

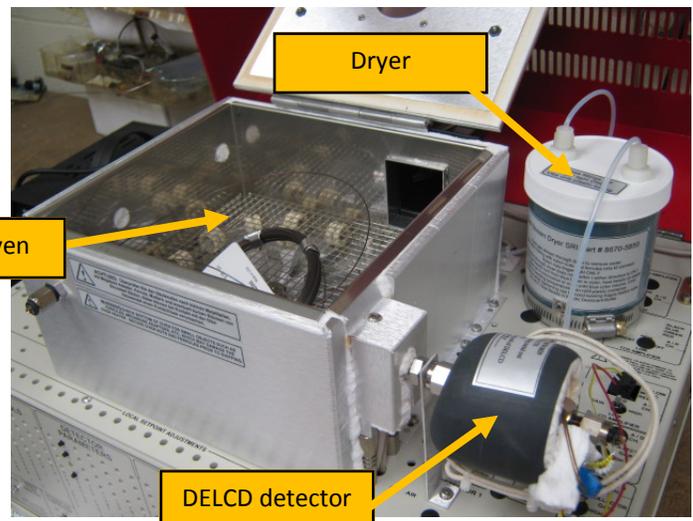
SRI 8610C GC for PCB in Transformer Oil Analysis

November 2014

The SRI 8610C Gas Chromatograph (GC) is installed on any bench top.

When the red lid is raised, you can see the column oven, injector (on-column type), detector (DELCD) and gas stream dryer. The GC will not heat while the lid is raised.

The DELCD detector will come in a separate box so it must be mounted to the GC.



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Slide the white ceramic tube of the DELCD into the 1/4" Swagelok fitting.

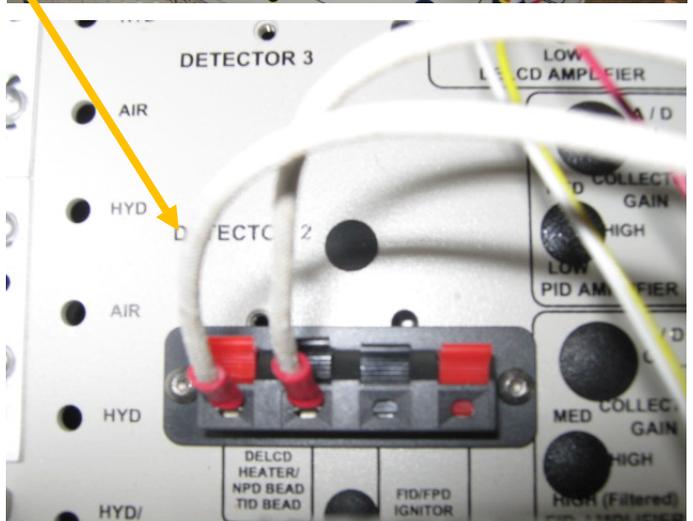
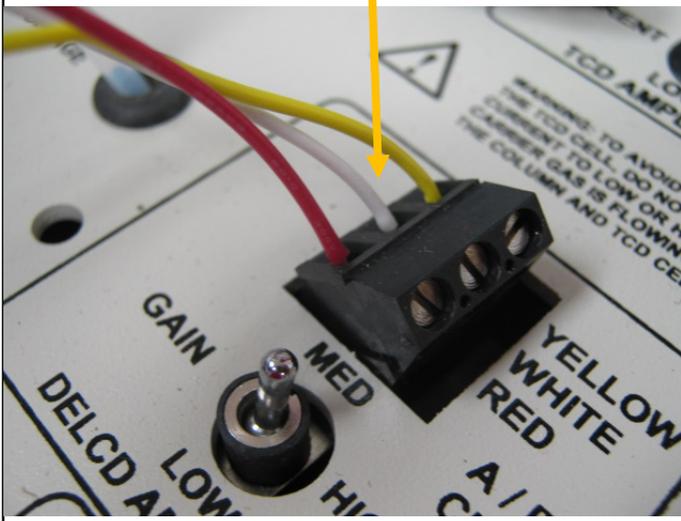
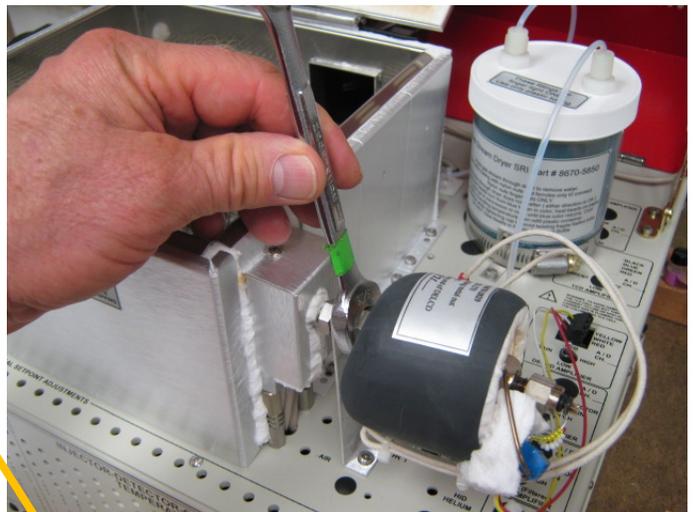
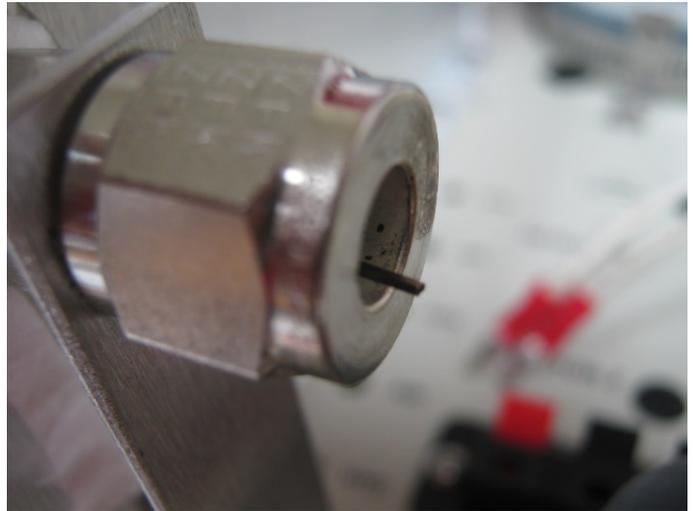
The end of the column should stick out 2 mm from the nut so it enters the DELCD tube.

