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.

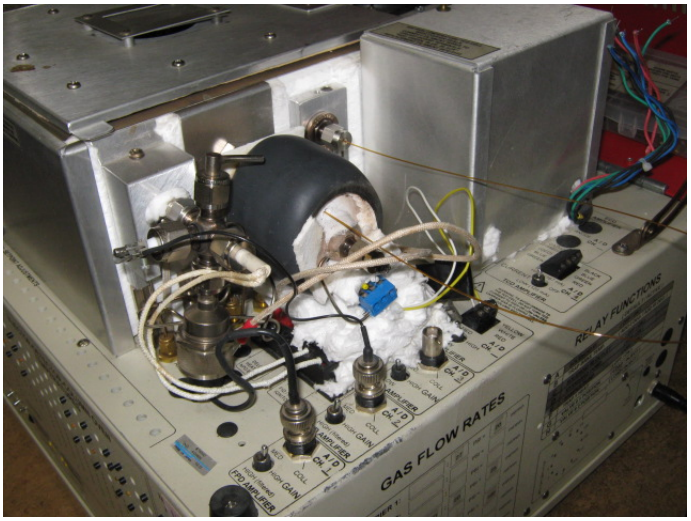
The modified DELCD reactor can reach temperatures of 1000C, and is constructed so that a tube such as the .53mm fused silica tube shown in the photo can pass through the hottest portion of the reactor.

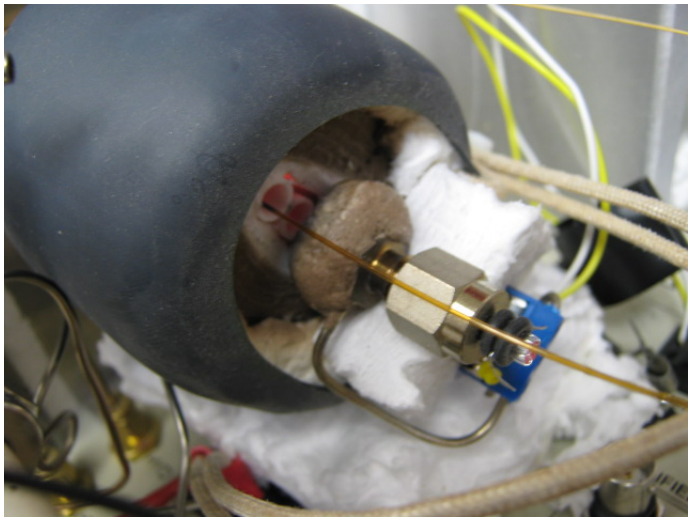
Sulfur species such as H2S COS, DMS and others are converted by the reactor heat into SO2.

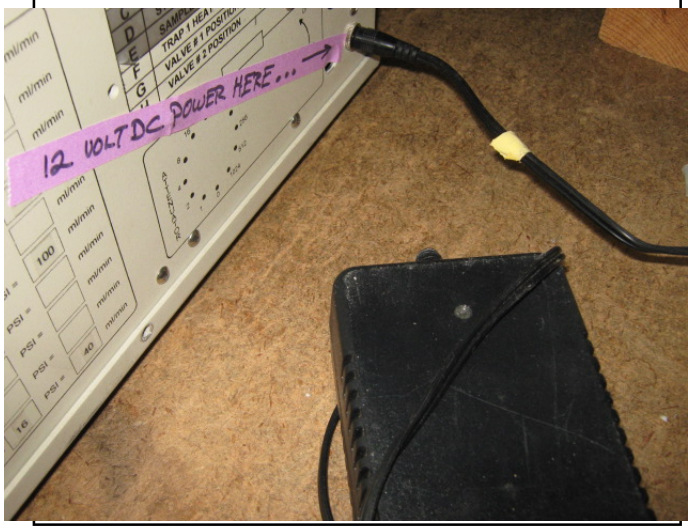
The SO2 is then separated from other compounds like CO2, CO and unreacted sulfurs by the 15meter capillary column. This is to insure that no other compound can interfere with the SO2 measurement.

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.







