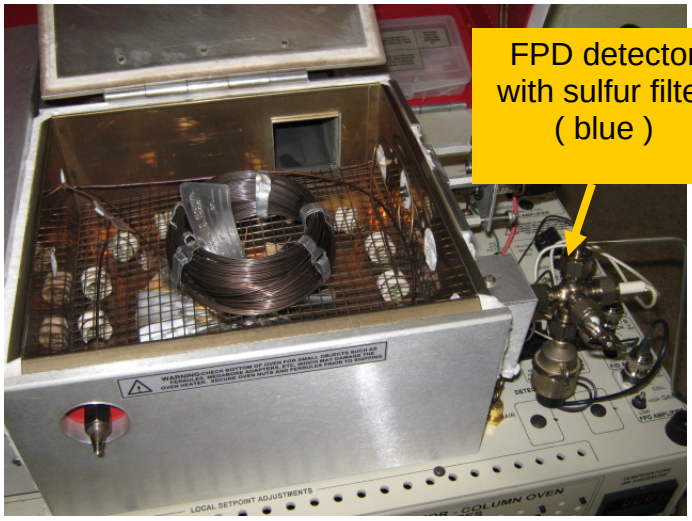
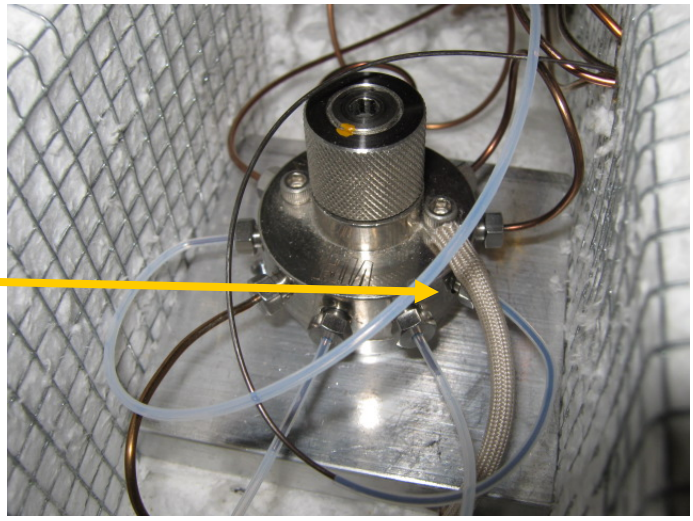
Sulfur Trapping at low PPB levels using the SRI 8610C Gas Chromatograph

The SRI 8610C GC is shown at right. Note the software controlled vacuum pump, tedlar bag and H2S sample cylinder.

A 60 meter MXT 1 metal capillary column is located in the column oven. The column has a 5 micron film thickness which is necessary to separate the Air and Methane peaks from the H2S peak. The FPD detector is located to the right of the column oven.



A Valco 10 port gas sampling valve (GSV) is located in the valve oven. Note that all of the tubes which are in the sample path are 1/16” Teflon with .030”id. Also note that the MXT 1 column is connected directly to port 9 of the GSV.