Tighten the graphite ferrule gently, not too tight, it just has to hold the DELCD in place.

Attach the two heater wires to the terminals.

Attached the 3 colored wires to the screw terminals.

Set the gain switch
To medium.



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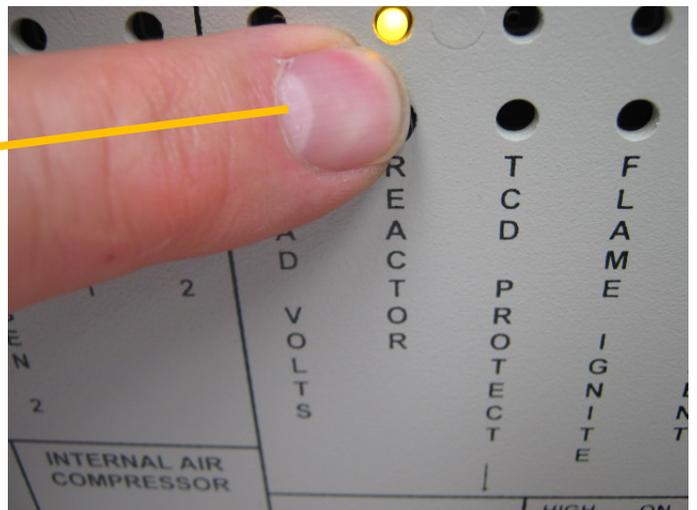
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Verify the carrier gas pressure is 10psi (.67bar). The carrier gas is room air from the built-in air compressor. Make sure the air compressor switch is UP (on). 10psi is about the maximum pressure since the air compressor outputs 12psi.



Verify the DELCD reactor temperature shows approximately 260 (250-260 is OK). The actual temperature inside the DELCD heater is about 1000C, but the readout shows 250-260 because of the way the platinum thermo-couple is calibrated.

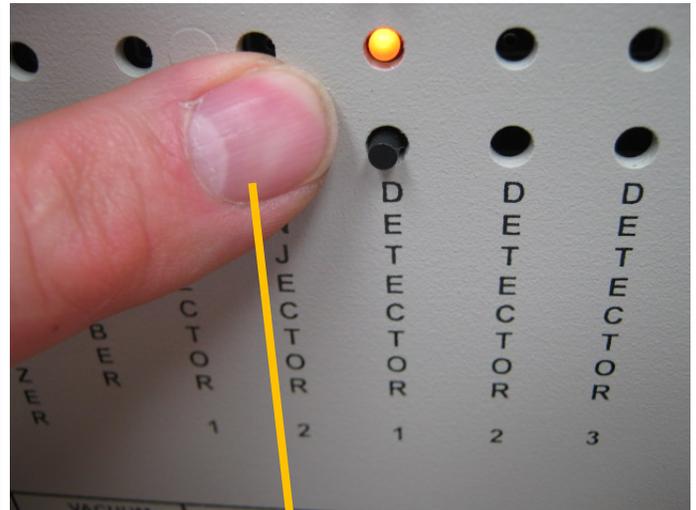
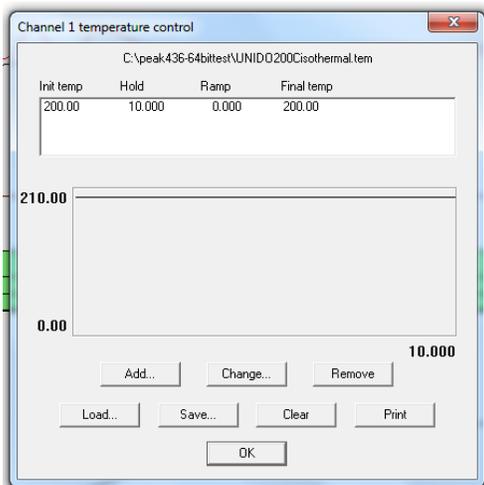


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Verify the DELCD block temperature is 250C. This is the aluminum block which supports the DELCD detector.

The column is mounted in the column oven. SRI has supplied a Restek MXT500 column, .53mm id .15micron film. In the PeakSimple software, set the oven temperature program as shown for a 10 minute isothermal program at 200C. Other temperature programs may be used if you prefer.



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The column is connected to the on-column injector with a 1/8" Swagelok nut and soft graphite ferrule.

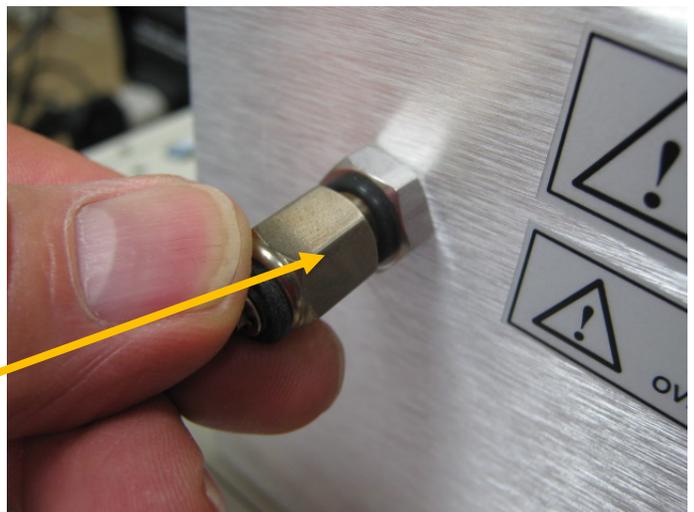


The septum nut contains a green septum call a "plug" type. Restek part# 27154. Verify the septum is inserted into the nut as shown. The narrow part of the plug faces out of the nut.



