ASTM 4815 GC operation

October 2022

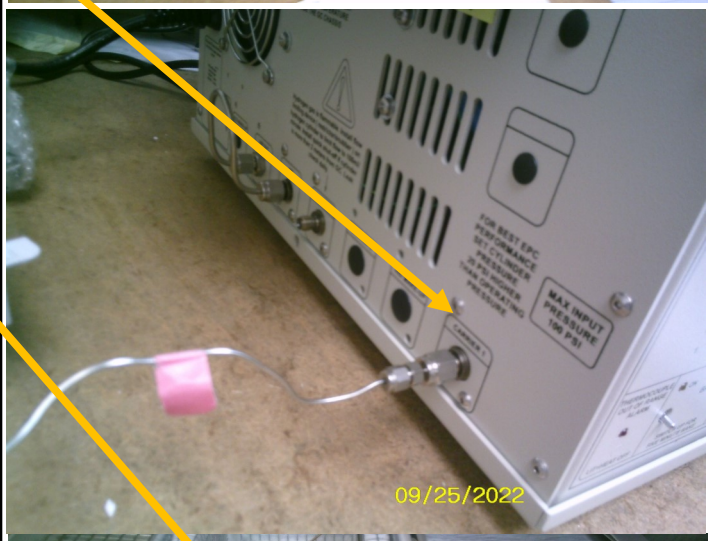
This SRI (or Buck equivalent shown here) GC is configured for ASTM 4815.

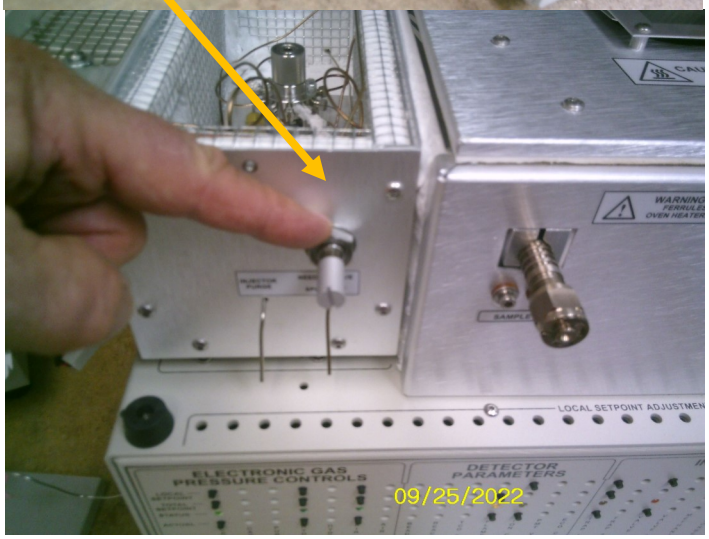
This method is intended for the analysis of oxygenates in gasoline, mostly ethanol, but there could be many different oxygenates.

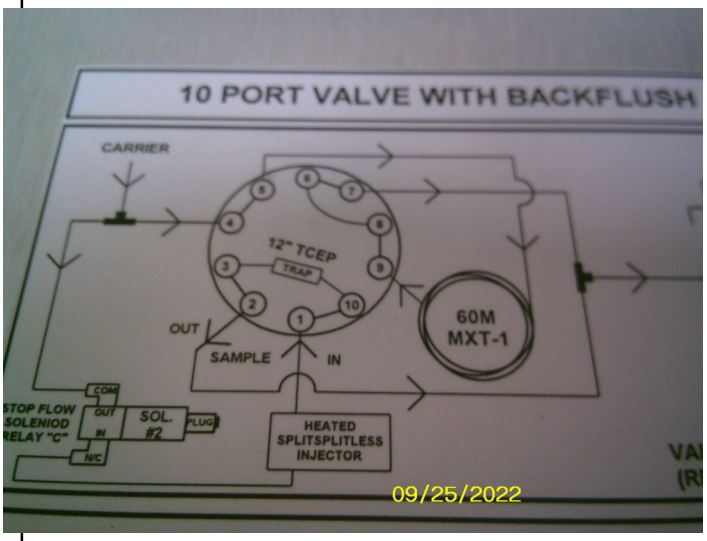
The GC can operate on just hydrogen carrier as shown here, or another carrier gas like nitrogen, argon or helium could also be used. In that case the hydrogen would be connected to the hydrogen inlet instead of the carrier inlet.

