommended in a few situations, as it can shorten the lifetime of the column if not done properly. 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, high-bleed 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.

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. Heat the injection port to the appropriate temperature and then run the oven program.
- 4. After the oven temperature reaches the set point, hold this temperature for the time listed in Table II.
- 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 an <u>MXT®</u> <u>"Y"-Union</u>, <u>"Y" Press-Tight</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 <u>e-mail message</u> away.

Table II: Conditioning times for capillary GC columns.

For these columns:

MXT[®]-1, MXT[®]-1HT, MXT[®]-1 SimDist, MXT[®]-2887, MXT[®]-500, MXT[®]-Biodiesel TG Rtx[®]-1, Rtx[®]-5, Rtx[®]5MS, Rtx[®]-5Amine, Rtx[®]-440, Rtx[®]-1614, Rtx[®]-2887, Rtx[®]-G27, Rtx[®]-Biodiesel, Rtx[®]-DHA (PONA), Rtx[®]-Mineral Oil, Rtx[®]-PCB, Rtx[®]-TNT, Rtx[®]-TNT2 Rxi[®]-1ms, Rxi[®]-5ms, Rxi[®]-5Sil MS, Rxi[®]-XLB, Rxi[®]17, Rxi[®]-17Sil MS, Rxi[®]-35Sil MS

Column Length (meters)	Film Thickness (µm)	Approx	Approx. Time	
		(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°-20, MXT°-35, MXT°-50, MXT°-65, MXT°-65TG, MXT°-200, MXT°-502.2, MXT°-624, MXT°-1301, MXT°-1701, MXT°-Volatiles

Rtx[®]-20, Rtx[®]-35, Rtx[®]-35 Amine, Rtx[®]-50, Rtx[®]-65, Rtx[®]-65TG, Rtx[®]200, Rtx[®]-200MS, Rtx[®]-502.2, Rtx[®]-624, Rtx[®]1301, Rtx[®]-1701, Rtx[®]-BAC Plus 1, Rtx[®]-BAC Plus 2, Rtx[®]-CLPesticides, Rtx[®]-CLPesticides2, Rtx[®]-Dioxin, Rtx[®]-Dioxin2, Rtx[®]-G43, Rtx[®]-OPPesticides, Rtx[®]-OPPesticides2, Rtx[®]-VMS, Rtx[®]-Volatiles, Rtx[®]-VRX Rxi[®]-624Sil MS

 $Stx^{*}\text{-}CLPesticides,\ Stx^{*}\text{-}CLPesticides2$

Column Length (meters)	Film Thickness (µm)	Approx. Time	
		(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
<u>></u> 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	Approx. Time	
		(min)	(hr)	
<u><</u> 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	
<u>></u> 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	