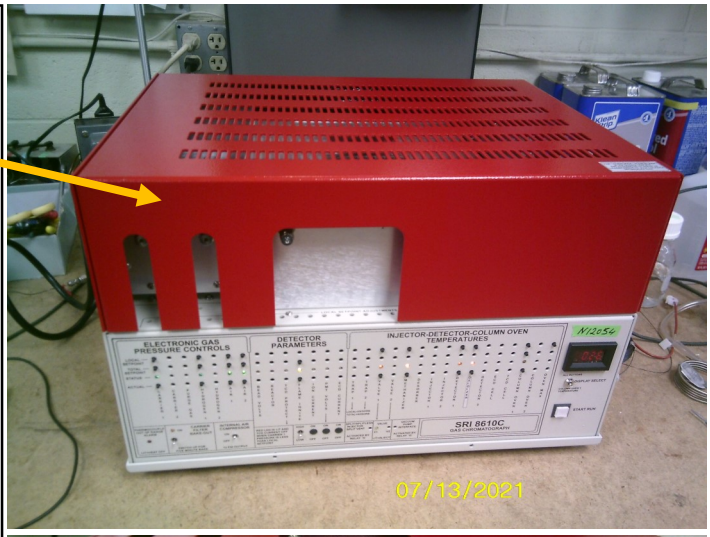
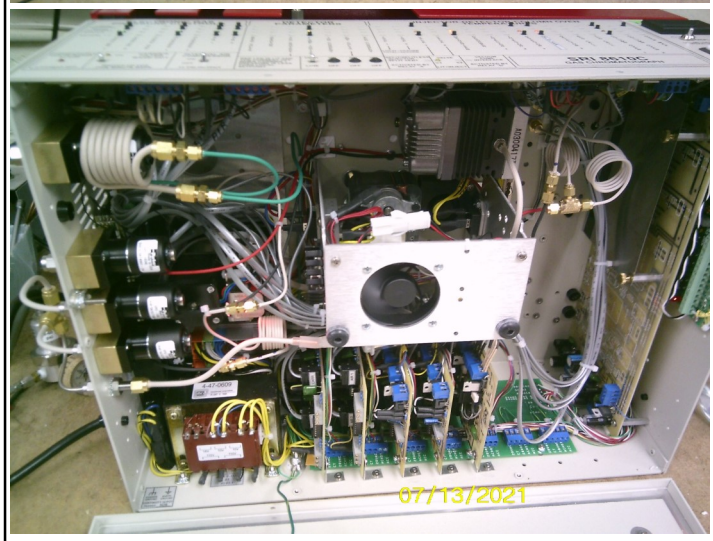
SCAQMD Method 25.3, EPA Method 25
GC with Oxidizer

The SRI Method 25.3 GC with Oxidizer (part# 8610-1025) is shown in the photo to the right.

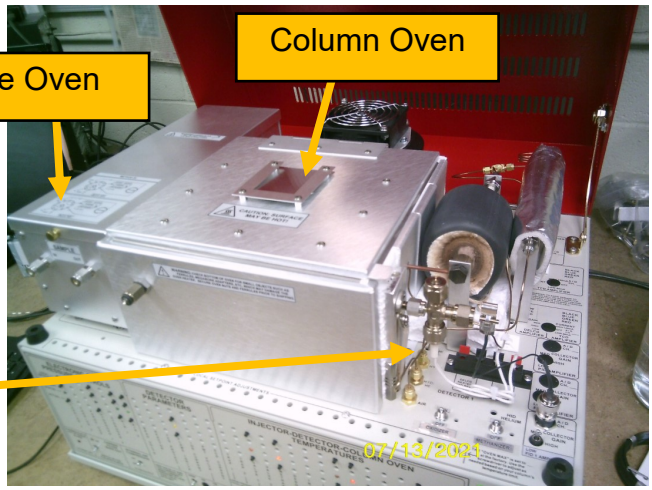
List price for the SRI Method 25.3 GC is $24,308. ***(2022 pricing, prices subject to change, consult most recent price list.)***



This is the inside of the GC. With the GC resting on its back, it's easy to work on if ever needed. You can see it is packed with gas controls and electronics.



This is what it looks like with the red lid raised up. When the lid is UP, all the heaters are turned off by an interlock switch actuated by the lid.