Do not overtighten the septum. Finger tighten just enough to make a seal. If the septum is too tight it will cause the syringe to plug up. A gently tightened septum can last for hundreds of injection.

Tighten just enough to reach the two black O-rings on the injector.



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The syringe (SRI part# 8670-9550) is a 10ul glass syringe with 6cm needle. The needle has a conical point rather than a sharp point so the septum lasts longer and the needle enters the bore of the column more smoothly.

The syringe should slide smoothly all the way into the injector. If it feels like the syringe snags or hits the lip of the column, then make sure the column end is cut smoothly and pull the column back a little further into the wide-bore adapter.

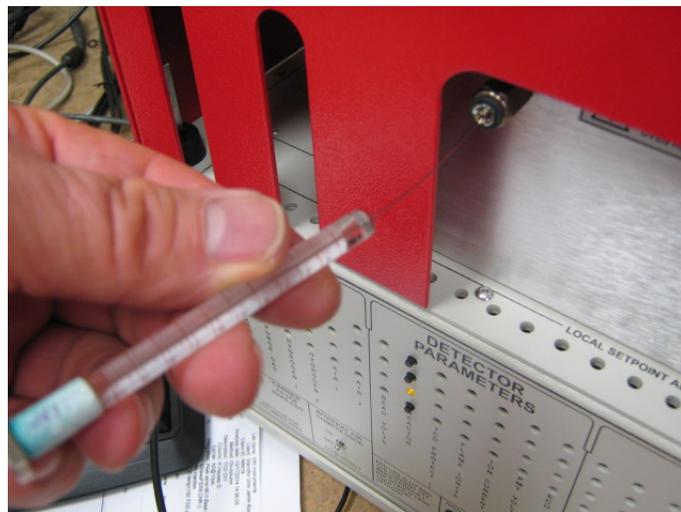
See the on-column injector documents on our website for more info:

[http://www.srigc.com/download/ColumnInstallation\(on-column%20injector\).pdf](http://www.srigc.com/download/ColumnInstallation(on-column%20injector).pdf)

Obtain some PCB standard such as the Restek 1016/1260 combination standard (part# 32039). This PCB standard is supplied at a concentration of 1000ug/ml (1000ng/ul).

Break the ampoule and transfer the standard into the supplied 1.8ml septum vial.

This is the primary standard. Keep it in the refrigerator when not in use.



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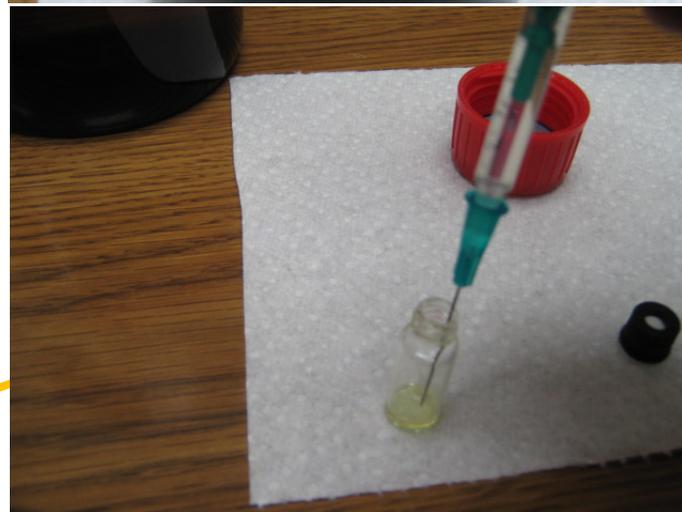
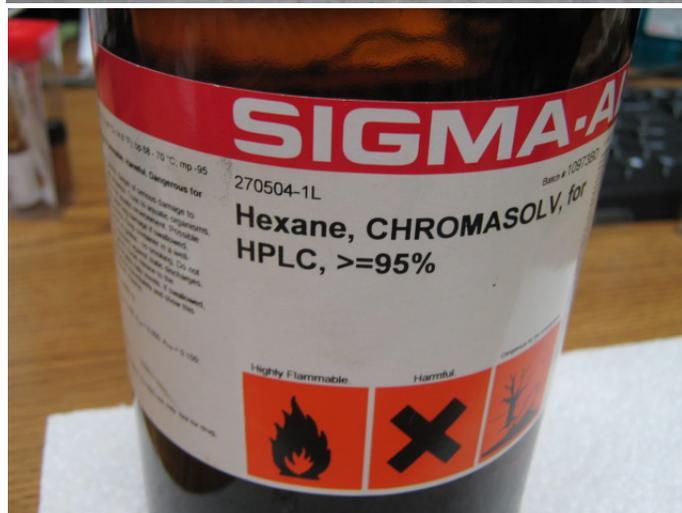
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Use a 100ul syringe to transfer 100ul of known "clean" (no PCBs) transformer oil into a vial.

Use a larger syringe to transfer 400ul of Hexane into the same vial, diluting the oil 1:5. This is done to reduce the viscosity of the oil so the syringe can more easily inject into the GC.

Add 5ul of the Restek PCB standard into the vial, diluting the 1000ng/ul by 100 times so the resulting concentration of PCB in the diluted oil is 10ng/ul.

This is the working standard.



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Make another 1:5 oil dilution in a separate bottle, but do not add the PCBs.

This will be the clean oil.