The heated injector is equipped with a stop-flow solenoid controlled by Relay C, and a split solenoid controlled by Relay A.

The split ratio is adjustable using this needle valve.







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ASTM 4815 GC operation

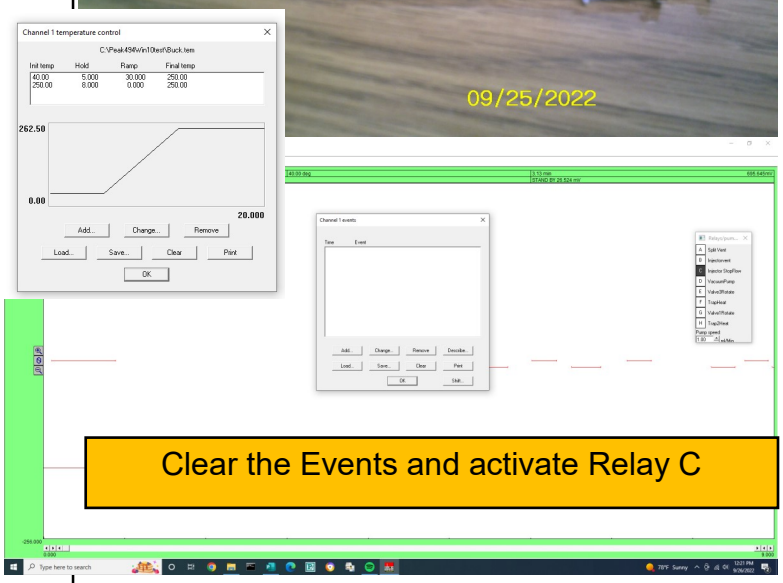
October 2022

The GC is equipped with a 12' long trap tube filled with a very polar powder called TCEP. The TCEP trap is encased in an aluminum heater block located between the ducts of the valve oven.

The trap heats to the setpoint when Relay F is activated. TCEP must not be heated higher than 150C. When Relay F is de-activated the black blower cools the trap down to the adsorption setpoint, typically 50C, but it can be set higher or lower.

Make a test sample with 100ul each of-methanol and hexane.

Set the temperature program as shown. Clear the Event table and manually activate Relay C. The Relay/Pump window appears when you click View/ RelayPump window. When you click the C box, the box changes to black and you will hear the sound of the InjectorStopFlow solenoid. This allows carrier gas to flow to the Heated Injector and from there through the TCEP trap. The outlet of the TCEP trap is connected to the FID detector inlet so any molecules which blow through the TCEP trap will be detected by the FID detector.

