EPA Method 16B GC configuration

November 2013

The SRI Modeod 16B GC configuration incorporates a FPD/FID combo detector plus a modified DELCD reactor to convert all sulfur species injected into SO2.

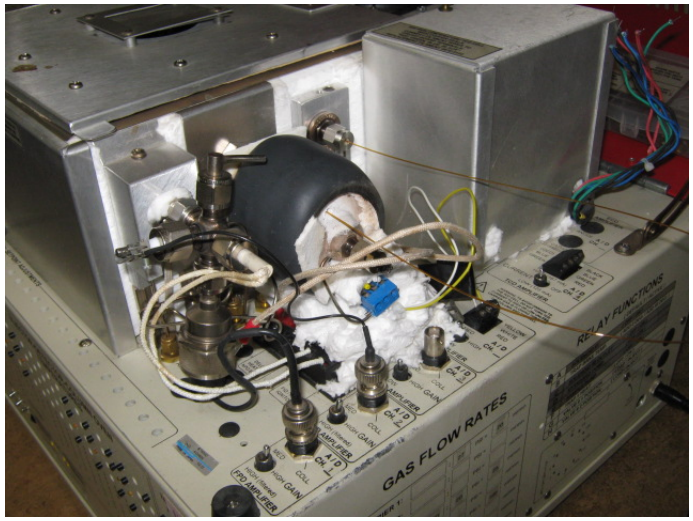
The modified DELCD reactor can reach temperatures of 1000C, and is constructed so that a tube such as the .53mm fused silica tube shown in the photo can pass through the hottest portion of the reactor.

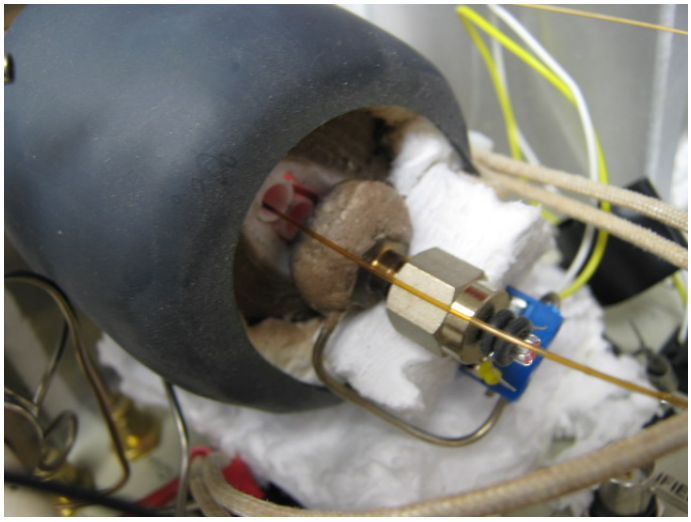
Sulfur species such as H2S COS, DMS and others are converted by the reactor heat into SO2.

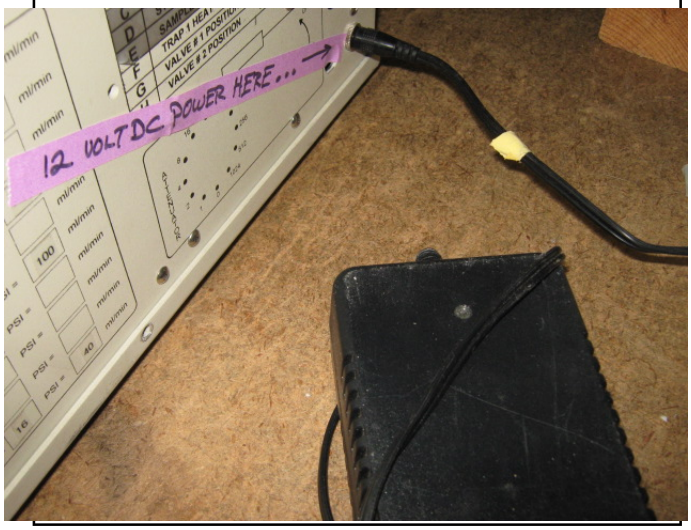
The SO2 is then separated from other compounds like CO2, CO and unreacted sulfurs by the 15meter capillary column. This is to insure that no other compound can interfere with the SO2 measurement.

Heat for the reactor is supplied by an external 12 volt power supply rated at 80 watts.

Plug it in on the right side of the GC.