SRI Tech Support: 310-214-5092

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Inside the column oven are two 1/8” metal packed columns coupled in series

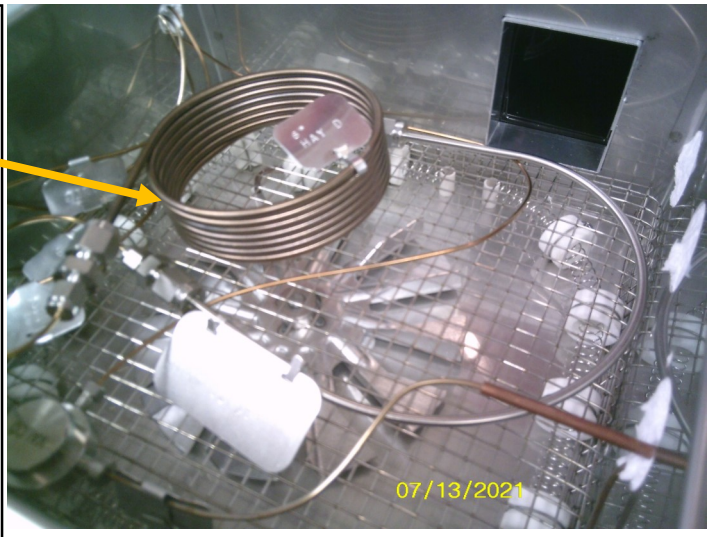
1 foot Tenax GR

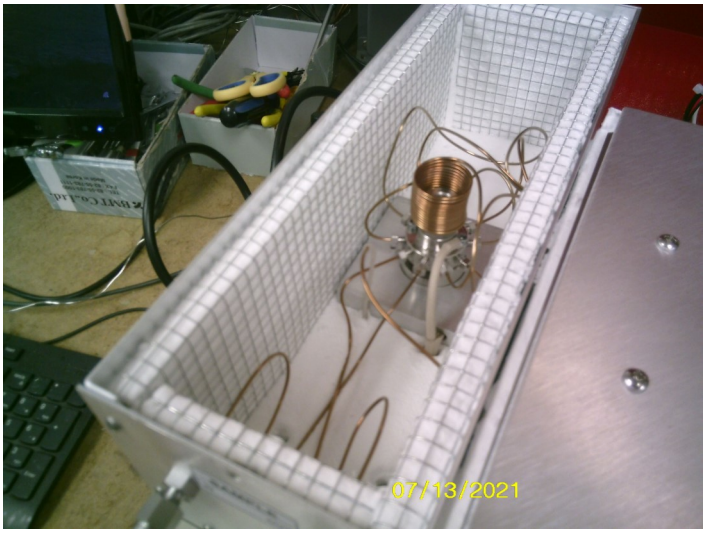
6 foot Haysep D

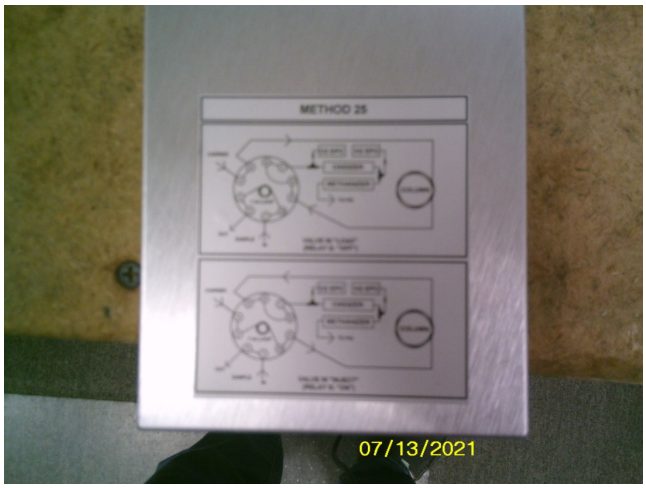
The Tenax-GR catches most molecules higher boiling than hexane (69C) while the Haysep D column separates CO, CH4, CO2, C2H2+C2H4 (as a single combined peak) C2H6, Propane, and backflushes everything higher boiling under control of the user. The Tenax-GR column releases the high boiling molecules better than the Haysep D would during the backflush period of the analysis.

The valve oven contains a two position 10 port gas sampling valve which injects the contents of a 1ml loop and also backflushes the column when its position is changes.

A map of the valve plumbing is printed on the cover of the valve oven so its easier to follow/understand the flow of gases through the system.







