

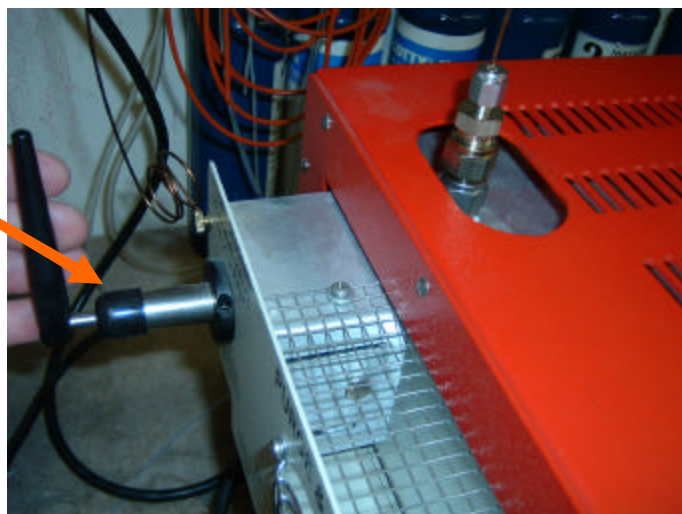
Thermal Soil Desorber Instructions

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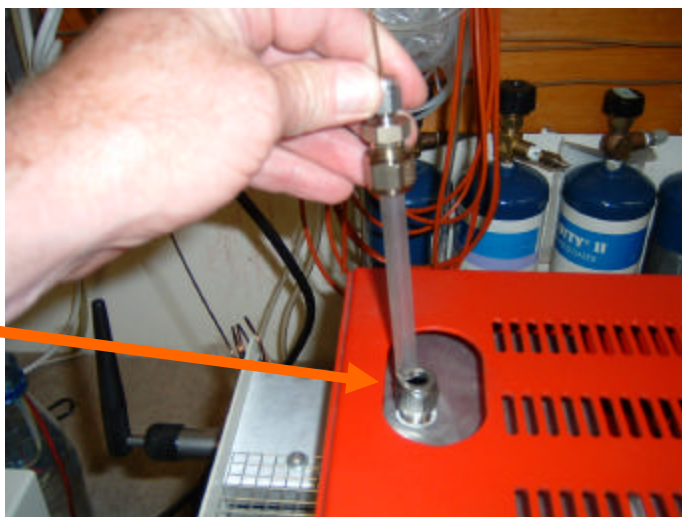
The SRI Thermal Soil Desorber is mounted in the valve oven section of the 8610C GC chassis. The 8610C GC shown in the photo is sitting next to the H2-40XR Hydrogen generator on the benchtop. With both H2 generator and built-in air compressor, the GC can operate *without* compressed gas cylinders.



The Thermal Soil Desorber includes a 10port manually operated Valco valve (equipped with the high temperature "T" rotor) which is heated up to 275C.



A 3/8" diameter ground glass tube (ground to consistent size and roundness) is filled with up to 1 gram of soil. The soil filled tube is then connected to a 3/8" swagelok fitting using a soft graphite ferrule and then inserted into the heater block.