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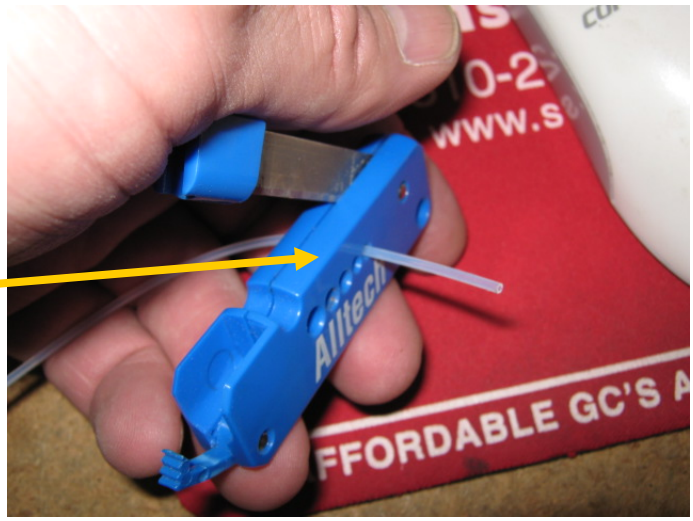
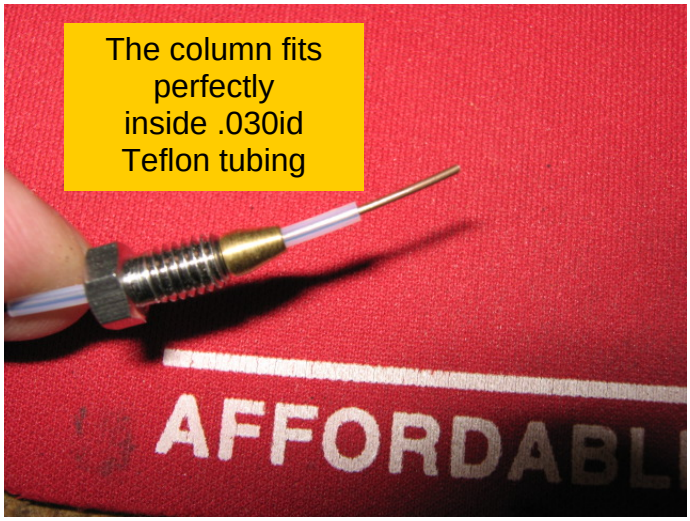
Sulfur analysis at the ppb level is difficult because sulfur sticks to many surfaces (adsorption). For this reason only Teflon or other extremely inert tubing can be used.

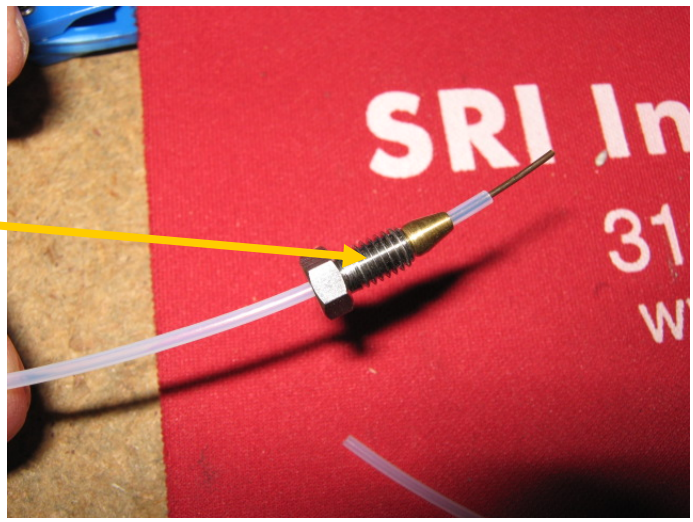
SRI uses 1/16” Teflon tubing with .030” id. This photo shows how to cut Teflon tubing clean and square. A razor blade can also be used if you are careful to get a square cut.



To connect the .53mm id MXT column to the Valco valve, a 1” length of Teflon tubing is cut and slid over the end of the column. A regular Valco nut and brass ferrule then secures the column to the valve.



The trap is connected to the Valco valve with longer lengths of Teflon tubing. In this case, a 1” section of column is inserted inside the end of the Teflon tubing to act as a reinforcement or internal stiffener. A regular Valco nut and brass ferrule secures the Teflon tubing in the valve. The short piece of column keeps the Teflon from collapsing when the ferrule is tightened.