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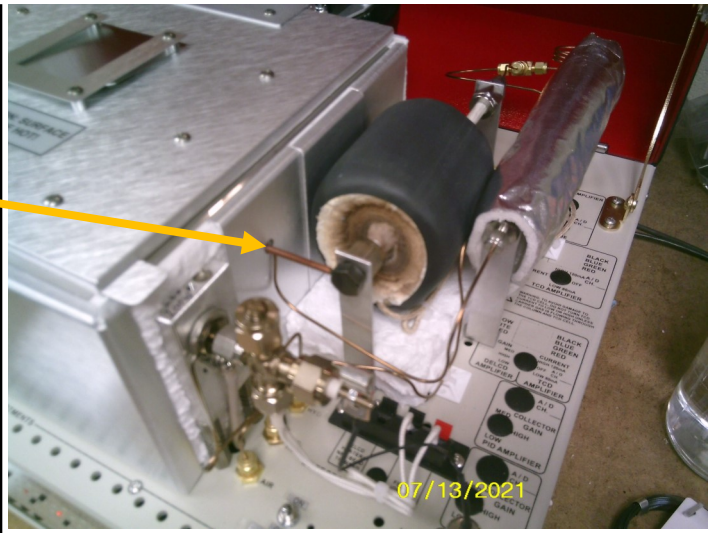
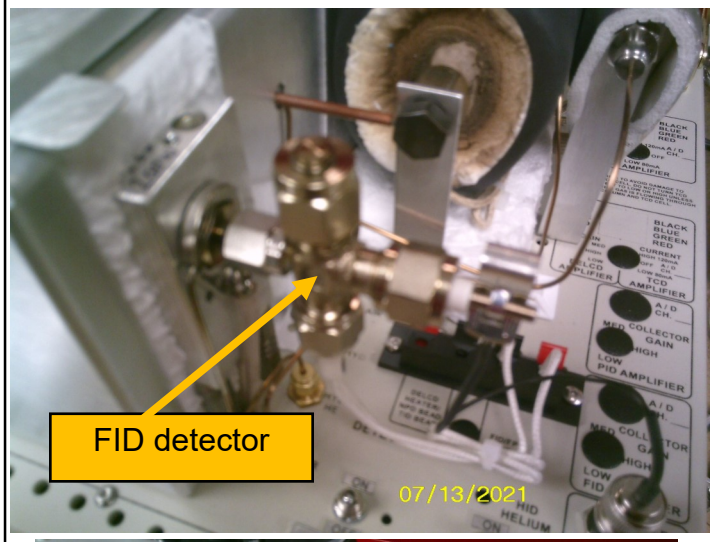
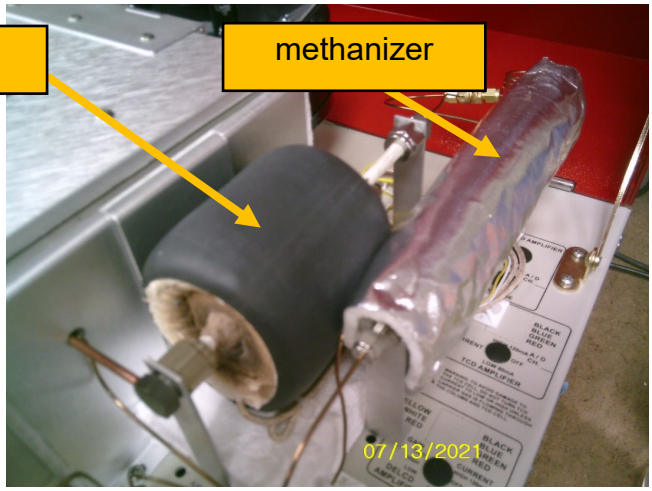
The FID detector, oxidizer and methanizer are located to the right of the column oven.



The FID (flame ionization detector) looks like a stainless steel “CROSS” fitting. It is shown in the photo with its insulation cover removed. A tiny hydrogen/air flame burning in the very center of the fitting ionizes any hydrocarbon that burns in the flame. The electronics measure the number of ions (electrons) that result.