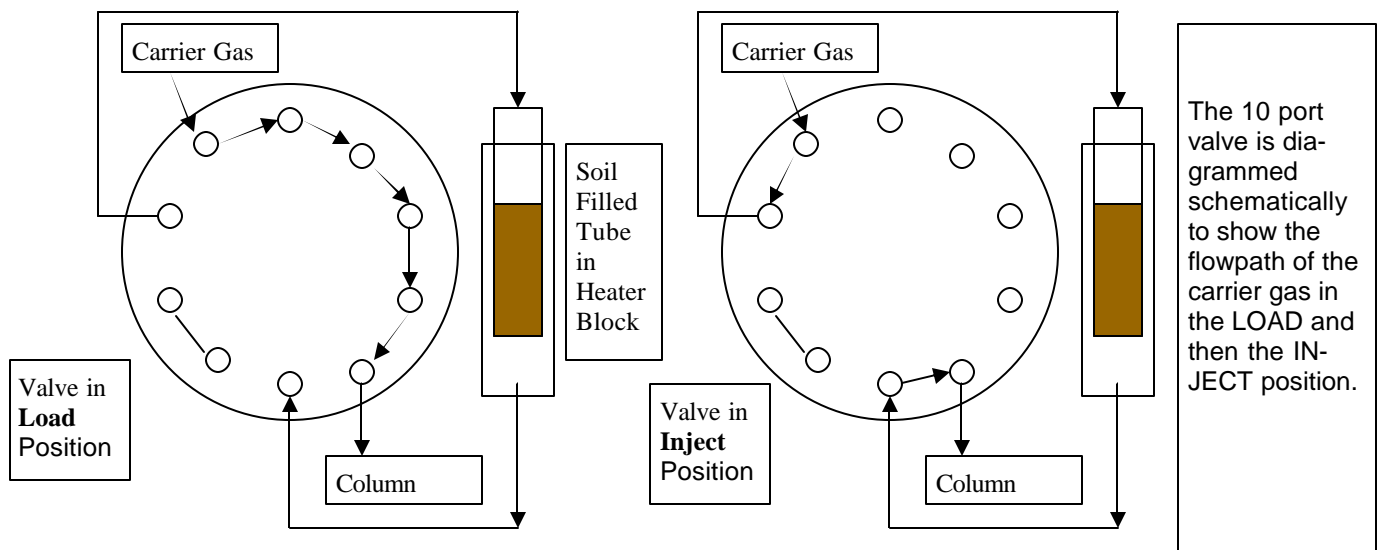
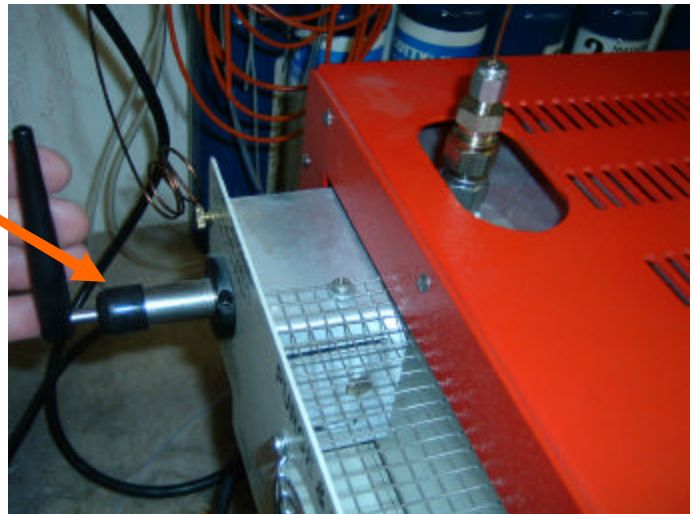
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The soil filled tube is secured into the heater block using a swagelok nut and soft graphite ferrule.



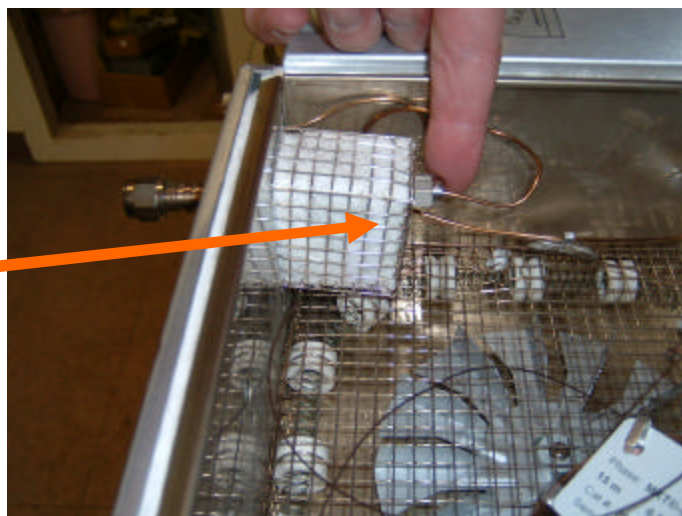
The handle of the valve is *immediately* rotated (at the same time the analysis is started) to redirect the flow of carrier gas through the soil. Any hydrocarbons in the soil (now heated to 275C) are purged into the GC's analytical column where they separate and are detected by the FID detector.