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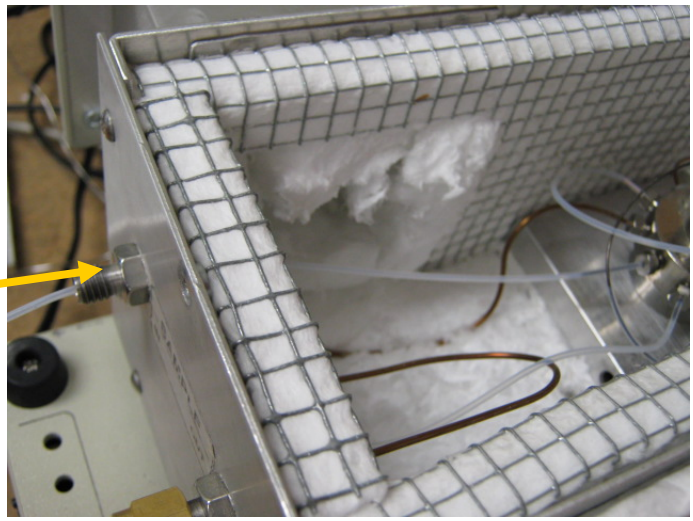
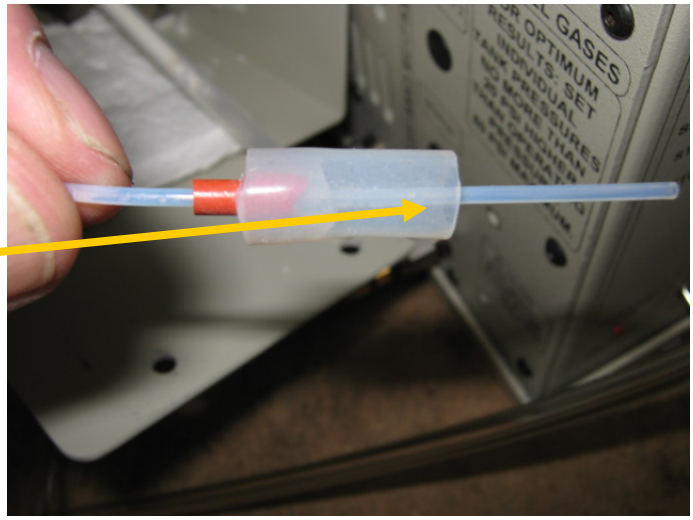
Teflon tubing must also be used to transport the sample into the GC from the Tedlar bag or other sample container. Note how the normal swagelok sample inlet fitting has been drilled out to allow the Teflon tubing to pass through.



Any contact with metal will destroy the sulfur sample, especially H2S.

At the Tedlar bag (or other sample container) it is important that the Teflon tubing penetrate as far into the bag as possible.

SRI uses different sizes of silicone tubing to make a secure connection to the bag. This photo shows the 1/16” od Teflon tubing with a little bit of 1/16” id silicone tubing (red color) slid over it. The red silicone tubing is then slid inside 1/8” id silicone and this is in turn slid inside 3/16” id silicone (both clear color). The end of the Teflon tubing sticks out past the silicone so it projects well into the valve on the Tedlar bag. Sample should not come into contact with the silicone tubing or it will be absorbed.



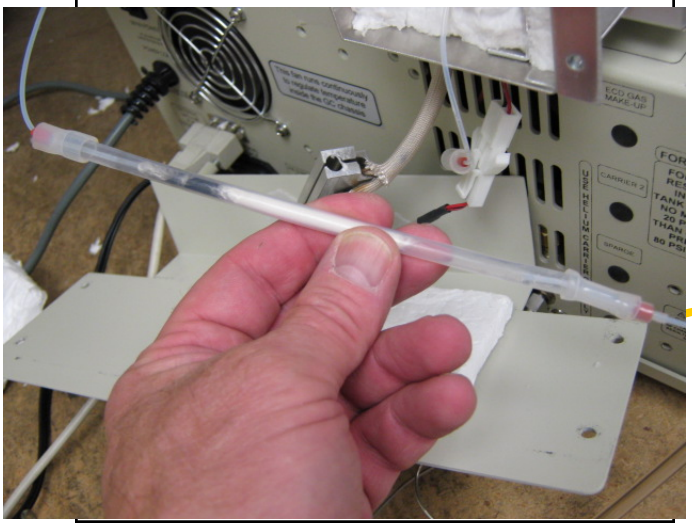
Sulfur Trapping at low PPB levels using the SRI 8610C Gas Chromatograph