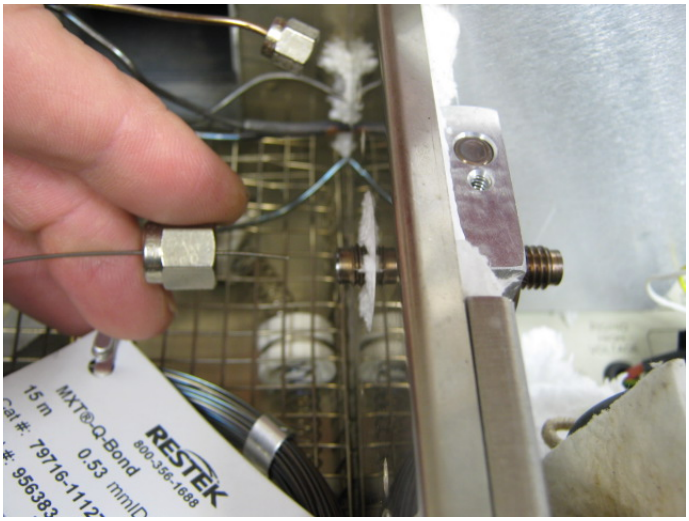




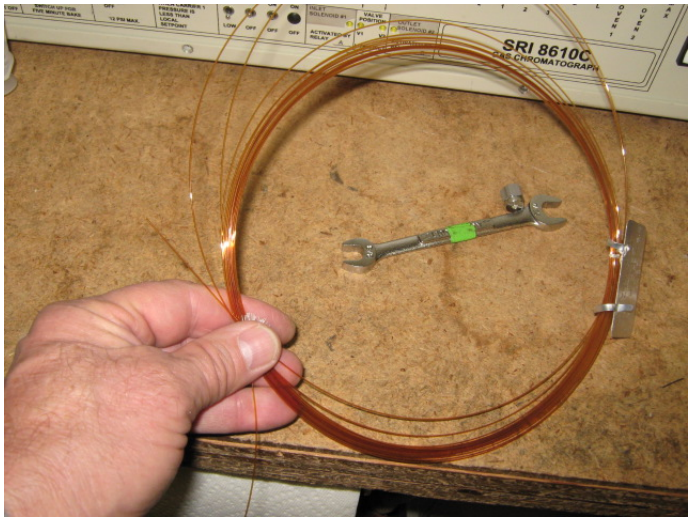
EPA Method 16B GC configuration

November 2013

The column is connected to a bulk-head fitting very much like the on-column injector except that the bulk-head fitting is located on the right side of the column oven.

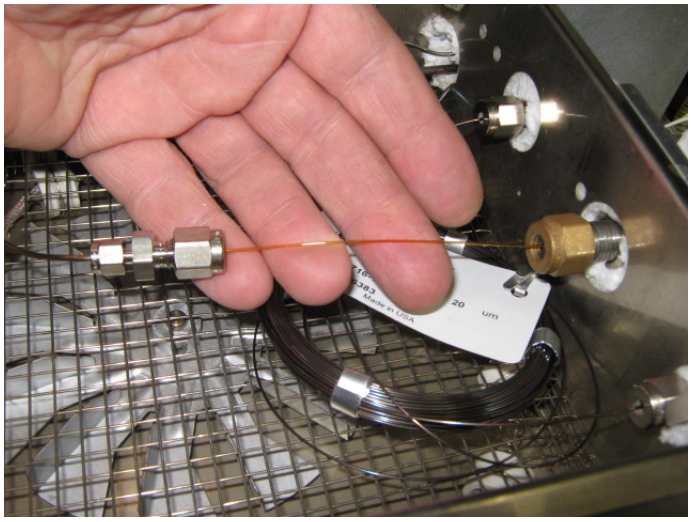


1meter of fused silica capillary tubing (FS)is then cut from a roll of pre-column or just about any junk column. Here we are using an old RTX-35 column for material.



One end of the FS tubing is connected to the injection valve using a 1/8” nut and graphite reducing ferrule.