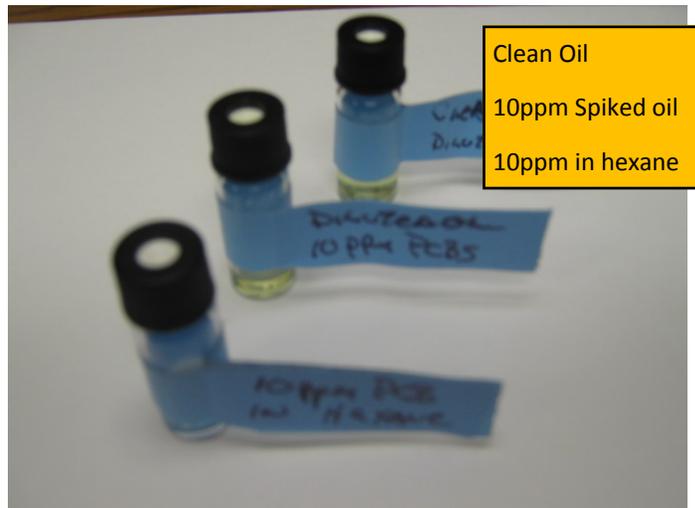
Make a third dilution of 5ul the Restek standard into 500ul of just pure Hexane.

This will be for comparison purposes and to understand the impact the transformer oil has on the sensitivity of the DELCD to the PCBs in the presence of the oil.

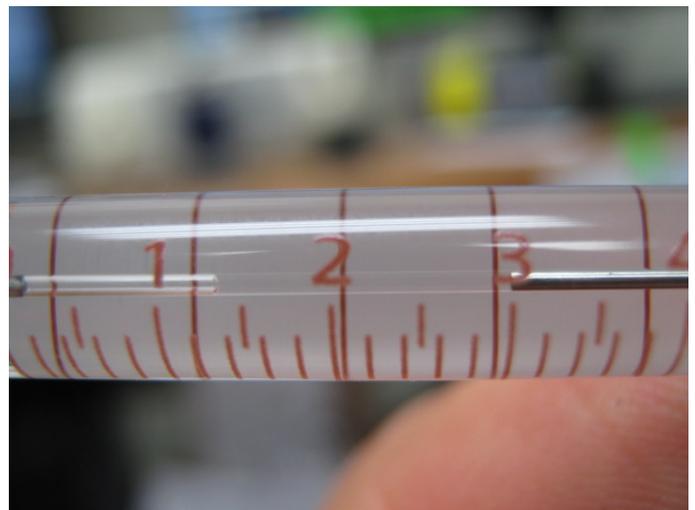
Make the real samples in the same exact way. Dilute the real oil sample 1:5 in hexane. 100ul oil plus 400ul Hexane.

Use the 10ul syringe to measure 1ul of the standards.

- 1) Pump the syringe many times to remove all traces of previous contents and air bubbles.
- 2) Push the plunger to the middle of the 1ul mark.
- 3) Wipe the syringe needle with a paper towel.
- 4) Pull the plunger back to the 3ul mark. You will see the liquid in the syringe barrel is 1.8 ul (approximately). This is normal because the needle has .8ul volume. Check to verify the volume is 1.8ul. It is important that every injection (standard as well as sample) contain the exact same amount of liquid.



Clean Oil
10ppm Spiked oil
10ppm in hexane



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Inject 1ul of 10ppm PCB in Hexane into the GC. It's a good idea to push the GCs start button just before you inject the sample.



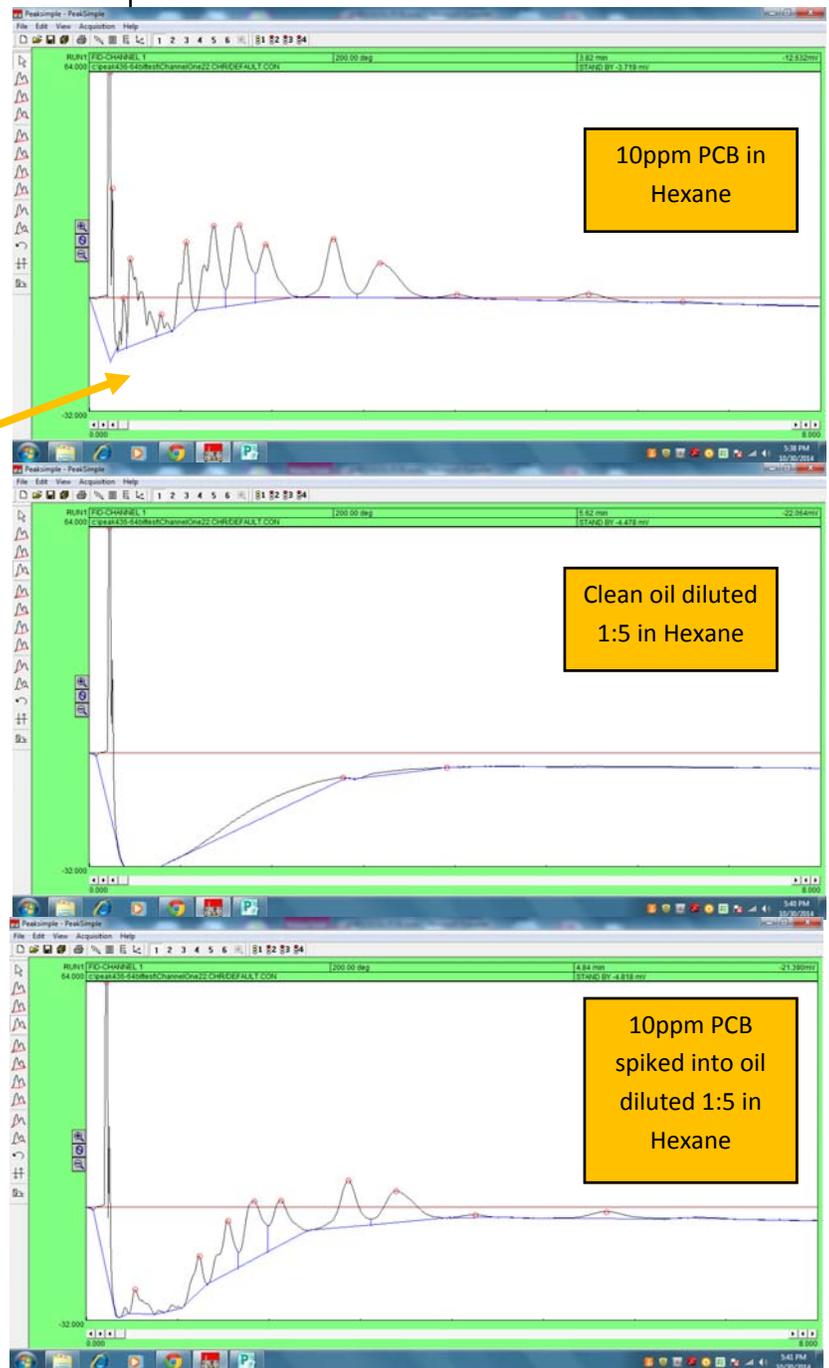
The chromatogram will look like the one at right.