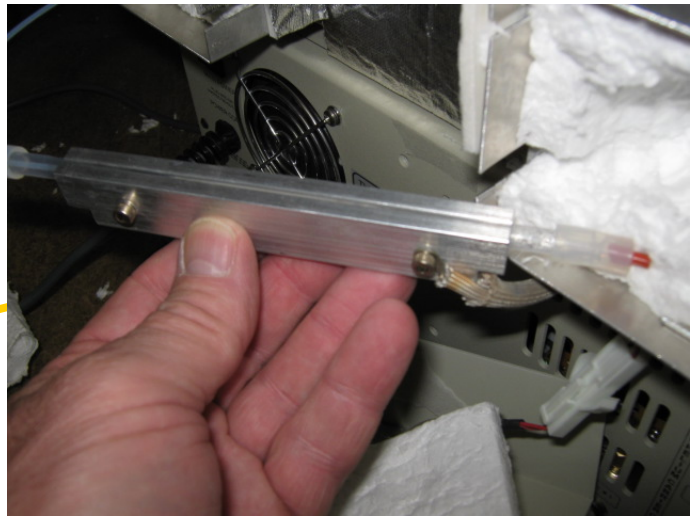
The trap which pre-concentrates the sulfur molecules is located between the ducts of the valve oven. Remove the cover by loosening the four brass thumbscrews.

The 1/4” od Teflon trap tube is secured inside an aluminum heater block.

The trap tube is normally packed with 80-100 mesh silica gel adsorbent, but can be packed with other materials.

Notice that the Teflon tubes leading from the trap to the valve are connected using the silicone tubing and that the end of the Teflon tubing projects well into the trap right up to the packing material.



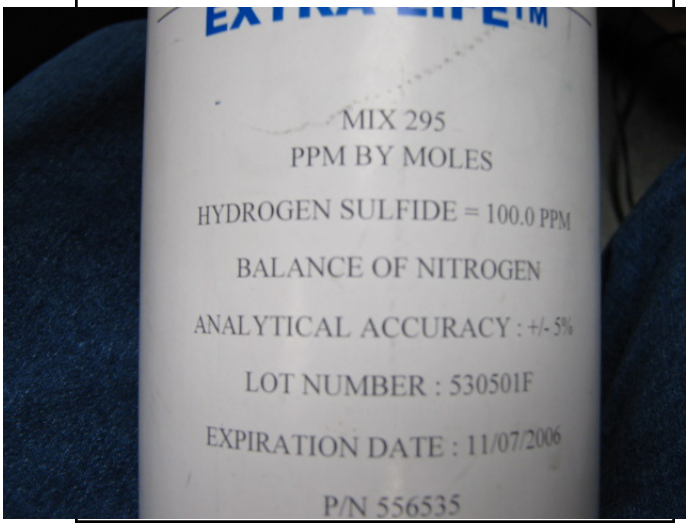


Sulfur Trapping at low PPB levels using the SRI 8610C Gas Chromatograph

To prepare a calibration sample, use a vacuum pump to deflate and then re-inflate a Tedlar bag with room air.

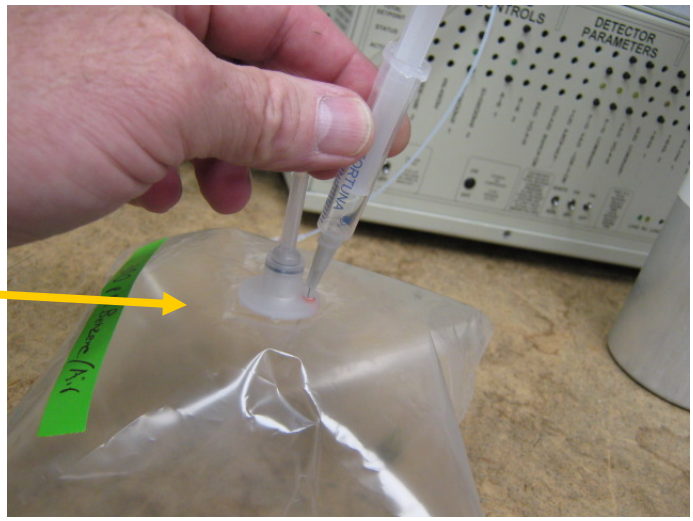
You can also re-inflate the bag with Nitrogen or other inert gas.