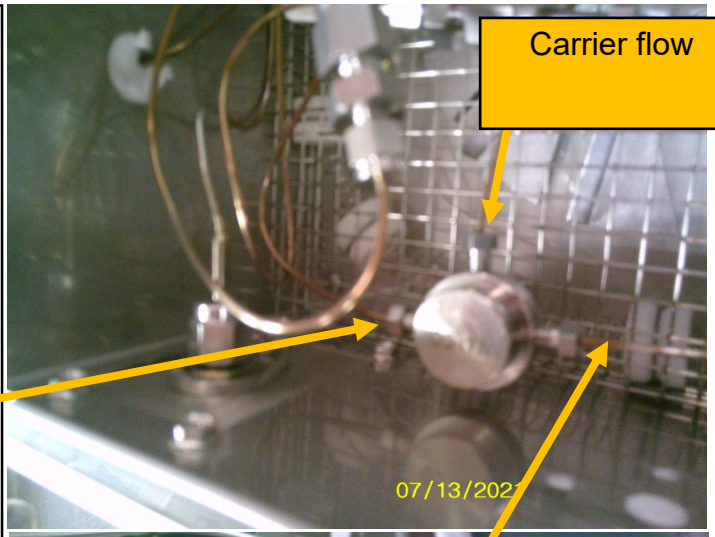




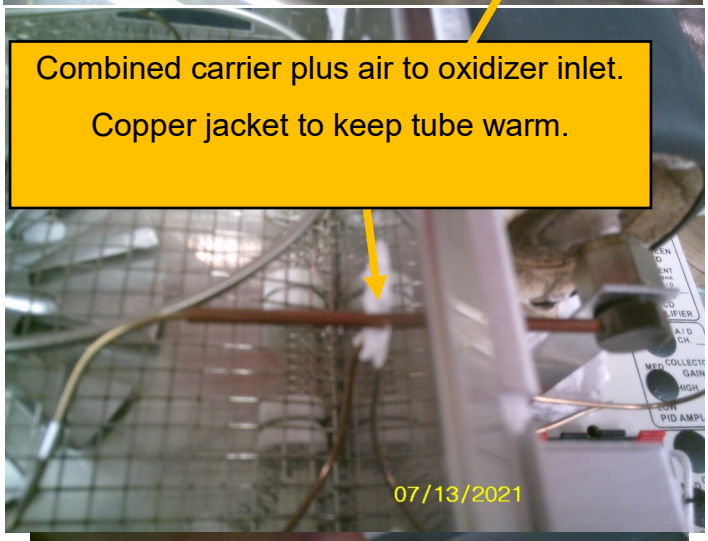
The oxidizer and methanizer (reducer) are mounted for convenient access near the FID detector. Both are heavily insulated because they operate at high temperature.

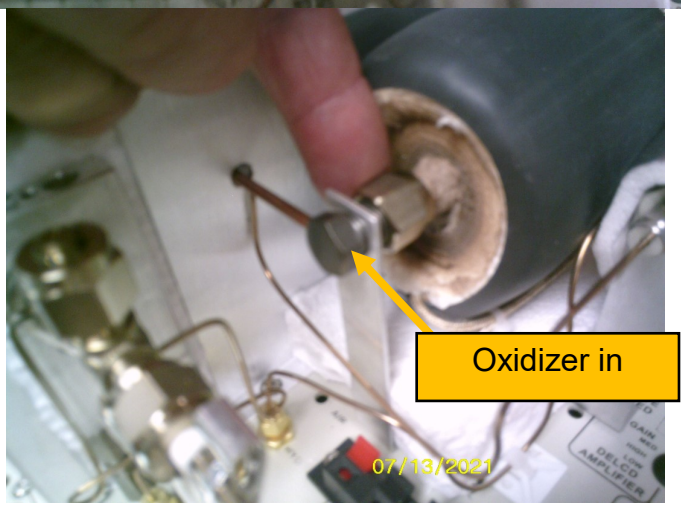
SCAQMD Method 25.3, EPA Method 25
GC with Oxidizer

The carrier flow exits the column and then connects to this 1/16” “tee” inside the column oven.



A flow of 1-2ml/minute of air (controlled by AIR EPC#2) joins the carrier gas and then flows through the oven wall to the inlet fitting of the oxidizer. To save space, this is a special side-entrance fitting supported by an aluminum bracket.





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GC with Oxidizer

The outlet of the oxidizer connects to a "1/16"tee" fitting where a flow of hydrogen mixes with the oxidizer exhaust (should be O2,N2, CO and CO2 mostly).

The copper tube is intended to catch any SO2 or ClO2 formed in the oxidizer.

The oxidizer is connected with 1/4" Swagelok nuts and 1/4" pure graphite ferrules.