The remaining tubing is inserted through the DELCD reactor

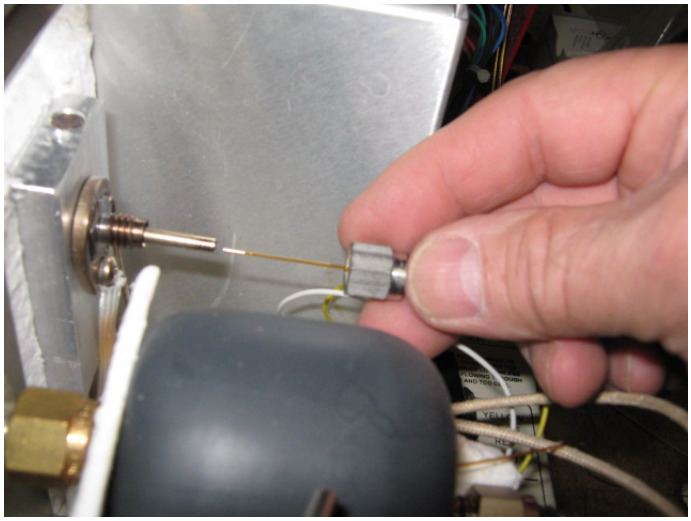


EPA Method 16B GC configuration

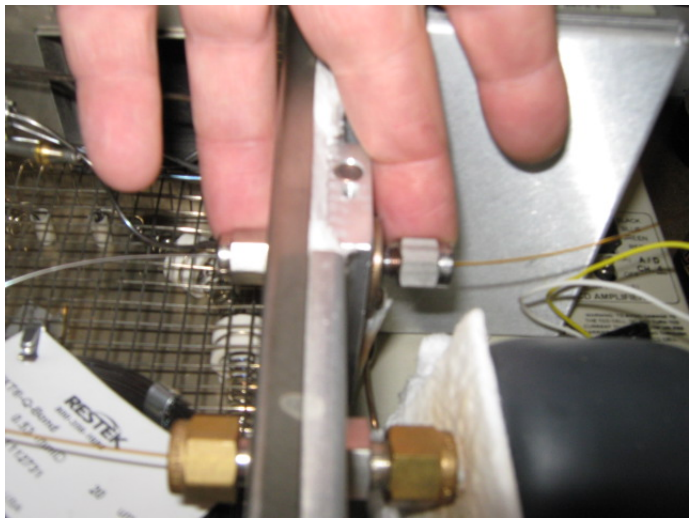
November 2013

As the tubing exits the reactor it will extend about 1 foot and then loop back to connect to the bulkhead fitting and column. This is hard to show in the photo because the tubing is so thin its hard to see.

Connect the FS tubing to the bulkhead fitting using the capillary adapter which aligns the FS and the column for minimum dead volume.



Tighten the graphite ferrules on both sides of the bulkhead fitting securely.



EPA Method 16B GC configuration

November 2013

Temporarily, remove one of the reactor heater leads to prevent the reactor from heating.

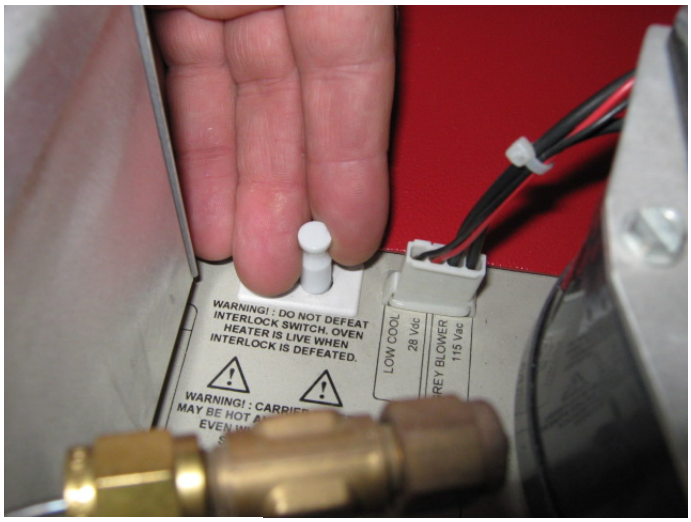
Since the FS tubing is fragile, and closing the GCs red lid might break it, operate the GC with the red lid UP.

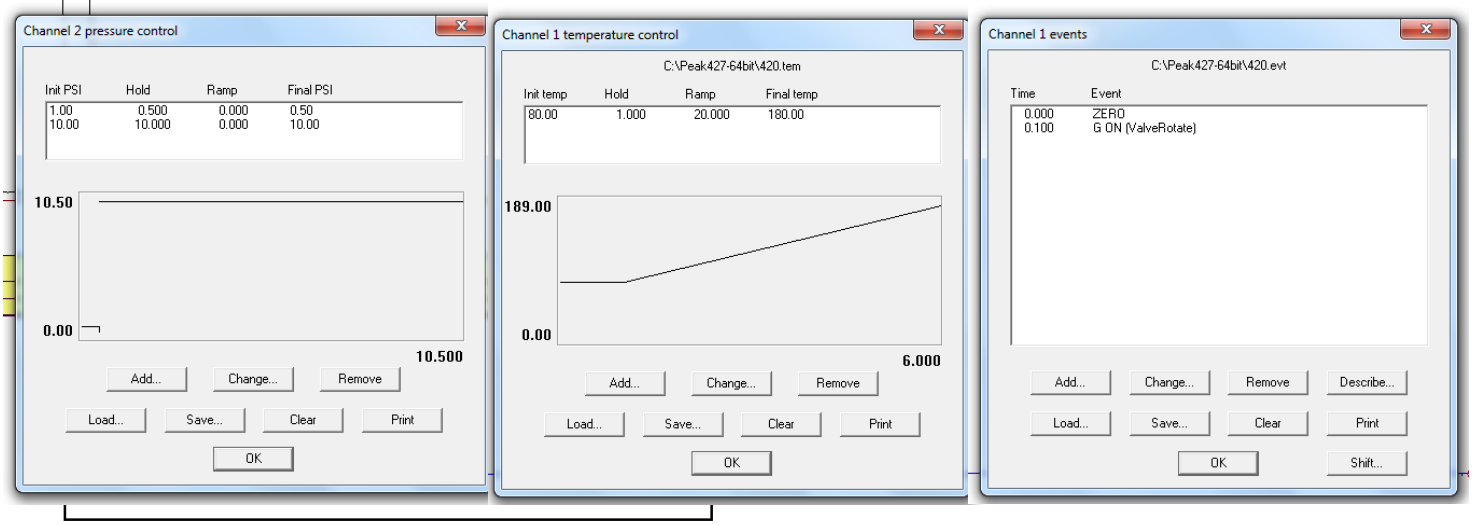
Defeat the interlock switch by pulling up on the white plunger. If you don't do this, the column oven will not heat.