Inject the clean oil for comparison.

Then inject the 10ppm PCB in diluted oil.

You will get a chromatogram like the one at right.

Since the oil is diluted 1:5, this chromatogram is what a transformer oil contaminated by 50ppm of PCBs will look like once it has been diluted and injected into the GC.



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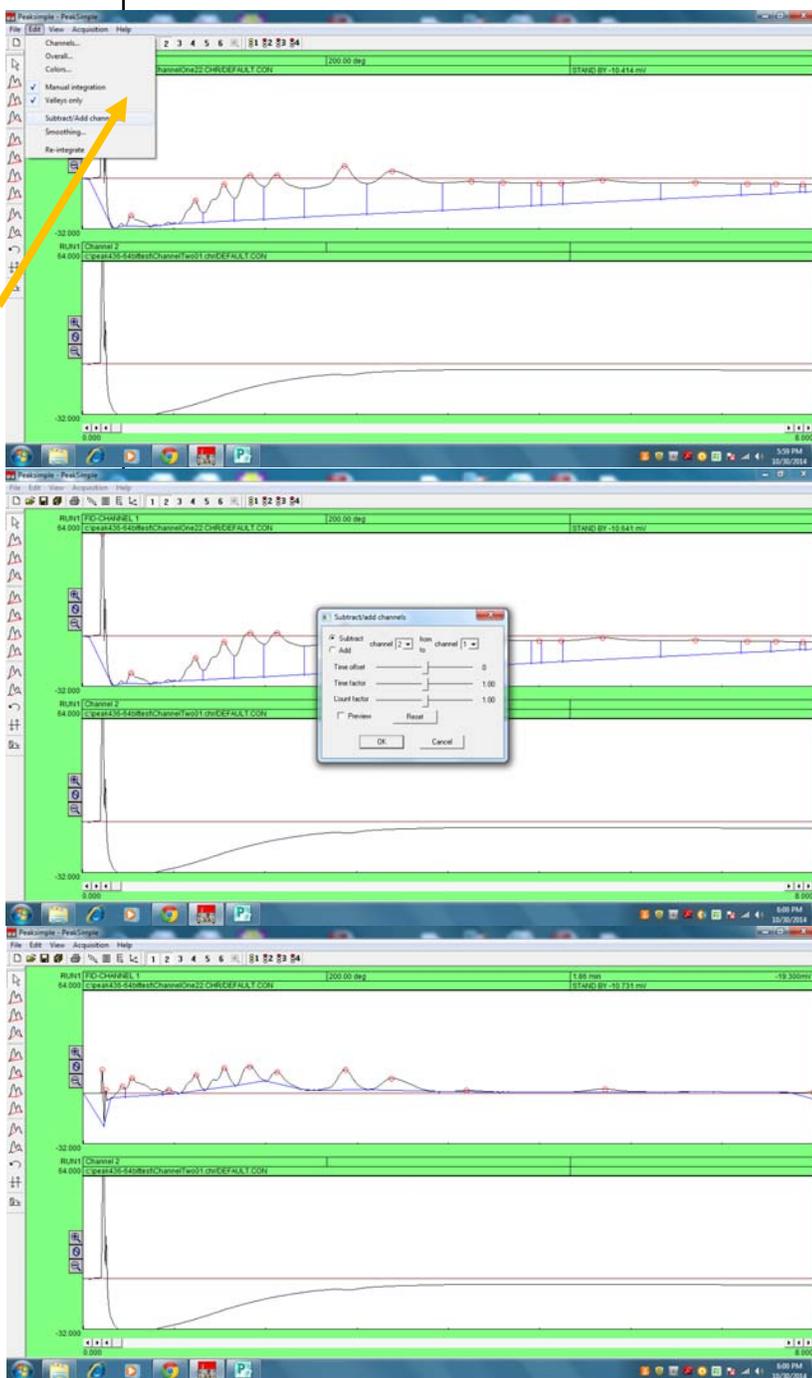
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Load the clean oil chromatogram into channel 2 and the 10ppm in oil into channel 1. The two screens will look like this.

Click File/Subtract/Add channels

Subtract the channel 2 data from channel 1.

The channel 1 data will then have a flatter baseline which is easier to integrate and calibrate.



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Point to the chromatogram with your mouse and click on the RIGHT hand button.