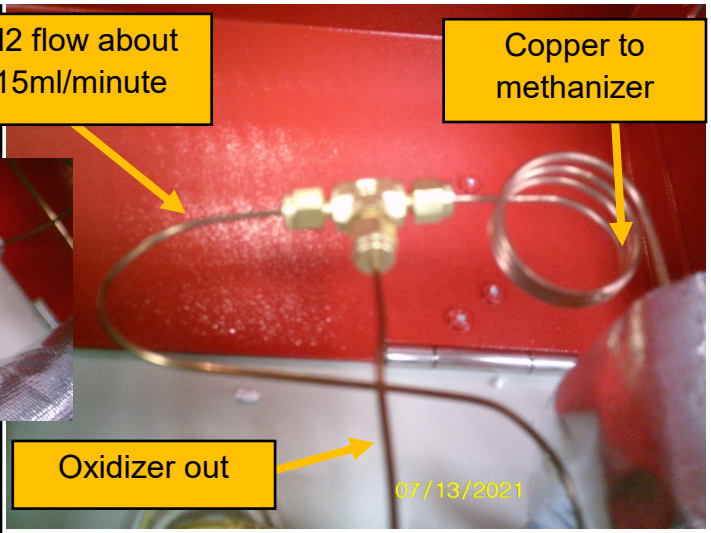
Since the oxidizer is made with a ceramic tube (withstands heat) you can't use metal ferrules or the tube will crack.

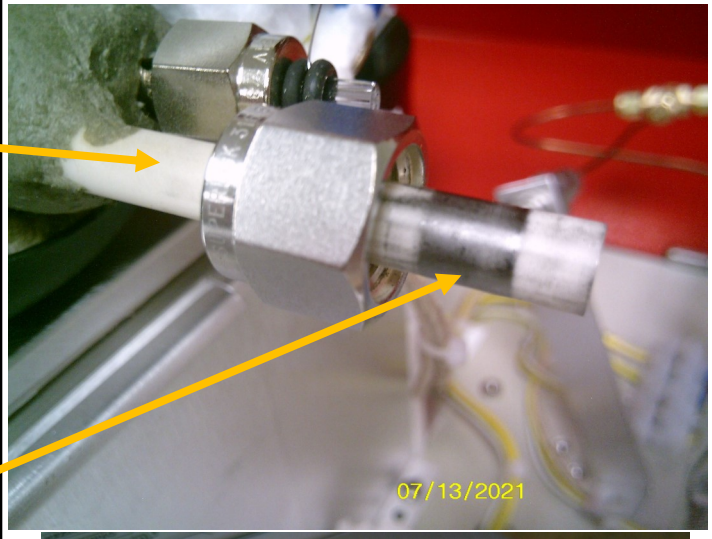
We use graphite ferrules because they are soft, seal well and withstand the high temperature operation.

Its important not to tighten the graphite ferrules too much .

You can see where the graphite ferrule has left a mark on the ceramic tube

Even though the upstream end of the column may be under 20 psi of pressure, the downstream end of the column, oxidizer and methanizer are very close to ambient pressure so you don't have to tighten vey much to stop any leak.





A new 1/4" Swagelok nut and graphite ferrule

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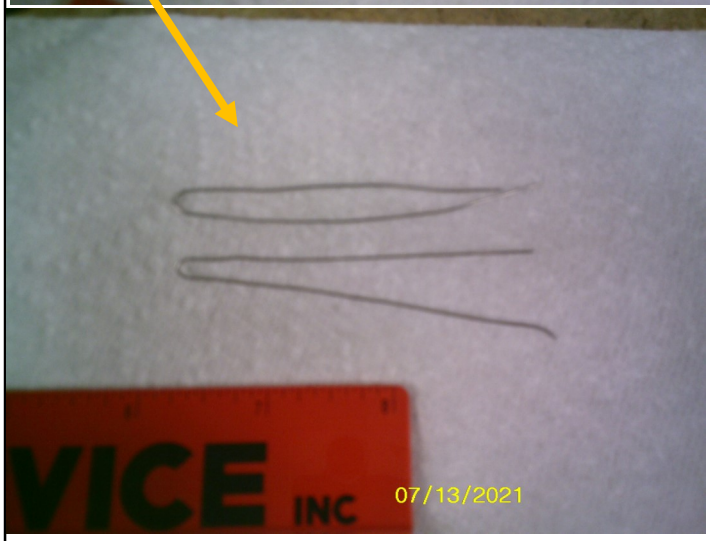
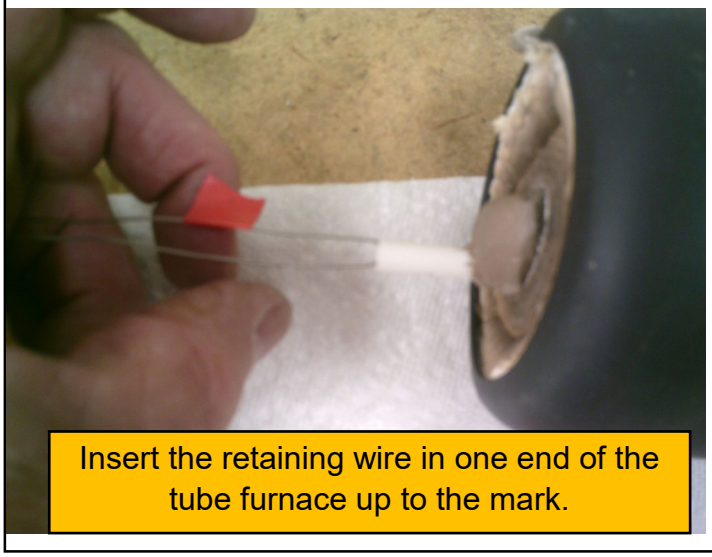
GC with Oxidizer