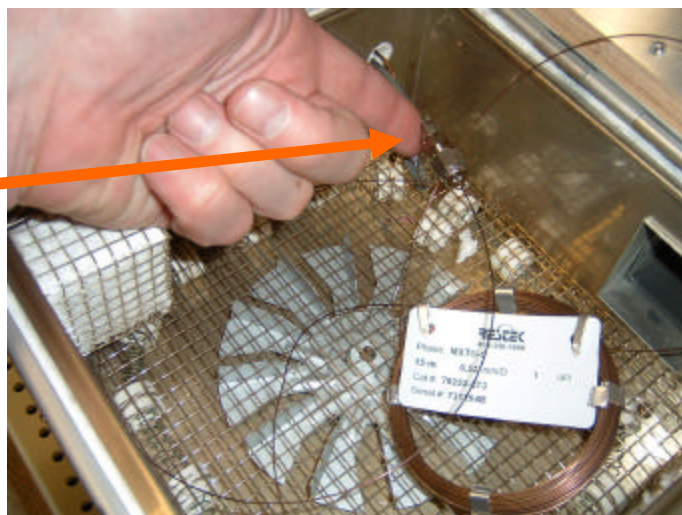
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The carrier gas is connected to the Thermal Soil Desorber by a 1/16" stainless steel tube. Connect this tube to the outlet of the injector port. The GC shown in the photo has a heated injector, but other SRI GCs may have the on-column injector which looks slightly different. The 1/16" tube is secured to the injector using a swagelok nut and 1/8" to 1/16" soft graphite reducing ferrule.



The analytical column is connected to another 1/16" stainless steel tube using a swagelok nut and 1/8" to .8mm soft graphite reducing ferrule. Any column may be used, but a 15 meter MXT1 with a 1 micron phase and .53mm OD is commonly used for diesel range organics analysis. Push the column through the fitting and into the bore of the 1/16" tubing to minimize dead volume.



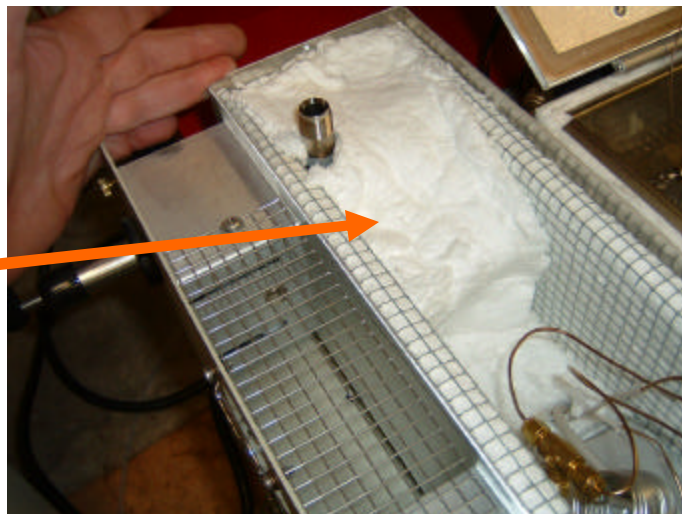
The other end of the column is inserted into the FID detector using a 1/8" nut and 1/8" to .8mm soft graphite reducing ferrule. The FID temperature should be set to 300C to prevent condensation of high boilers if diesel range organics are being analyzed. The FID detector is commonly used for this although other detectors may be useful for specific applications.