Enter the temperature program and event tables as shown.

Also enter a pressure program in channel 2. Channel 2 must be active.

The pressure program keeps the flow low while the sample is going through the reactor, then speeds the flow up to complete the analysis.

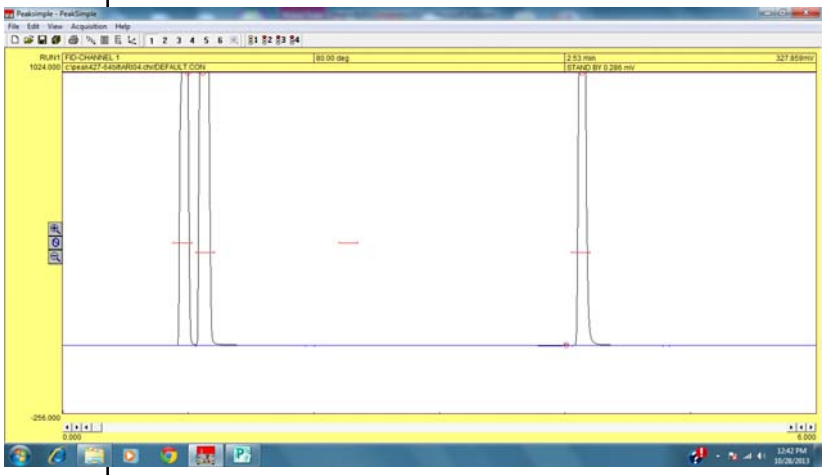




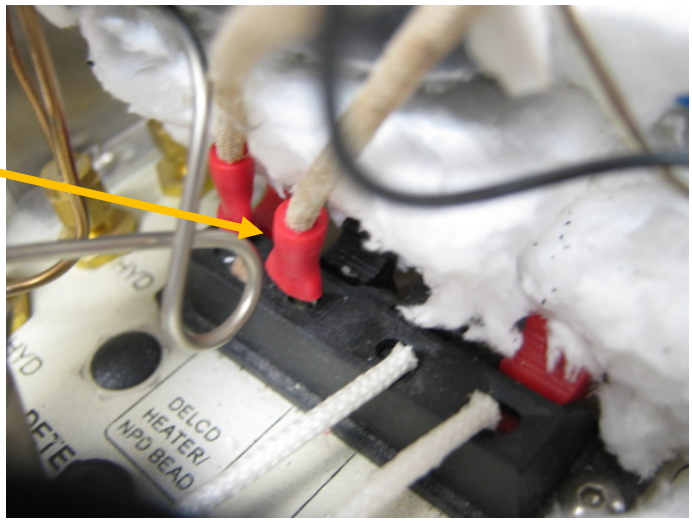
EPA Method 16B GC configuration

November 2013

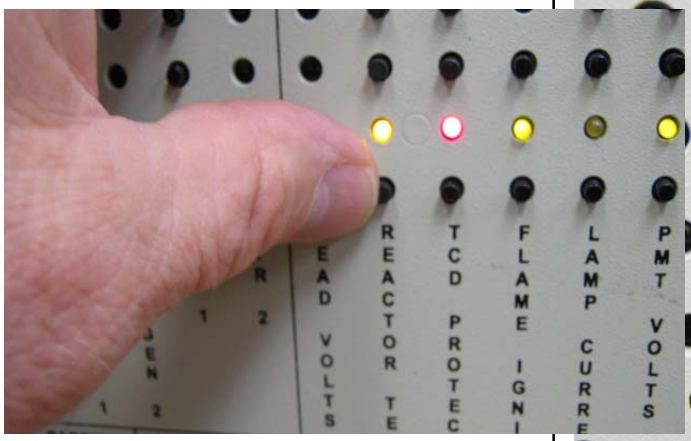
Inject a sample containing other sulfur species. Here we have injected H2S, COS and DMS but no SO2. Identify and calibrate the peaks if necessary. Here we show 40ppm each H2S, COS and DMS (dimethylsulfide).



Plug in the reactor.