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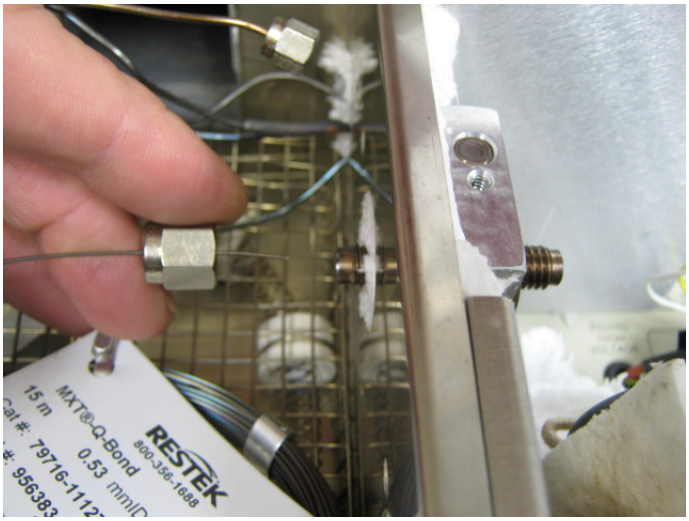
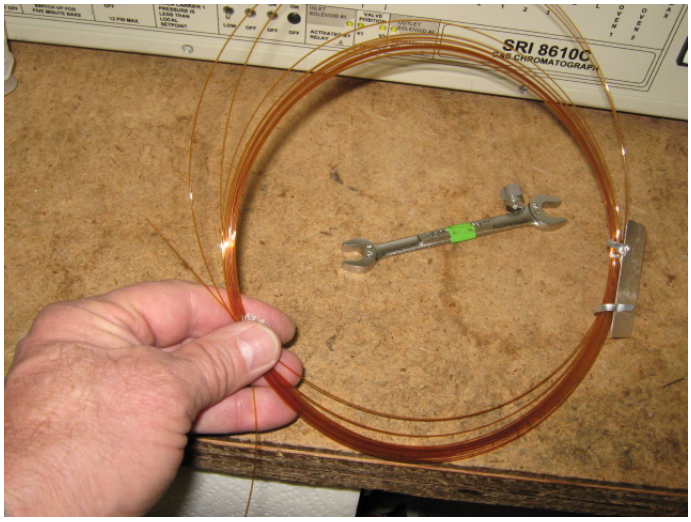
The column is connected to a bulk-head fitting very much like the on-column injector except that the bulk-head fitting is located on the right side of the column oven.

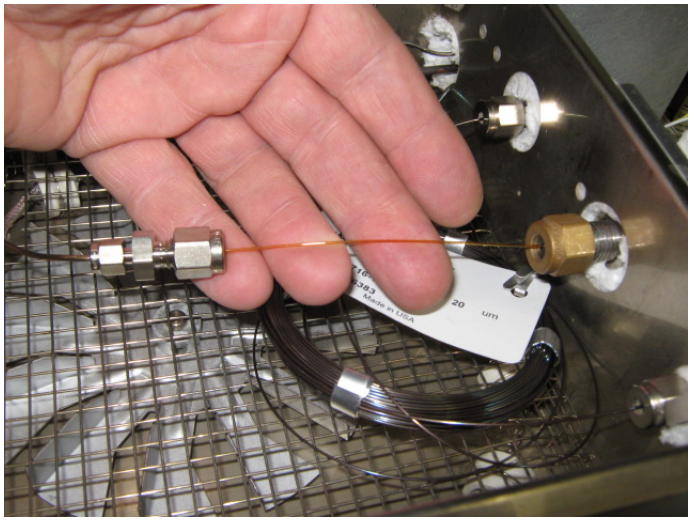


1meter of fused silica capillary tubing (FS)is then cut from a roll of pre-column or just about any junk column. Here we are using an old RTX-35 column for material.