Load the sample into a polypropylene syringe. Fill the syringe multiple times to make sure the syringe is saturated with sulfur (so the syringe itself will not absorb the sulfur). In this case the sample cylinder contains 100ppm H2S in Nitrogen.

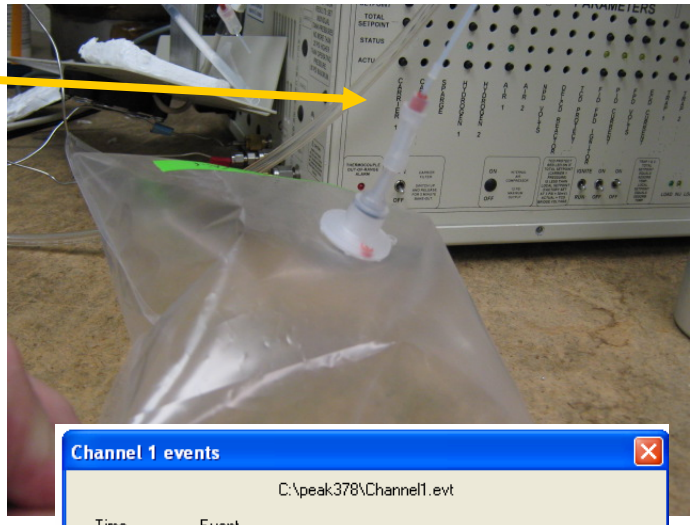


Sulfur Trapping at low PPB levels using the SRI 8610C Gas Chromatograph

Inject the H2S standard mixture into the Tedlar bag through the septum in the bag. In this case we are injecting 1ml of 100ppm H2S into 1000ml (1 liter) of air in the bag. This results in a dilution of 1000 times so the resulting concentration of H2S in the bag is 100 ppb (parts per billion).
Add any other sulfur molecules in the same way.

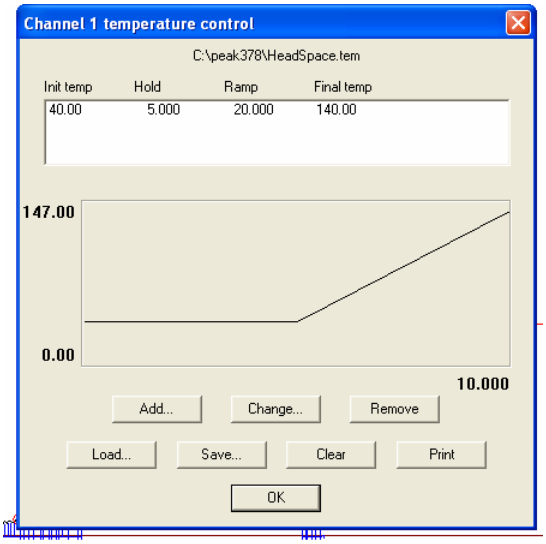


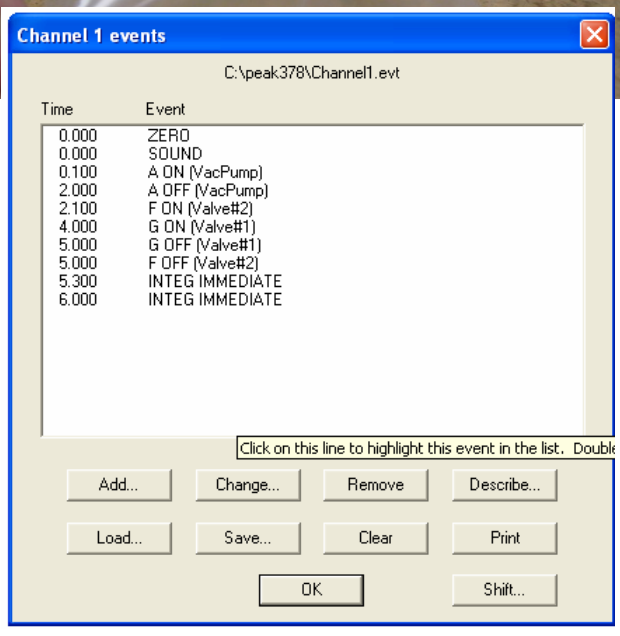
Then connect the bag to the Teflon sample inlet tube leading to the GC.