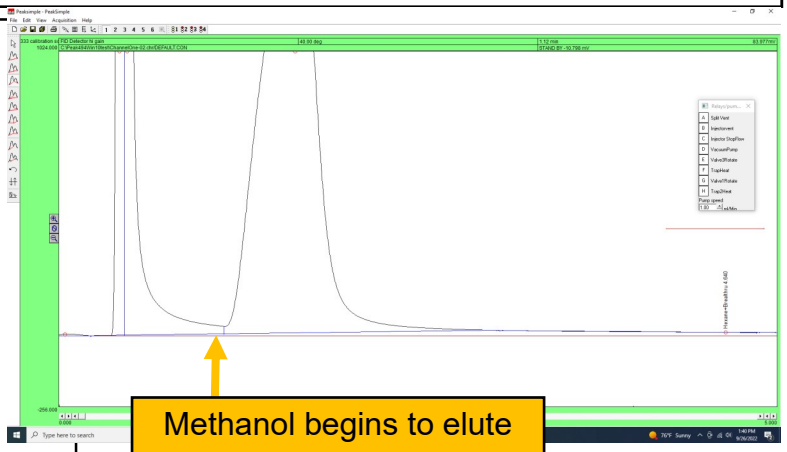


Clear the Events and activate Relay C

ASTM 4815 GC operation

October 2022

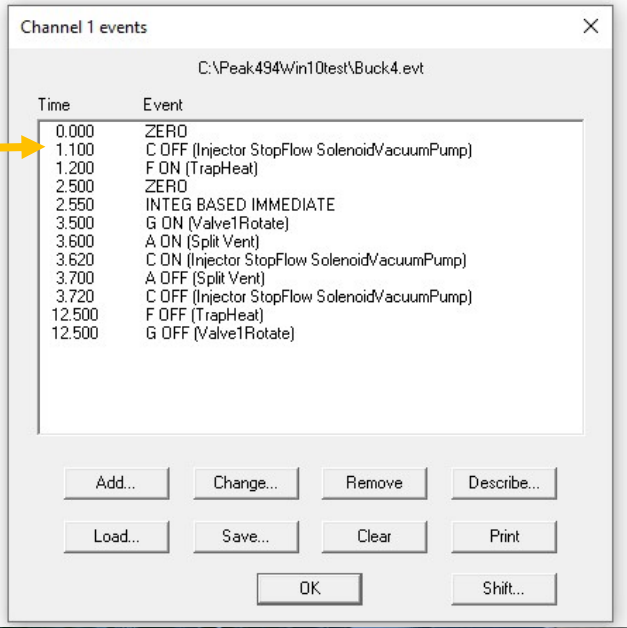
When you inject 1ul of the test mix containing 50/50 hexane and methanol you will see a chromatogram like this.



The first peak is the hexane which because it is non-polar, travels through the polar TCEP trap very quickly.

The second peak is the methanol, which because it is polar travels through the polar TCEP trap slower than the hexane and all non-polar molecules below hexane's boiling point.

Find the time just before the methanol elutes from the TCEP trap and enter it in the Event table as the time to turn Relay C off.



This will allow all the non-polar (up to the hexane boiling point range) molecules to elute and all polar molecules (oxygenates) to remain on the TCEP trap.

Higher boiling Non-polar molecules like toluene may also remain on the TCEP trap.

Inject the test sample again with the Event table active. Remember to turn Relay C on manually 10 seconds before you make the 1 ul injection. Relay C will turn off automatically at 1.10 minute.

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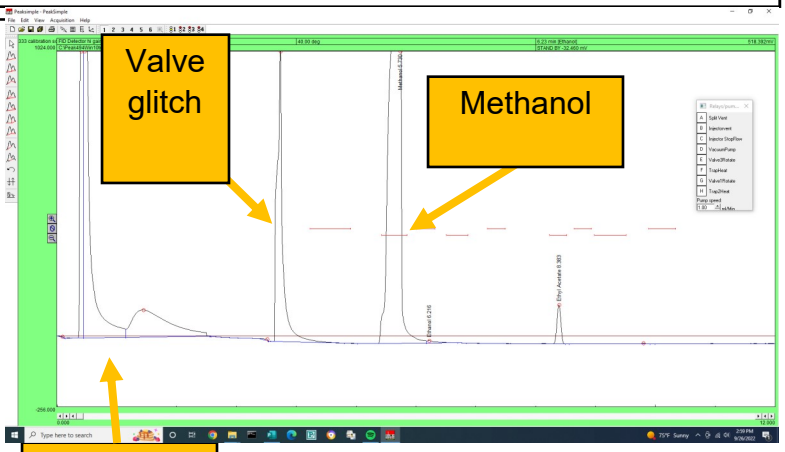
ASTM 4815 GC operation