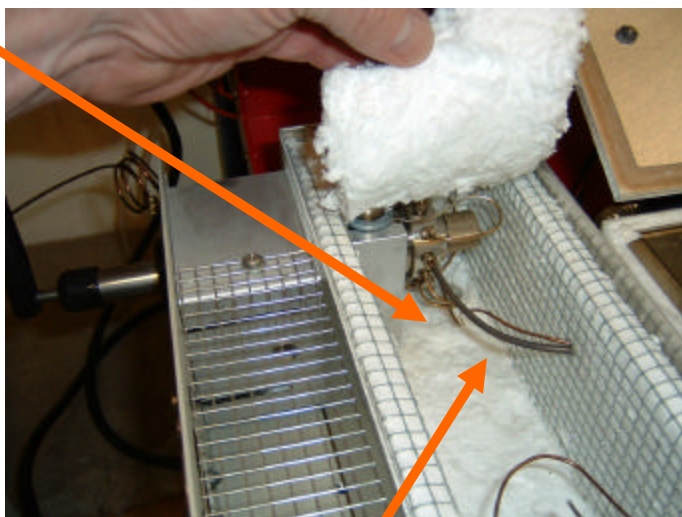
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The Desorber's heated block must be heavily insulated to allow the temperature to reach 275C. The insulation (spun borosilicate glass blanket) is formed over the heated block area to form a hot pocket which also keeps the tubing leading from the valve to the column oven hot.



If the connecting tubing is not hot enough, the high boiling molecules will condense and may not reach the column.



The stainless steel tubing is covered in copper to provide better thermal stability and heat transfer.

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To prepare a sample, place some glass wool in the glass desorber tube. Form the glass wool into a loose plug about halfway up the tube.



Weigh the tube and hit the tare button on the balance.

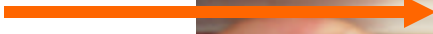


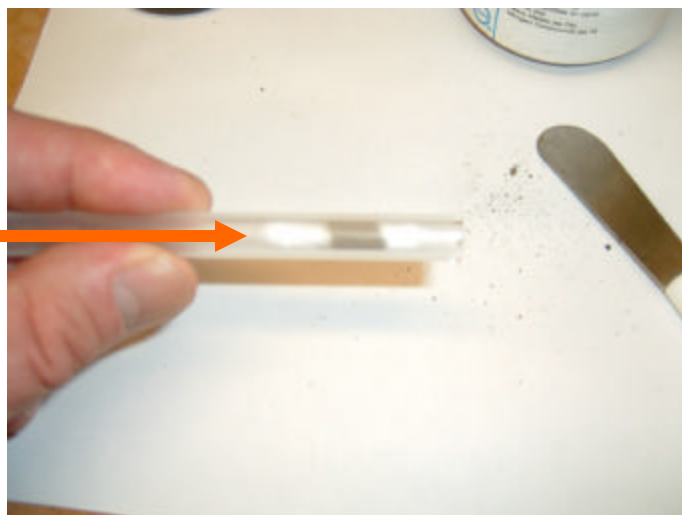
Load the tube with up to 1 gram of soil.




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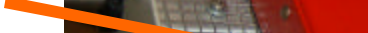
Place another plug of glass wool to sandwich the soil between the glass wool plugs. The glass wool should be tight enough to keep the soil in place but not overly compressed. 