Immediately (don't wait too long) Start the GC analysis.

The temperature program and event table used for this example are shown below.





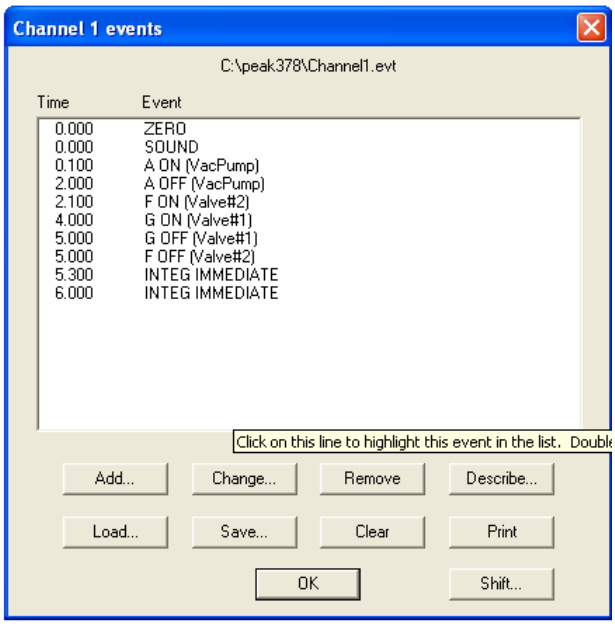
Sulfur Trapping at low PPB levels using the SRI 8610C Gas Chromatograph

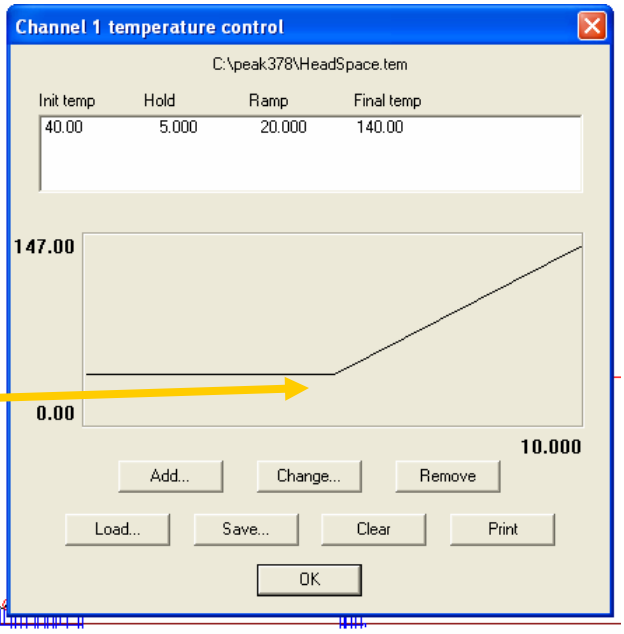
The event table has Relay A (the vacuum pump) turned on from .1 minutes to 2 minutes so the vacuum pump sucks the sample in the Tedlar bag through the trap for 1.9 minutes at the beginning of the analysis. The trap is at room temperature (25C).

The actual flow depends on the strength of the vacuum pump and on the restrictiveness of the trap. The flow is reproducible from run to run however.