Inside the oxidizer is 12 of the chromia catalyst pellets. SRI will normally pre-install the catalyst so these photos show how we do it in case the tube heater ever needs to be replaced. The catalyst pellets are re-usable, seemingly forever.

Bend two 6inch long 22 gage nicrome wires in a U shape as shown in the photo

Use a piece of tape to mark a depth of 1 inch and another at 2 inches. These wires will secure the pellets in the middle of the tube heater. Its important that all the pellets are hot or the final peak shape will not look good. The wires ensure all the pellets are in the hottest part of the tube furnace. Adjust the bend in the wires so they make just a little friction fit inside the alumina tube. Don't force the fit and break the tube.





Insert the retaining wire in one end of the tube furnace up to the mark.

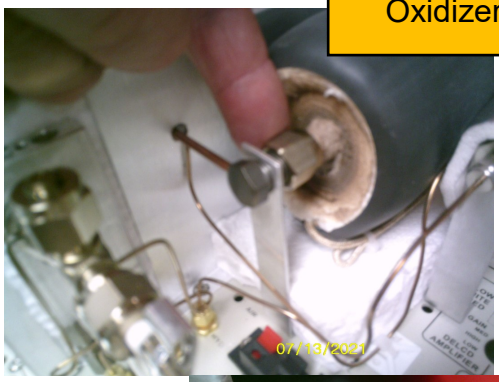
SCAQMD Method 25.3, EPA Method 25
GC with Oxidizer

Drop the chromia pellets into the tube furnace one by one, 12 in total. They are a loose fit so they just slide in.

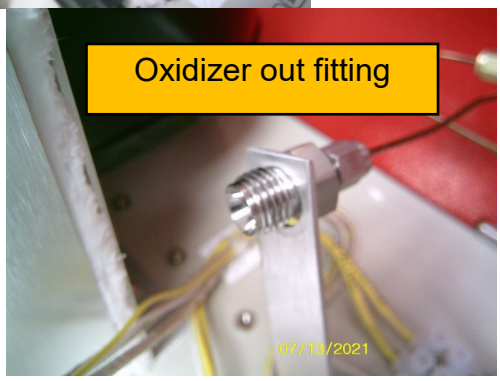
Insert the other retaining wire and clip off the wires sticking out of each end.

Reinstall the Tube furnace tightening the graphite ferrules first by hand/finger tight and then a final partial turn with two wrenches. Make sure to hold both side of the connection with a wrench and try to feel as the soft graphite ferrule gets just a little squish.

Oxidizer in fitting



Oxidizer out fitting



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GC with Oxidizer

Connect the carrier gas to the GC. Normally this would be nitrogen with the cylinder pressure regulator set to 30-40psi.

Inside the GC the electronic pressure regulator (EPC) re-regulates the cylinder pressure to a slightly lower value (typically 20 psi). This is set via screwdriver adjustment on the GC front panel.

Connect hydrogen (also at 30-40 psi) to the Hydrogen inlet fitting.

Some GCs may be equipped with a built-in air compressor in which case no air cylinder is required. If you want to substitute cylinder air (at 10-30 psi) you can do so by connecting to the “tee” fitting.

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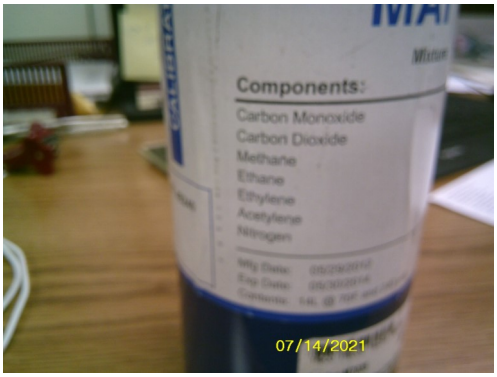
GC with Oxidizer

Verify the FID flame is lit but with the oxidizer and methanizer heaters turned off.