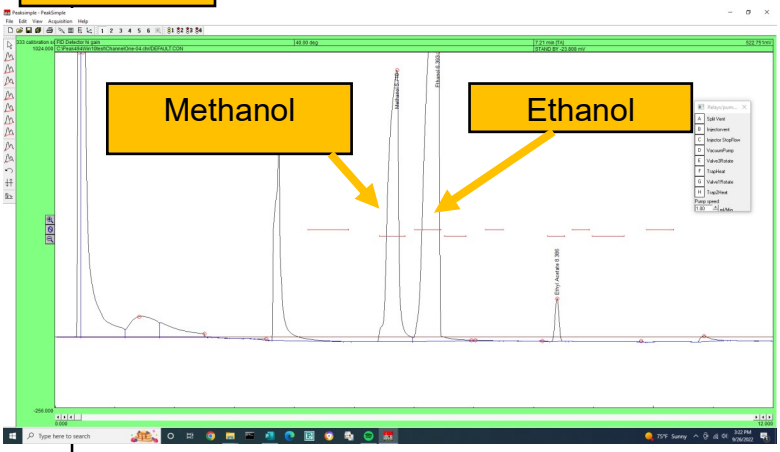
October 2022

You should see a chromatogram like this.



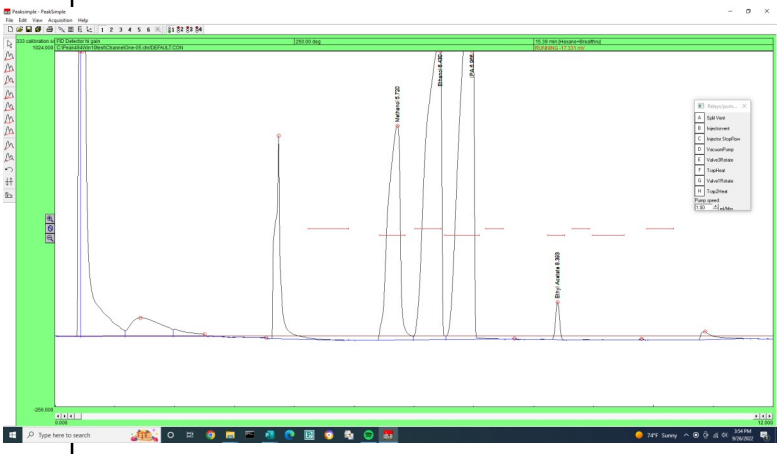
Add 100ul of Ethanol to the test standard.

Using the same method inject 1ul into the GC. You should see both a methanol and ethanol peak.



Add 100ul of IPA to the test standard and inject 1 ul into the GC as before.

You should see a chromatogram with the 3 peaks.



ASTM 4815 GC operation

October 2022

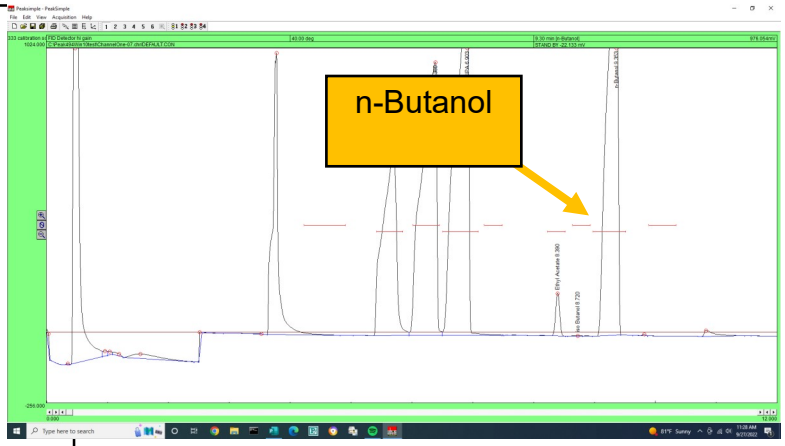
Continue this process by adding as many oxygenates as desired to the test mix. The chromatogram at right shows n-Butanol added to the test mix.