Typically the flow rate will be about 300ml/minute, so almost 600ml of sample from the bag will be sucked through the trap in 1.9 minutes.
At 2.10 minutes the trap starts to heat up (Relay F on). At 4.00 minutes the Valco valve rotates to the load position (Relay G on). By this time the trap is hot (180C) so the sulfur molecules are easily flushed from the trap onto the column.

The column temperature remains at 40C until the H2S peak has eluted (5 minutes) then ramps up to clean out the column and elute any other sulfur molecules with higher boiling points (CS2 etc).





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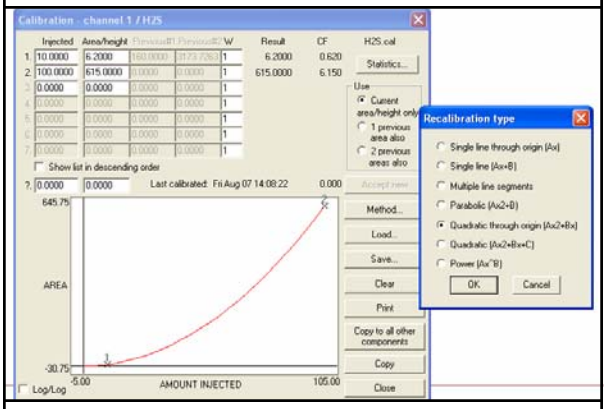
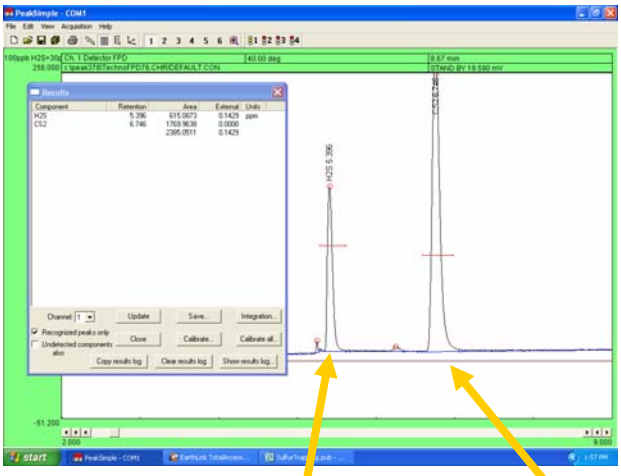
The top chromatogram shows H2S at 100ppb and CS2 at 30ppb.

The CS2 peak is bigger than the H2S peak because the CS2 traps better than the H2S and has two sulfur atoms per molecule rather than one. In general, the higher the sulfur molecule's boiling point, the better it traps.

The second chromatogram shows H2S at 50ppb. Note that the area is 1/4th as big as the area for the 100ppb peak. This is normal because the FPD detector responds to the square of the sulfur concentration, so half the amount injected produces 1/4 the area. The calibration curve is quadratic as shown below.

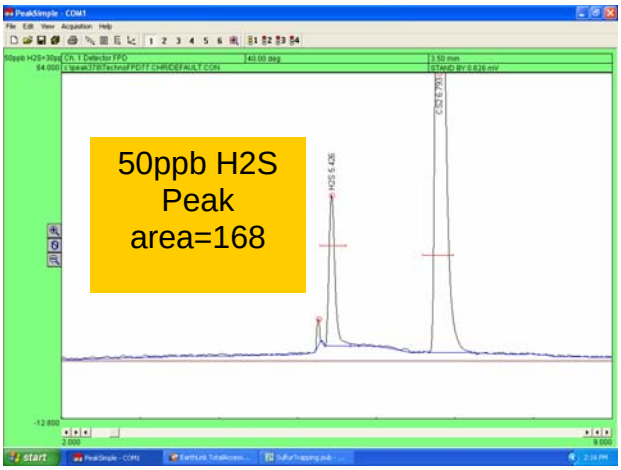
The third chromatogram shows 10ppb H2S again with 30ppb CS2.