From the menu select “Add Component”

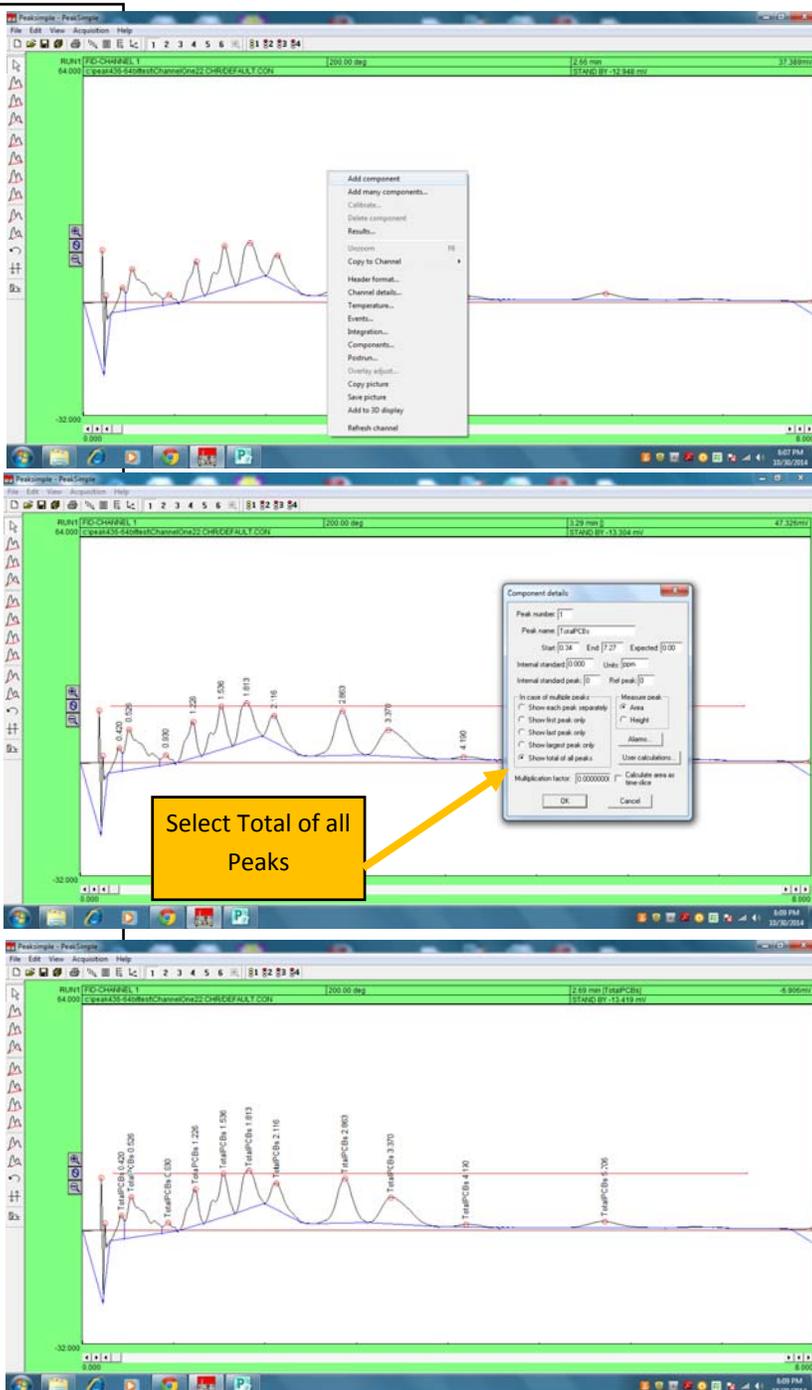
Stretch the Red Retention Window from just after the solvent peak to the last peak in the analysis.

See the document on the SRI website for more info on moving retention windows.

<http://www.srigc.com/PeakSimple%20Advanced.pdf>

Right click and then select “Edit Component”. Fill out the Component detail screen as shown. Make sure to select “Total of All Peaks” so PeakSimple adds up all the PCB peaks into a total.

The chromatogram will now look like this. Note that the blue baseline may connect to some of the valleys between the PCB peaks rather than draw a straight line below the peaks.



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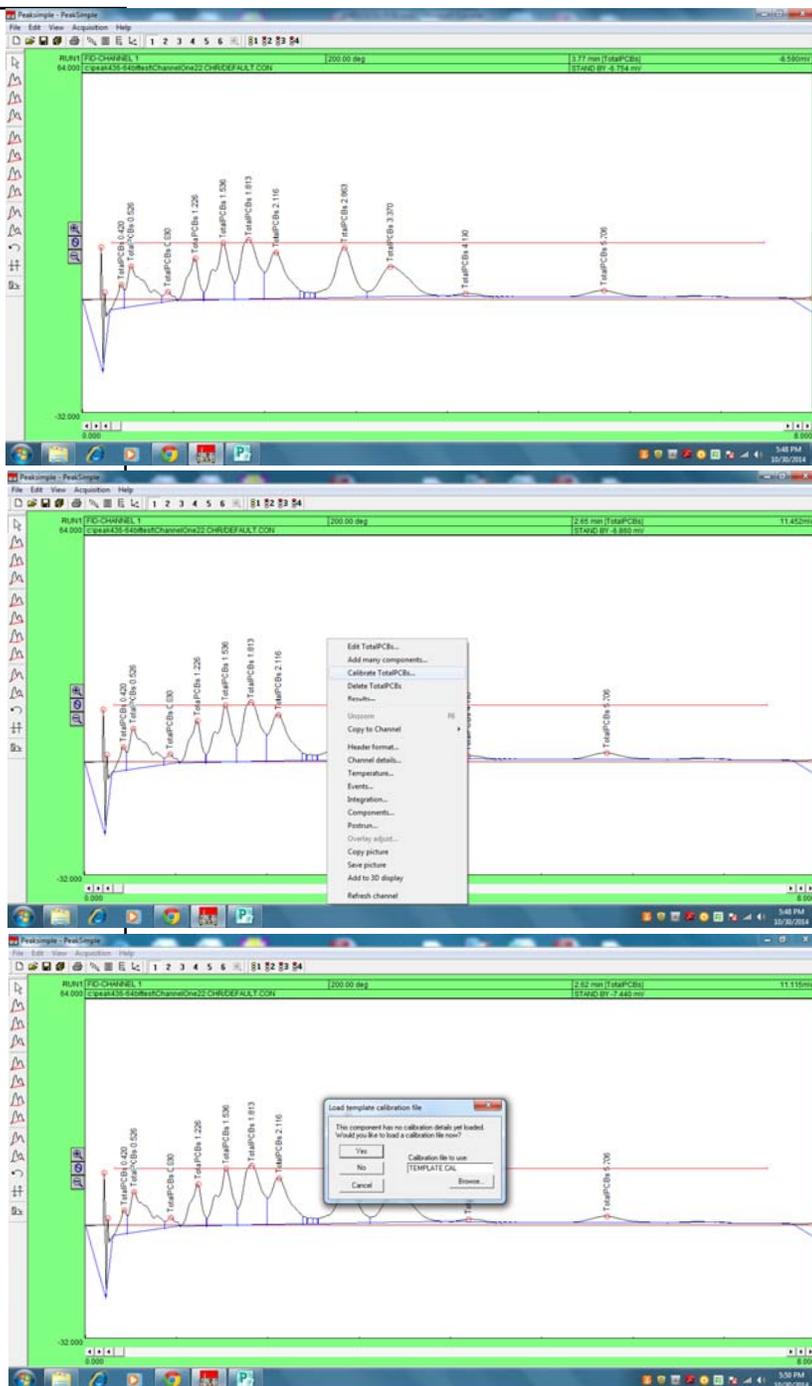
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Correct the baseline using the manual integration tools (or modify the integration parameters) so the blue baseline defines the bottom of the PCB peaks correctly.