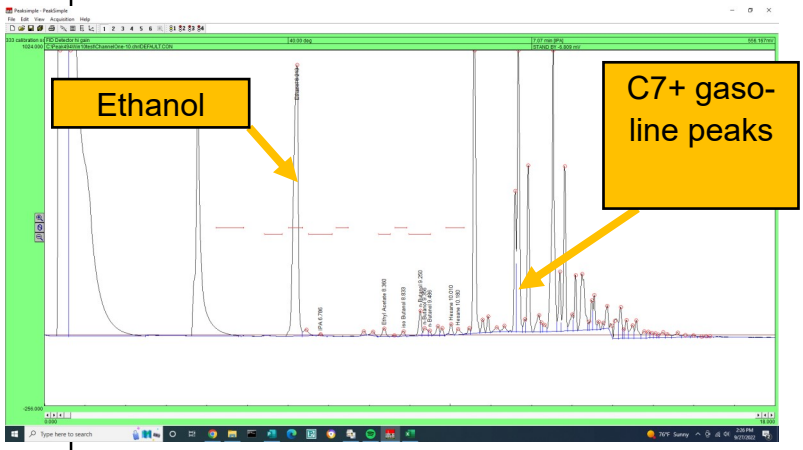
After all the oxygenates are added to the mix, each one may be 10% of the total.

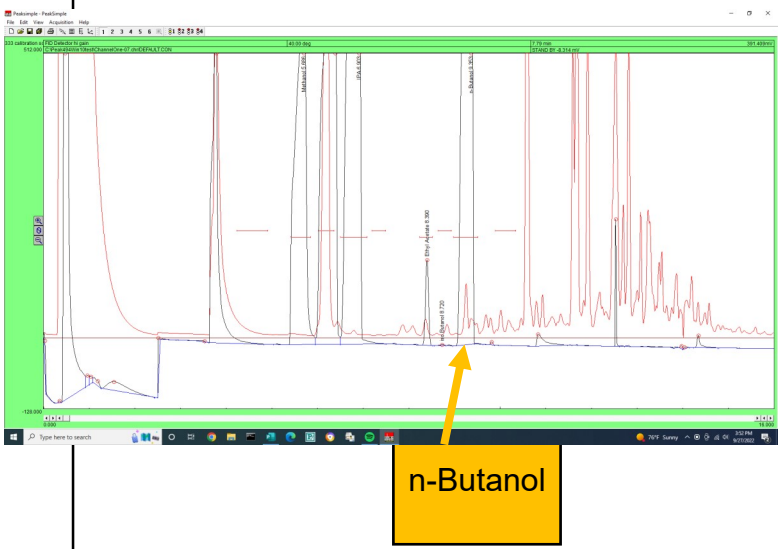
If so, you can calibrate on this. If only ethanol will be measured, a separate calibration mixture will need to be prepared with the ethanol at an appropriate concentration (typically 10%).

Shown here is 1ul California gasoline which has ethanol in it. You can see the ethanol peak appears in an area with no interfering gasoline peaks..

Here is the test chromatogram (in black) with n-butanol overlaid with the gasoline chromatogram (in red). You can see the n-butanol elutes just as the gasoline peaks start, so n-butanol is the highest boiling oxygenate which this method can detect without interference.







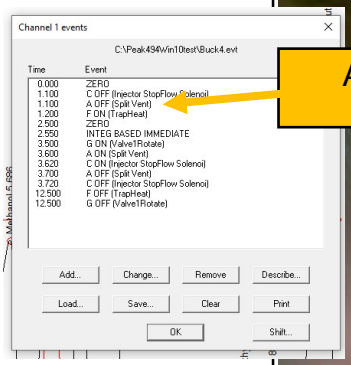
ASTM 4815 GC operation

October 2022

The ASTM 4815 GC is equipped with a Heated Split/Splitless injector. The split ratio is adjusted with a needle valve.

You may not need to split the sample since a 1ul gasoline injection stays on-scale, but you can make the peaks smaller if you wish by operating the injector in the Split mode.

To operate in the split mode you manually activate both Relay A (split vent) and Relay C (stop flow) prior to injecting the sample.