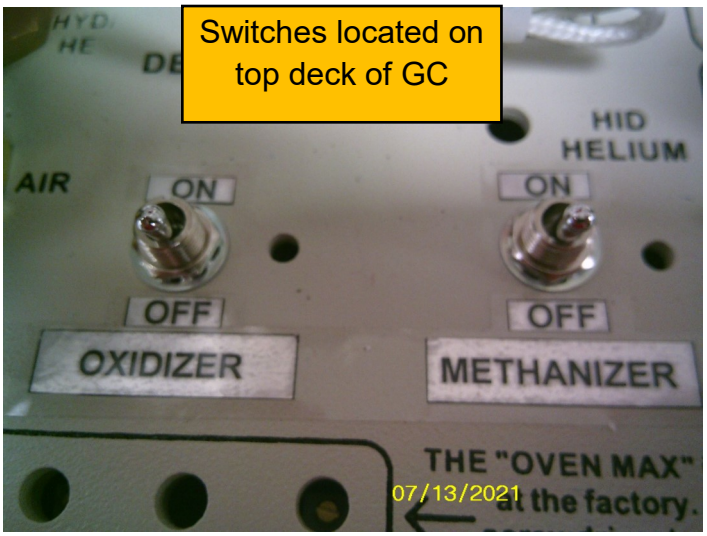
Setup the column temperature program and event table as you wish. We used the conditions shown at right

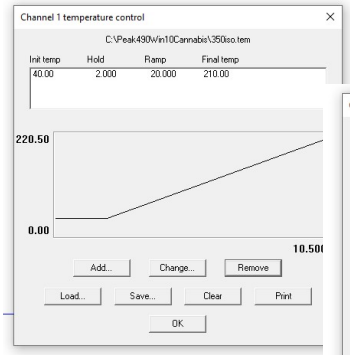
The calibration standard we used was 1% each.

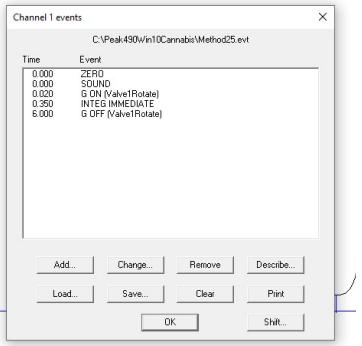


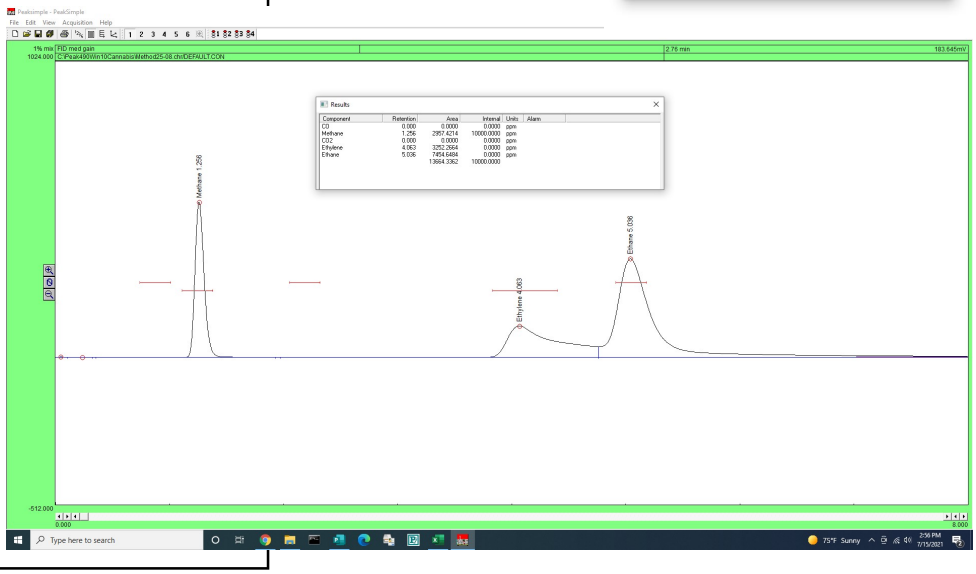
With the oxidizer= 35 and the methanizer=35 (both turned off, so at about ambient temperature), the chromatogram at right resulted.