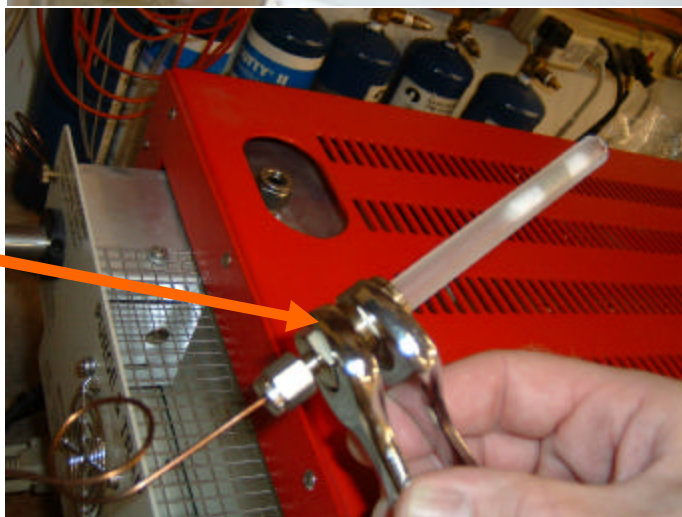


If the soil is very wet, or is mostly clay (rather than a sandy soil) mix the soil 50/50 first with sodium sulfate. The sodium sulfate will absorb the water and the clay particles will coat the sodium sulfate crystals. This improves the efficiency of desorption for clay soils especially. 



Weigh the tube again and record the weight plus any dilution factor if the sodium sulfate was used.

Attach the tube to the carrier gas fitting using two wrenches and a 3/8" soft graphite ferrule. If the fitting is not over-tightened, the ferrules can be re-used many times. 



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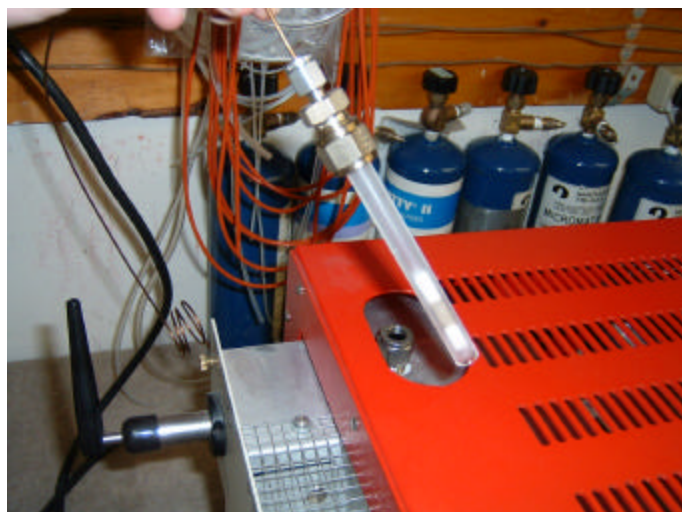
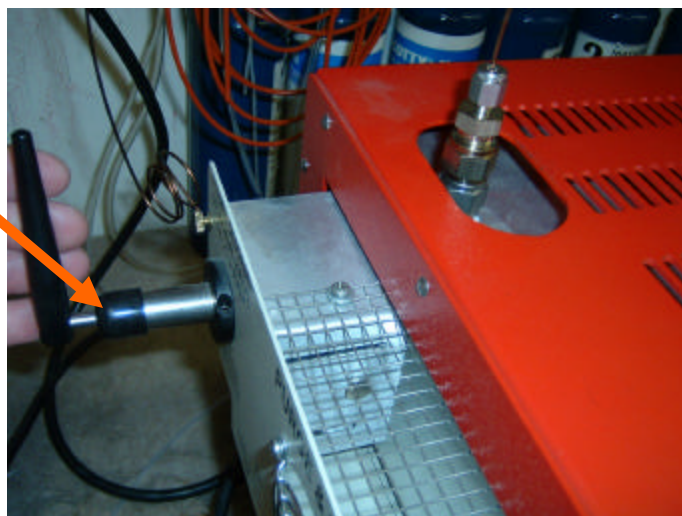
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Connect the tube to the hot (250C) desorber cell.