A off

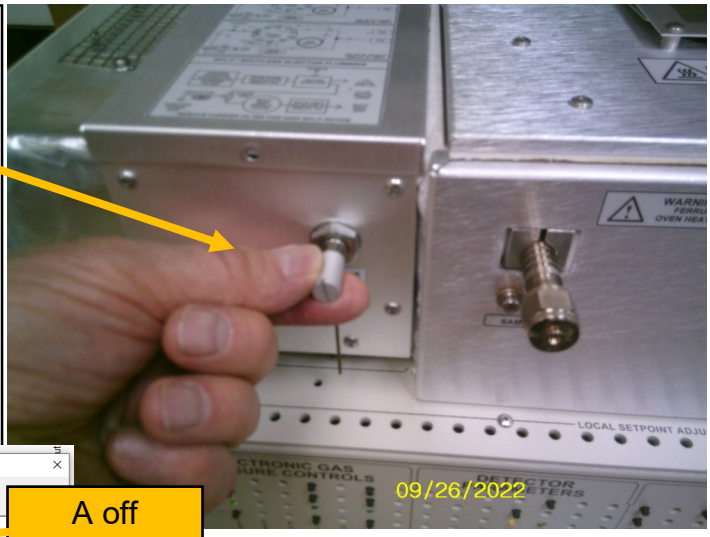
You modify the Event table to also include a command to turn Relay A off either at the same time as Relay C off, or earlier.

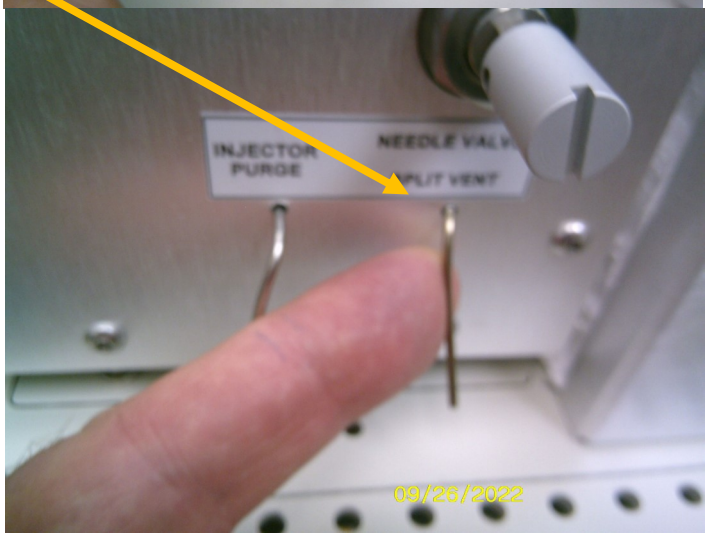
The split flows constantly through the “Injector Purge” line at a very slow rate to make sure no condensed vapors diffuse back into the system.

The main split flows out of this tube only when Relay A is activated.

You can set the split ratio using a flowmeter or by trial and error.

The flow through the TCEP trap is slower when using the split so the timing of Relay A and C OFF will change compared to when not using Relay A. You will have to perform the timing experiment again to determine the best time to turn Relays A and C OFF.