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



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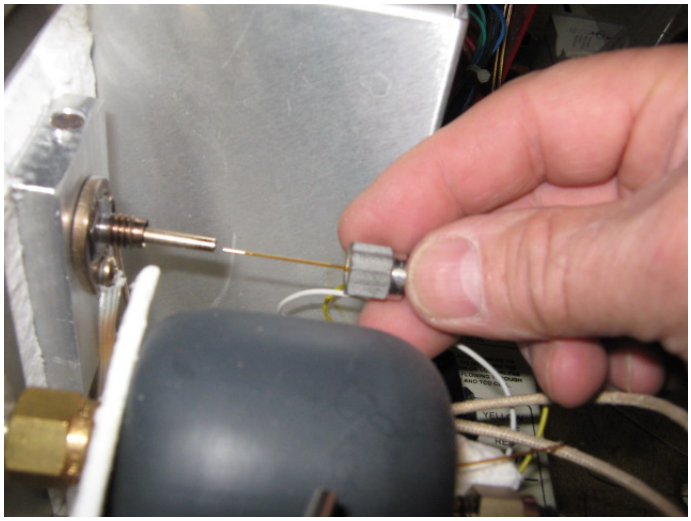
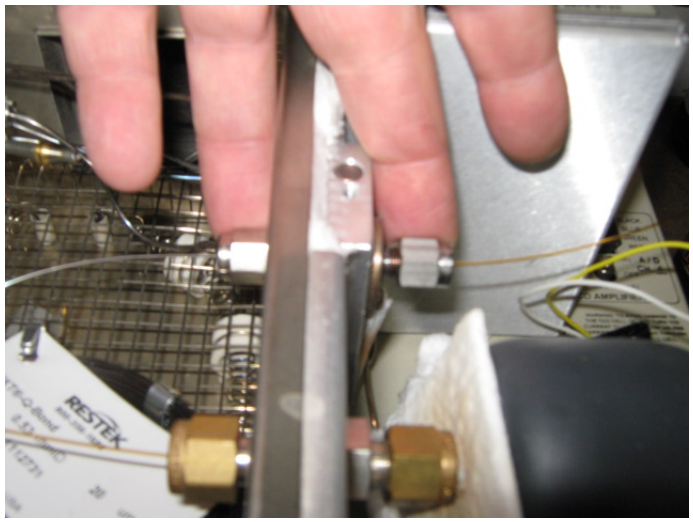
As the tubing exits the reactor it will extend about 1 foot and then loop back to connect to the bulkhead fitting and column. This is hard to show in the photo because the tubing is so thin its hard to see.

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.



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

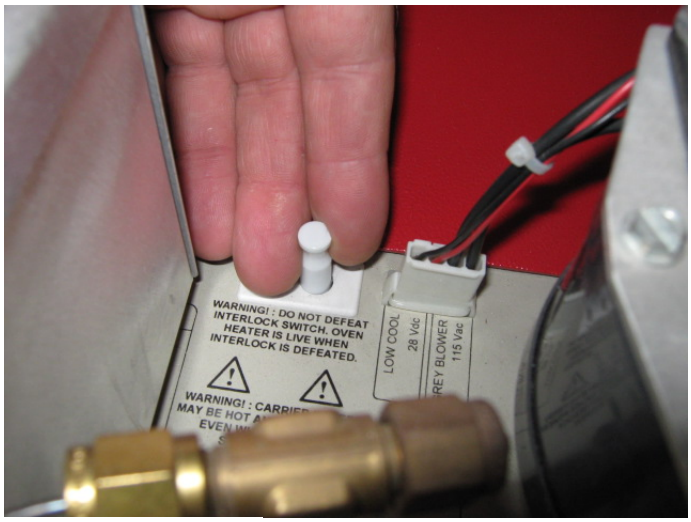
Since the FS tubing is fragile, and closing the GCs red lid might break it, operate the GC with the red lid UP.