Immediately rotate the handle of the Valco valve clockwise (36 degrees) until it hits the stop. Start the Peak-Simple Data System at the same time by pressing the start button on the GC or the spacebar on the computer keyboard.

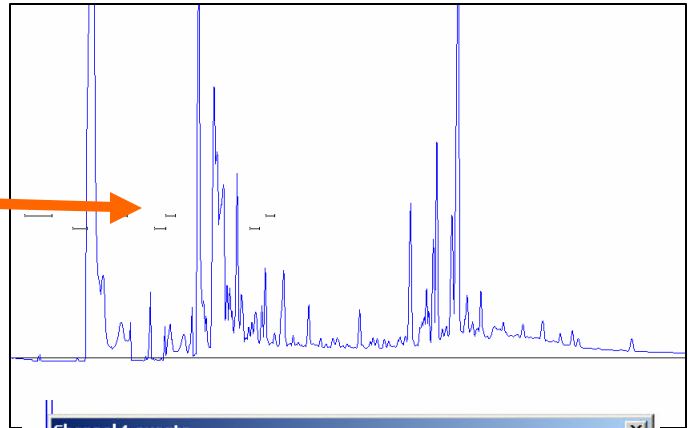
Wait 1 minute. Then rotate the Valco handle all the way counter-clockwise and remove the desorber tube (BE CAREFUL, ITS HOT) . Place the tube in the hole in the grate to cool while the run is in progress.



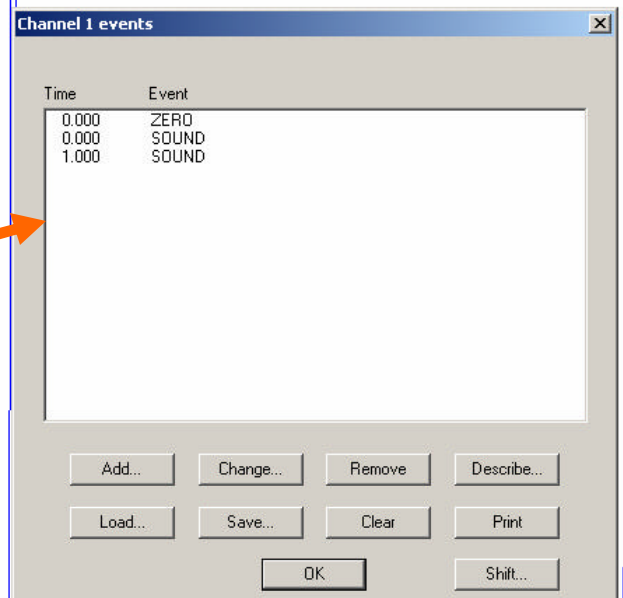
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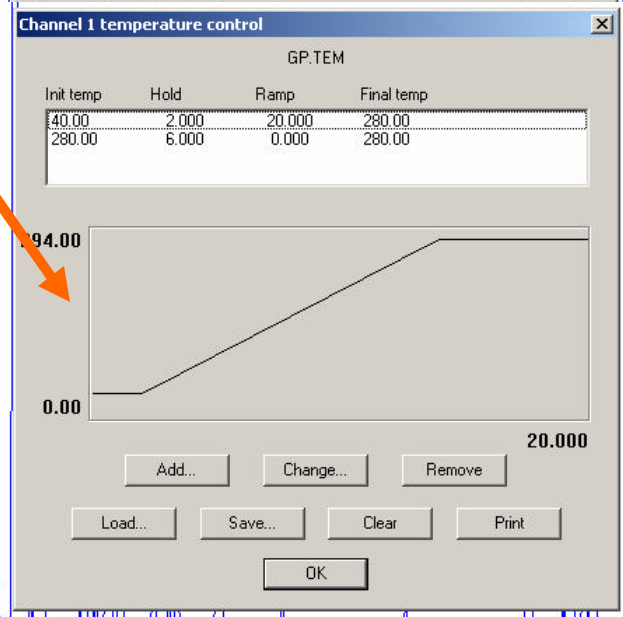
The hydrocarbons in the soil are desorbed into the column and separated.