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ASTM 4815 GC operation

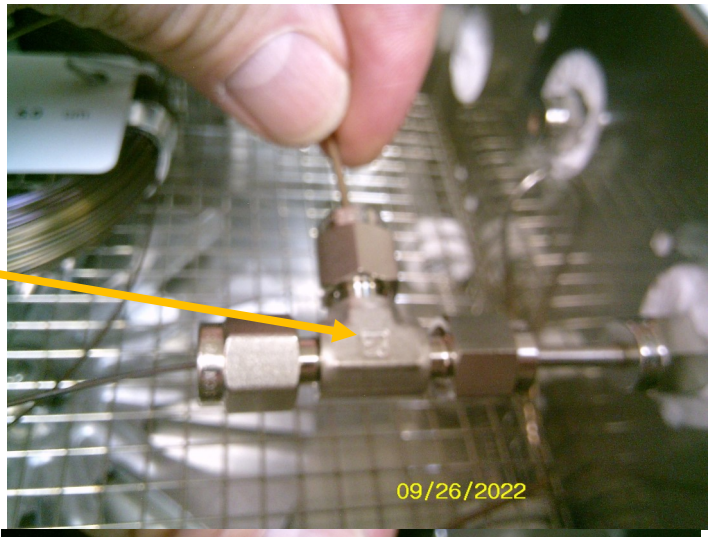
October 2022

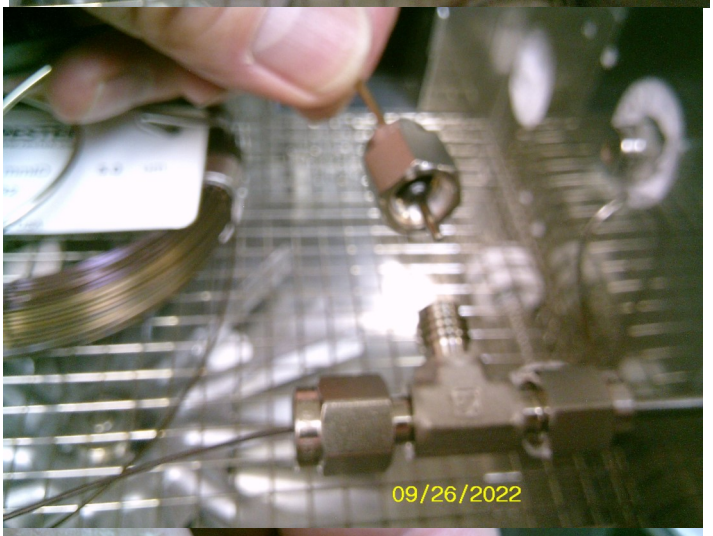
The exit of the TCEP trap is connected to a “tee” fitting in the column oven just up-stream of the FID detector inlet. The main column (60MXT1) is inserted through this “tee” and into the FID jet.

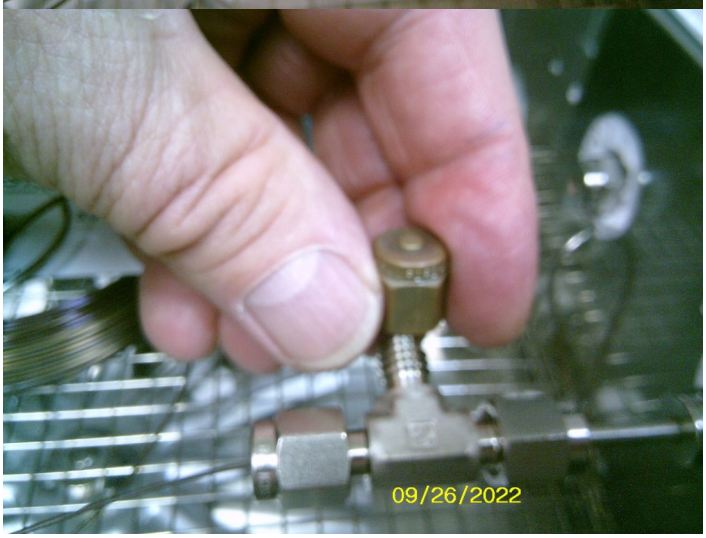
This is useful because you can see the peaks which blow through the TCEP and visualize the timing of Relay A and C.

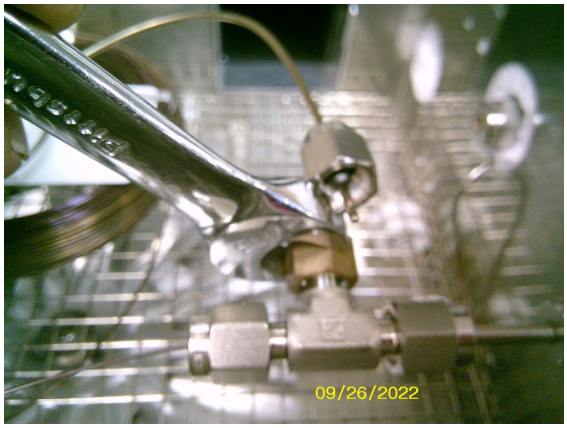
If you wish, you can remove this tube from the “tee” and replace it with a cap fitting as shown. But then you would not be able to know the relay A and C timing.

The FID baseline may be slightly cleaner with just the one flow of carrier gas however.









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