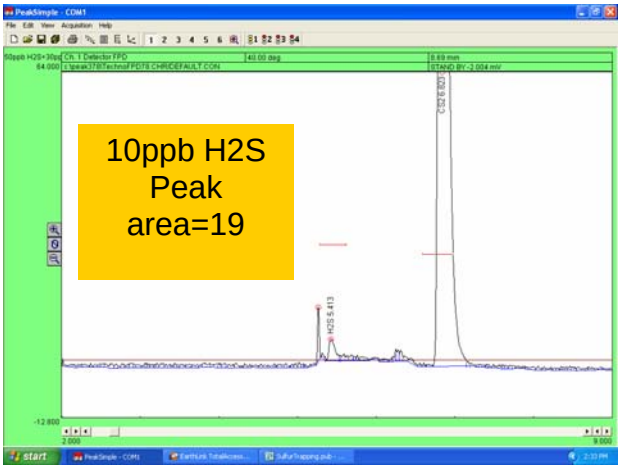


100ppb H2S Peak area=615

30ppb CS2 Peak Area=1769



50ppb H2S Peak area=168



10ppb H2S Peak area=19

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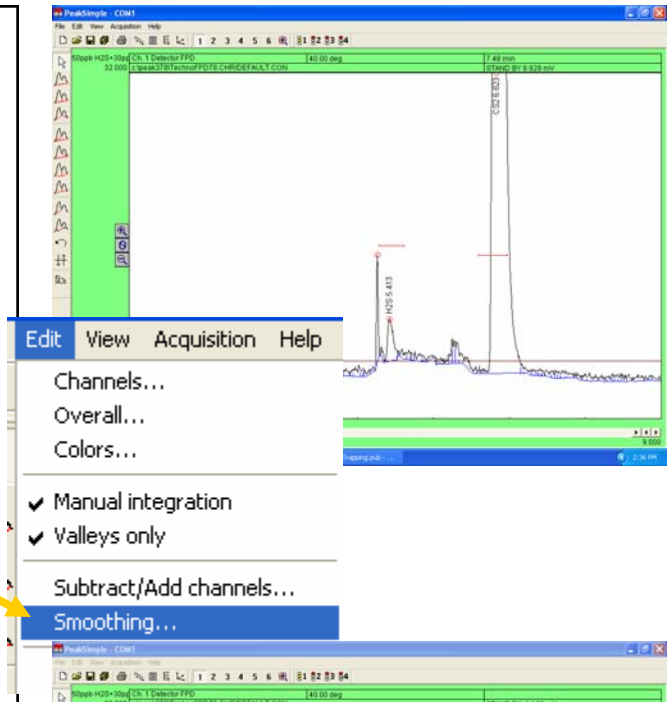
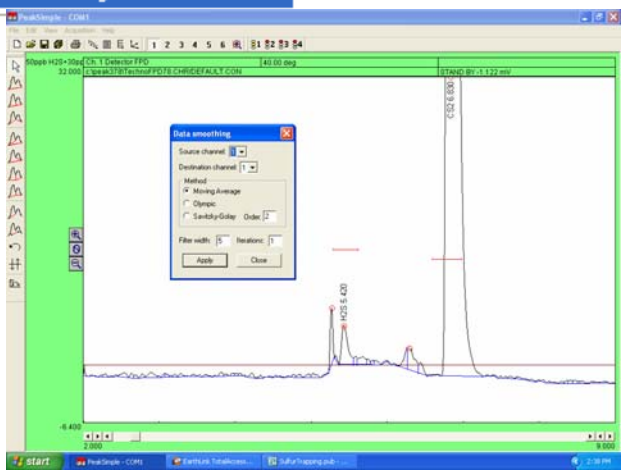
At the 10ppb level, the detector noise on the baseline can make it more difficult to integrate the peak.

It is sometimes helpful to use PeakSimple software's "Smoothing "function.

Click Edit/Smoothing and then specify the parameters for the Smoothing Algorithm.



The Smoothed data makes it easier to determine the start and end of the H2S peak.



It is also important to place the baseline so the air peak which elutes just prior to the H2S does not distort the H2S area. Using the Manual Integration Tools can help with this.