It is helpful to enter an Event table similar to the one shown to auto-zero the detector signal at the beginning of the run, and to make a sound at beginning of the run (to verify that the data system has been started), and another sound at the 1.00 minute mark to remind you to remove the desorber tube.



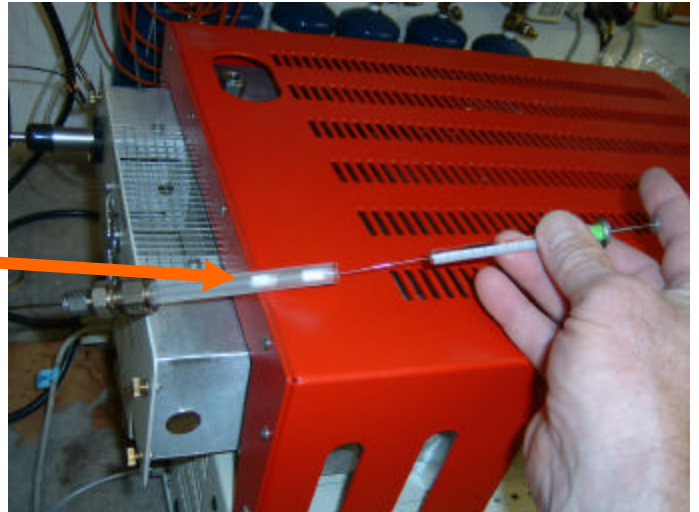
You will also need to enter an appropriate oven temperature program such as the one shown. Generally, the temperature should be low (40-50C) for the first two minutes to focus the desorbed hydrocarbons at the beginning of the column. The temperature is then ramped to 250-350C at 10-30 degrees per minute to elute the hydrocarbons. The exact program selected will depend on the type of sample, required resolution and time available.



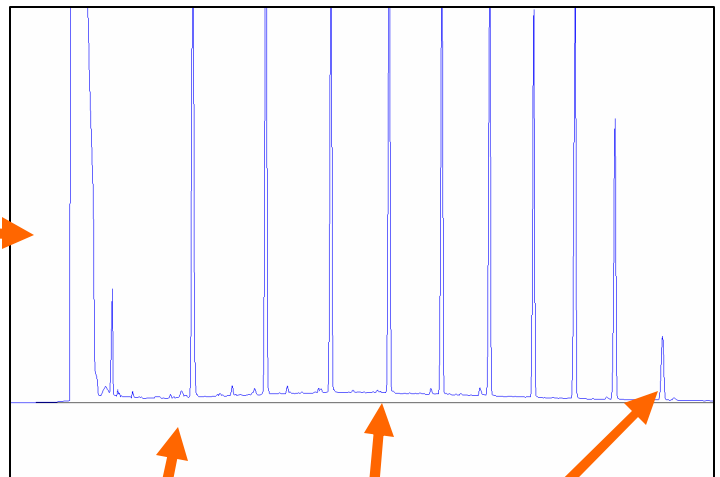
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To inject a calibration standard use a 10ul syringe to deposit a calibration solution on the previously desorbed soil in the tube.



At SRI we use a mixture of normal C10, N-C12... through normal C28 hydrocarbons at 1000ppm in methanol as a test mix for the Thermal Soil Desorber. If we spike 1 ul of this mixture into 1 gram of soil, this is the equivalent of 1 ug of each hydrocarbon or 1 ppm by weight. The resulting chromatogram is shown at right.



We evaluate the chromatogram by looking for:

- 1) Sharp well defined peaks. This indicates that there are no cold spots which would smear the peaks
- 2) Roughly equal area counts for each peak. Sometimes the C28 peak is lower because the temperature is not hot enough to completely desorb it from the tube.

Normal C10

Normal C28

Normal C16