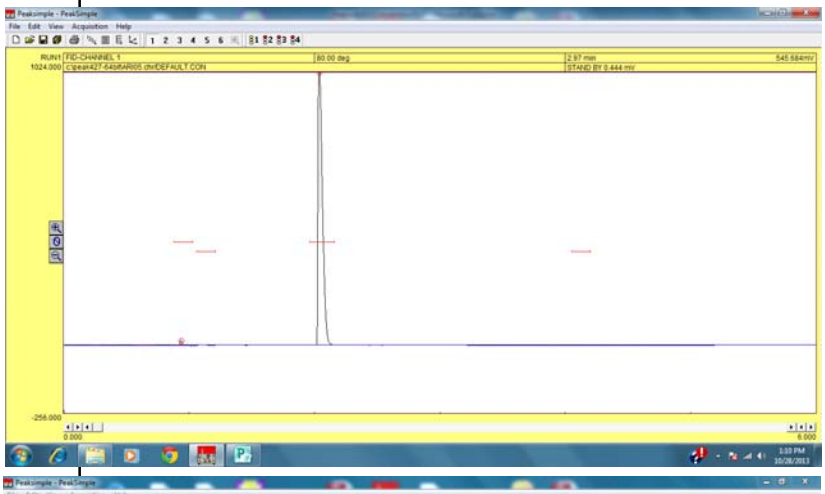
Then verify that the reactor temperature increases to about 260. This will take several minutes. The real temperature of the reactor is about 1000C, as the readout temperature must be multiplied by a factor of four.



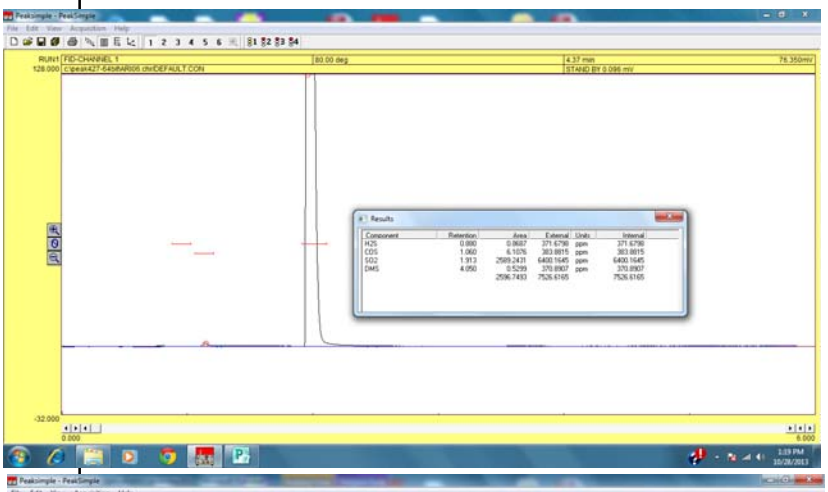
EPA Method 16B GC configuration

November 2013

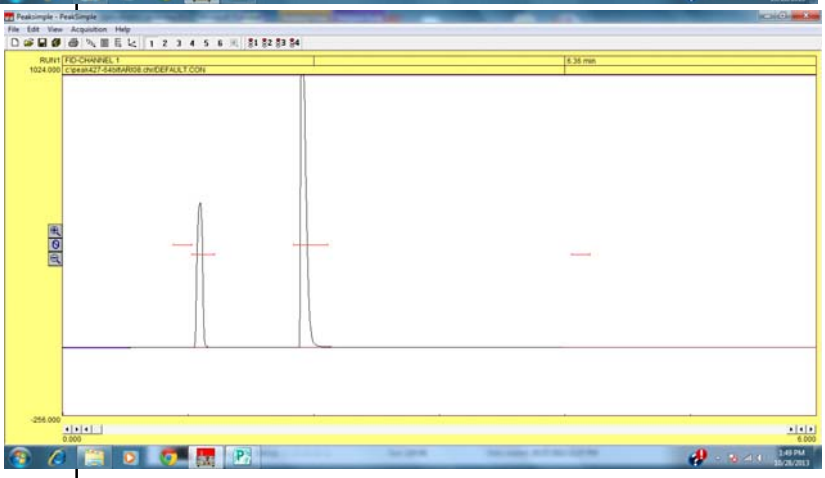
The chromatogram at right shows the same sample (40ppm each H2S, COS and SO2) with the reactor hot (260). The H2S, COS and DMS have all disappeared and a SO2 peak shows instead.



The small amount of residual unreacted COS is less than 1% of the SO2 peak.



It would be nice to use metal capillary tubing instead of FS because it does not break, but the COS peak residual is larger when metal tubing is used. Probably because it conducts heat more readily and thus runs cooler.