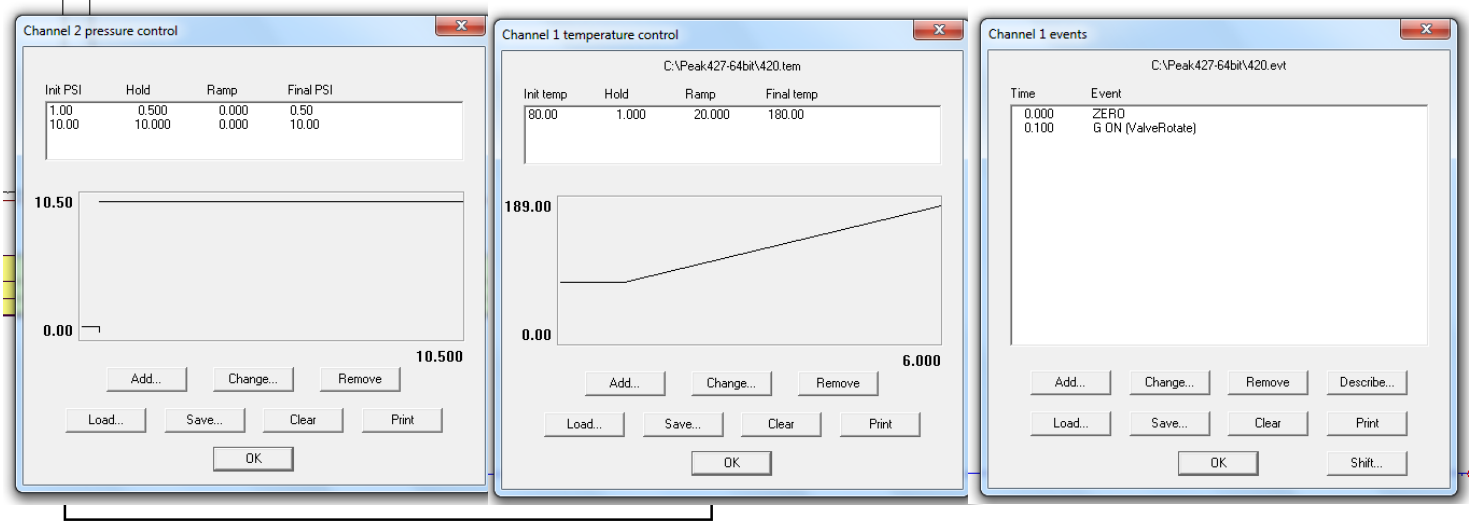
Defeat the interlock switch by pulling up on the white plunger. If you don't do this, the column oven will not heat.

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.





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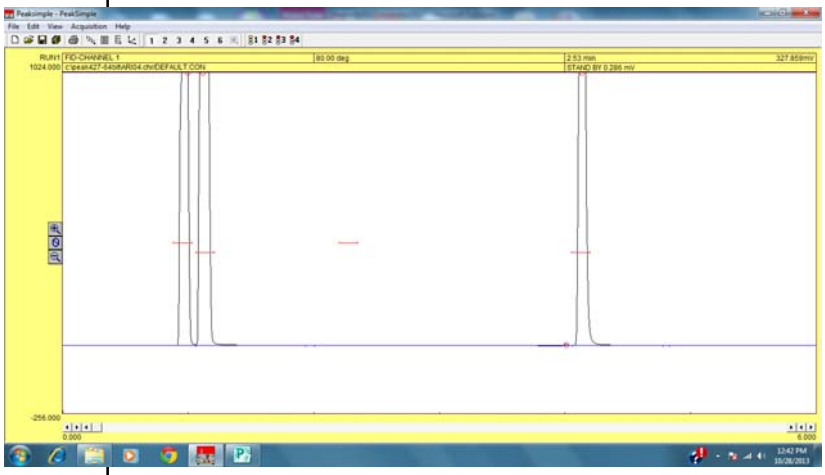
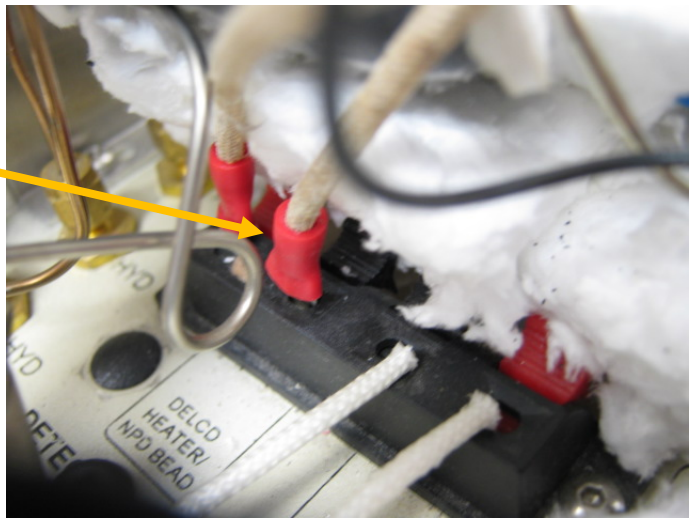
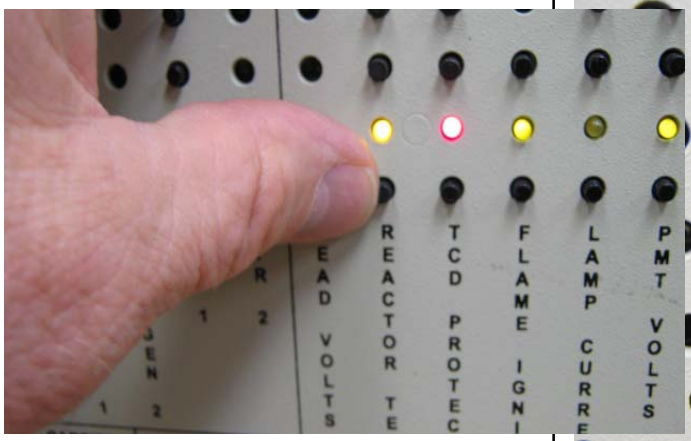
Inject a sample containing other sulfur species. Here we have injected H2S, COS and DMS but no SO2. Identify and calibrate the peaks if necessary. Here we show 40ppm each H2S, COS and DMS (dimethylsulfide).