Right click on the chromatogram and the select Calibrate TotalPCBs.

Since there is not already a calibration curve, you will see this screen. Click NO.



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Click Level 1 if it is not already selected.

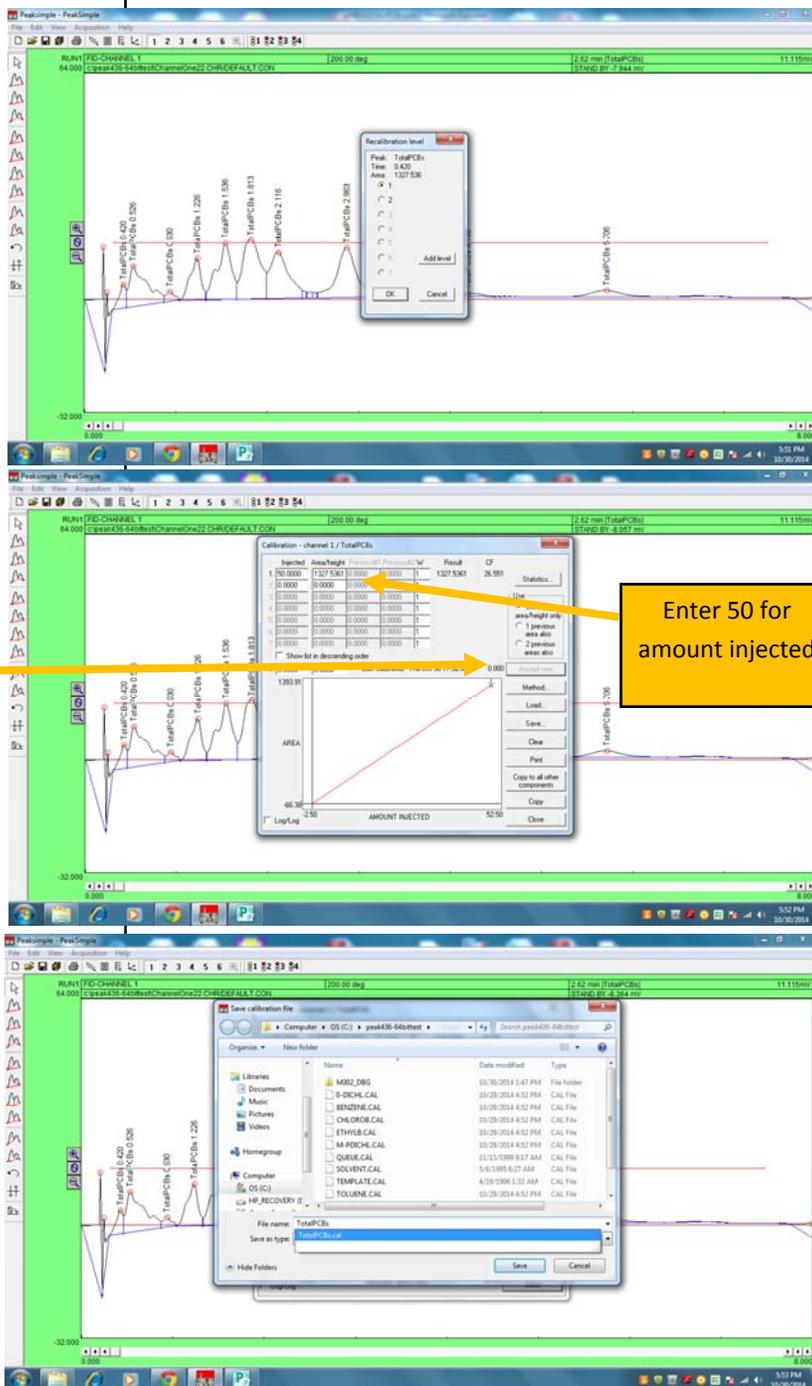
This takes you to the blank calibration curve. Enter the number 50 in the top left cell.

If we enter the number 50 for the amount injected, the answers for the subsequent injections of real transformer oil will be calculated for the un-diluted oil.

Then click the button labelled "Accept New".

This writes the area of the totaled PCB peaks (500-1500) into the top row 2nd column, and draws a line from zero on the curve to the data point just entered.

Click "Close" and PeakSimple will prompt for a filename to assign the calibration curve. Once named, further modification to the calibration curve are saved automatically.