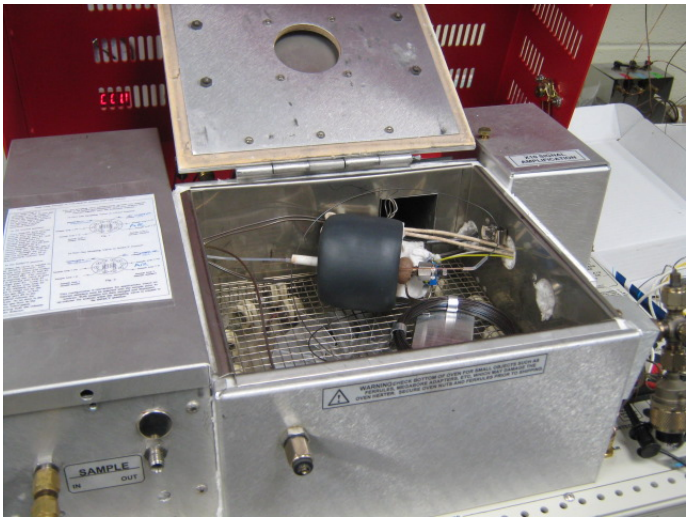
EPA Method 16B GC configuration

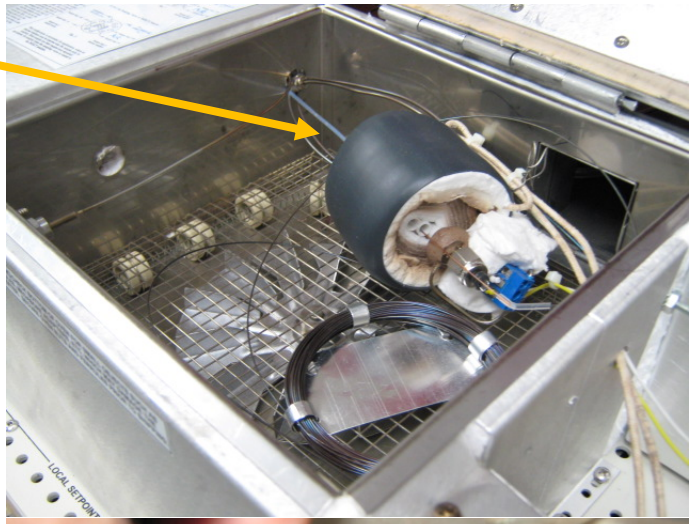
September 2015

In September 2015 we modified the previous Method 16B design by moving the reactor inside the GCs column oven. The previous design with the reactor outside the column oven required a bend in the silica tubing which caused frequent breakage.

The new design eliminates any bending of the silica tubing as the sample exits the valve oven through a Teflon tube which is directly in-line with the reactor.

Another feature of the new design is the use of silicone tubing to connect the Teflon tube to the silica tube, and then also to the column. The weight of even a 1/16" union is enough to stress the silica tubing after the reactor heat burns off the polyimide coating so the silicone tube union is better because of its very low mass.



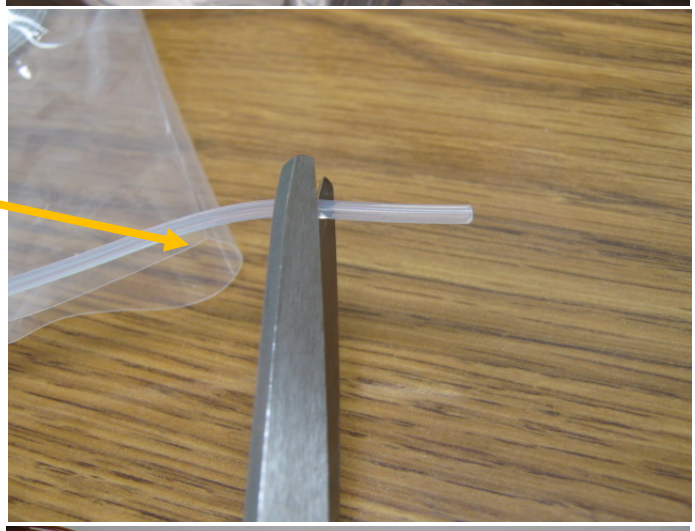


EPA Method 16B GC configuration

September 2015

Use silicone tubing like shown at right. Note that the inside diameter is .020", so it stretches and fits tightly on .53mm (.030") tubing (the fused silica tubing which goes through the reactor). It can also stretch over 1/16" (.062") Teflon tubing.