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.



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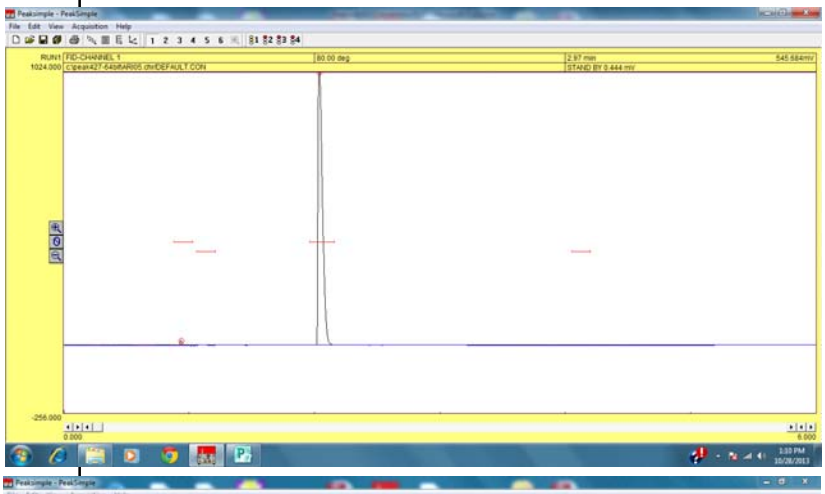
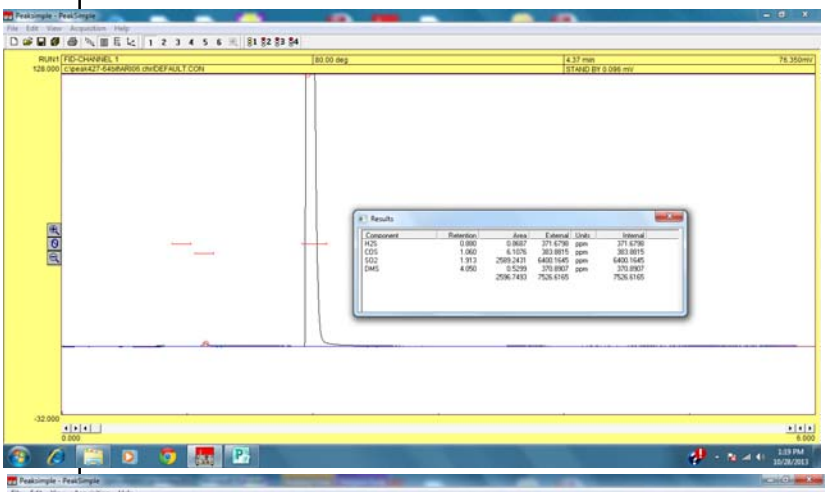
The chromatogram at right shows the same sample (40ppm each H2S, COS and SO2) with the reactor hot (260). The H2S, COS and DMS have all disappeared and a SO2 peak shows instead.



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.