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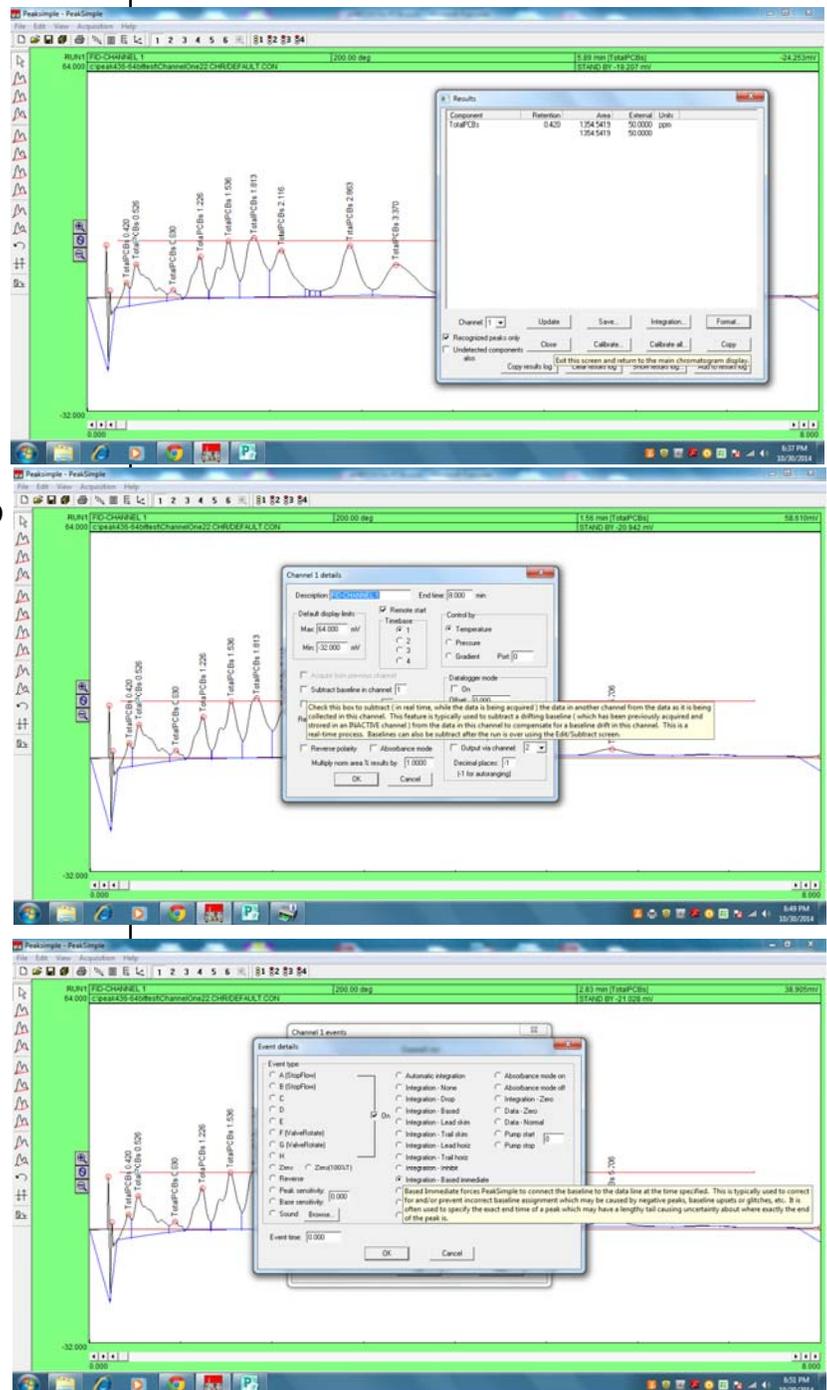
Click View/Results to see the calibrated answer (50ppm).

The SRI GC system is now calibrated and ready to measure PCBs in transformer oil.

The baseline subtraction can also be performed in real-time by clicking the checkbox in the "Channel Details" screen.

Some of the baseline corrections can be automated using the Event table commands. The "Based Immediate" command is especially useful to get the baseline to integrated the PCBs properly.

Note the "tooltips" instructions which pop up when you mouse over the radio buttons.



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Periodically check the dessicant in the gas dryer. The DELCD detector is more sensitive with dry air than wet air.

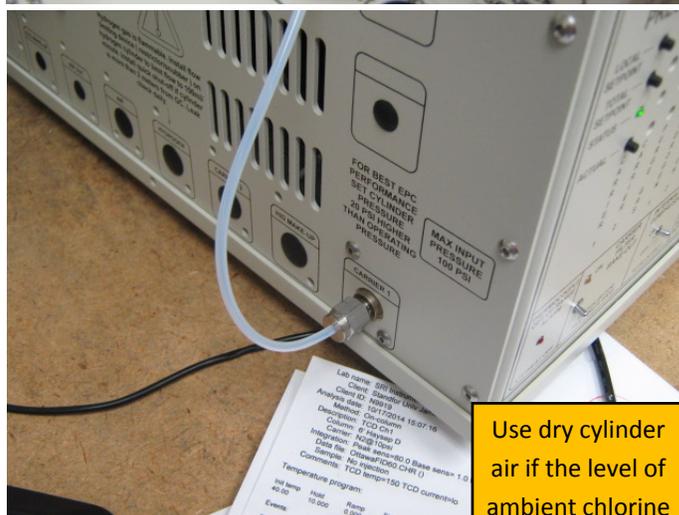
If the dessicant turns brown then it is time to either re-charge the dessicant (see website for instructions) or replace the dryer with a spare.

The dried air from the dessicant is connected to the carrier gas in fitting on the GC left side. If required, dry air from a gas cylinder can be connected instead.

The DELCD gets more sensitive the dryer the air is.

It is normal for the DELCD sensitivity to increase slightly if injections are not made periodically. If you let the system run all night, the first 2 injections in the morning will be larger than subsequent injections of the same standard.

Try to avoid injecting more than about 20ppm chlorine as this can also temporarily affect the DELCD sensitivity.



Use dry cylinder air if the level of ambient chlorine is high.