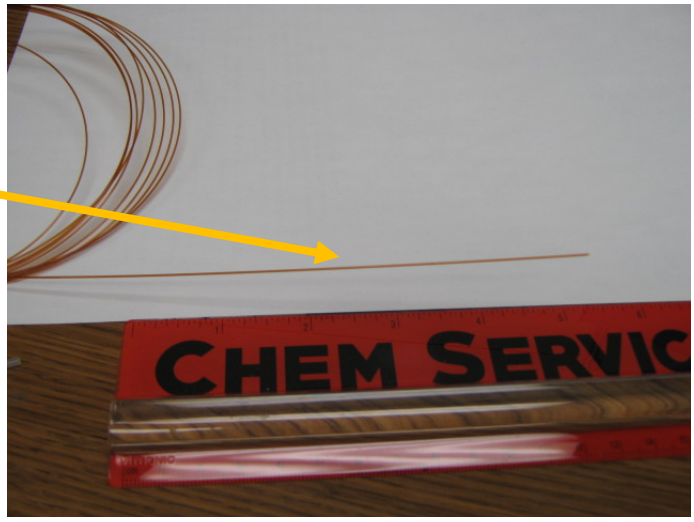
Cut two 1/2" pieces of the silicone tubing.



Cut a 5" piece of the silca tubing.

You can use an old column since any stationary phase will burn off once the reactor heats up.

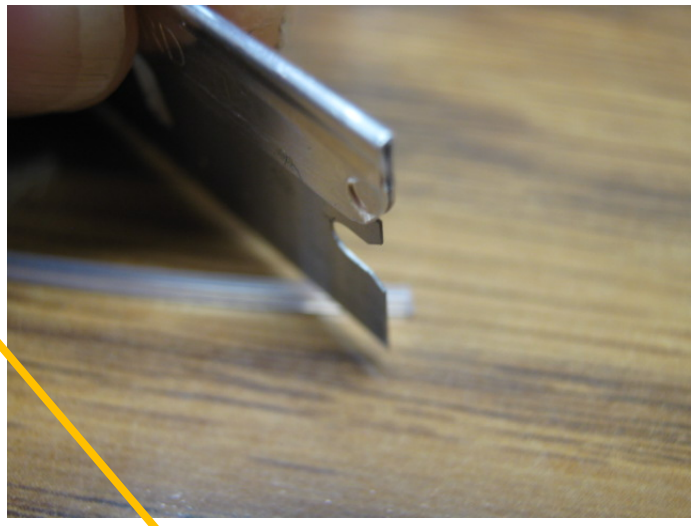
We have to use the silca tubing be-cause of the 1000C temperature in the reactor. Any other material would melt.



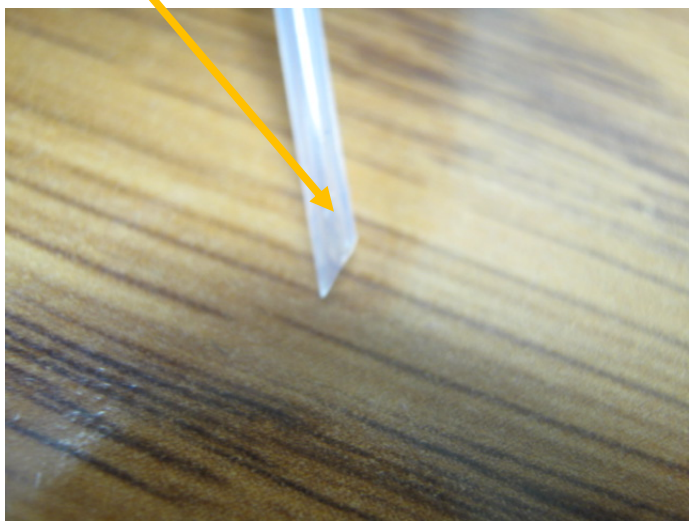
EPA Method 16B GC configuration

September 2015

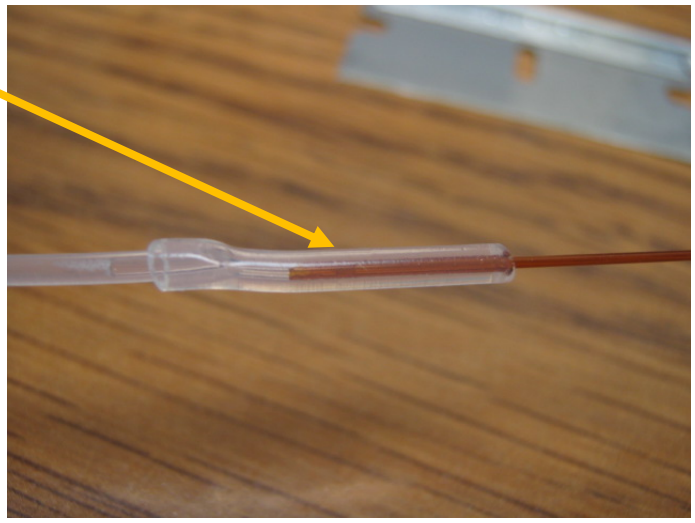
Use a razor blade to cut the Teflon tubing (coming from the gas samping valve) at a 45 degree angle.



The angle makes it easier to wiggle the silicone over the Teflon. It also helps to lubricate the Teflon with a little saliva. It may help to hold the Teflon tubing with a small piece of sandpaper if it slips in your fingers.



Assemble the union between the Teflon tubing and the silica tubing as shown

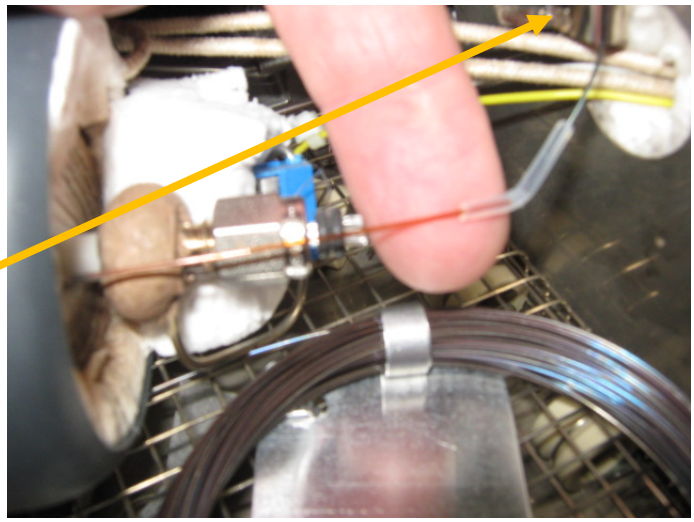


EPA Method 16B GC configuration

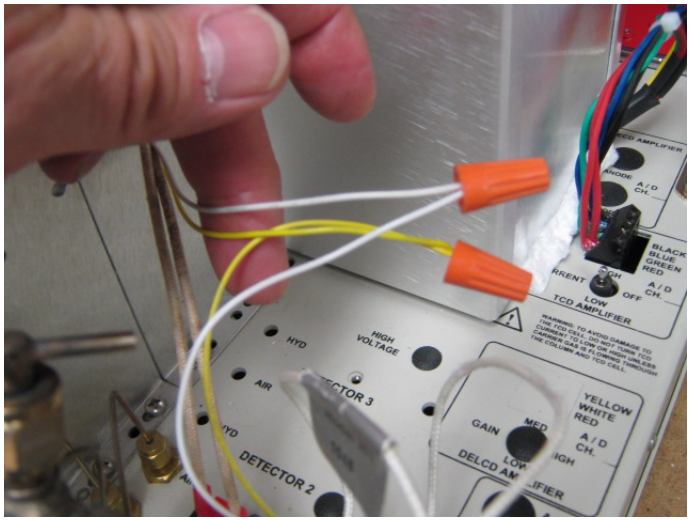
September 2015

On the downstream side of the reactor assemble the union to connect the silica tubing to the .53mm column.

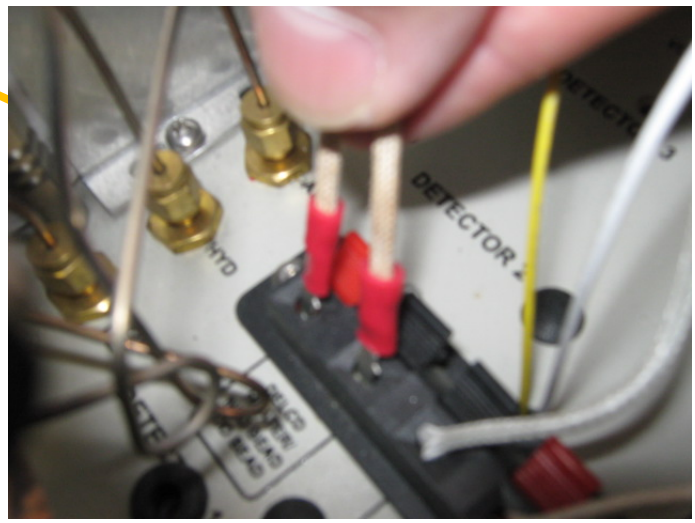
There is a little clip mounted in the oven which helps hold the column in a steady position. Bend the column gently to align the column with the silica tubing. If the column is not well aligned, the stress may break the silica tube once the coating burns off.



Connect the reactor's thermo-couple wires (white and yellow) with the same color wires on the right side of the column oven.



Connect the reactor heat wires to the terminal board on the right side of the oven.



EPA Method 16B GC configuration

September 2015

One other change on the new design is to modify the temperature program as shown.

The OVEN MAX setting on the GC is set to 130, so the initial oven setpoint of 150 causes the Thermo-couple Out of Range Alarm (TORA) to disable the oven heater and fan. Because the reactor is located in the column oven, the fan cools the reactor and prevents it from reaching its 242 set-point. By tripping the TORA, the oven fan is off at the beginning of the analysis (which is when the reactor needs to be at its hottest). Once the sample passes through the hot reactor (.5 minutes), then the normal oven program takes over, the oven heater is energized, and the fan spins.