We calibrated on the sharp and symmetrical methane peak to make it read 10,000ppm. The ethylene and ethane looked like they were partially absorbed by the cold catalyst. CO and CO2 were not detected.









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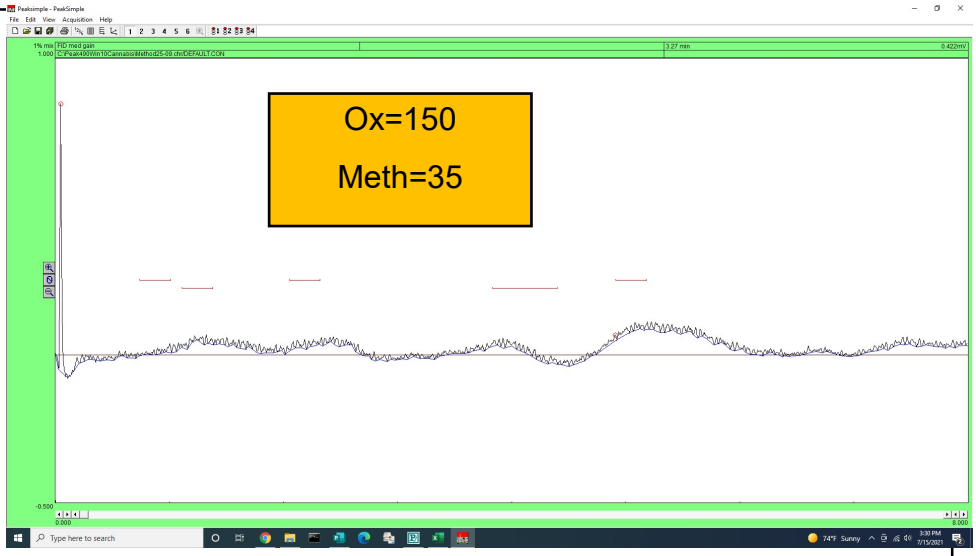
GC with Oxidizer

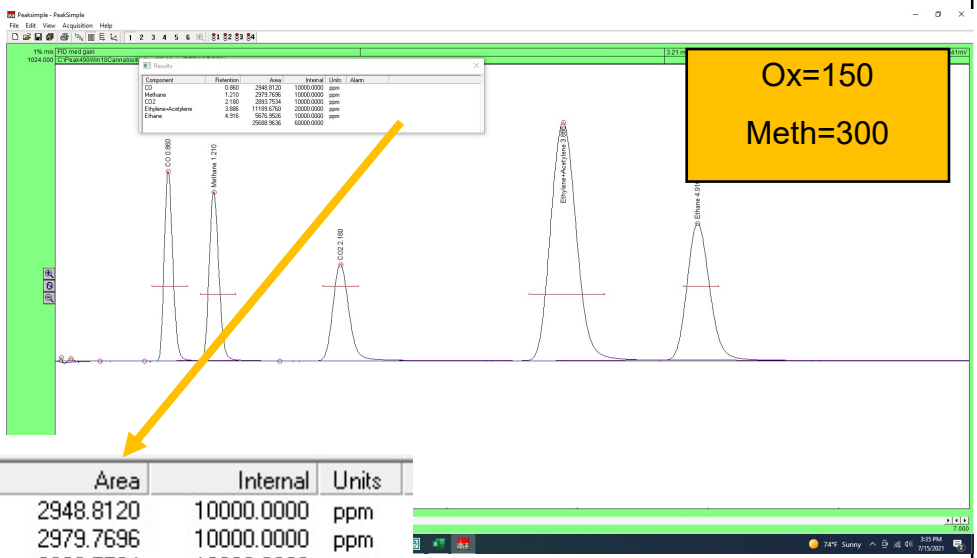
The same 1% mix was injected after the oxidizer temperature was increased to a reading of 150 by turning on the oxidizer heater switch. No peaks were detected showing all the sample had been 100% oxidized to CO or CO2.



The oxidizer temperature reads low by a factor of 4 lower than what it really is. So a reading of 150 really mean the oxidizer temperature is 600C.

The reason we do this is because all the other thermocouple circuits which measure temperature in the GC are calibrated for a Type K thermocouple. The same circuit is used to control the temperature of the oxidizer, but it has a platinum/rhodium thermocouple which has a different temperature coefficient

The same sample was injected with the methanizer reading 300C. All the peaks in the sample including CO and CO2 were detected. Note the similar peak areas for CO, CH4 and CO2 (1 carbon each) , and the proportionally larger areas for acetylene/ethylene and ethane (2 carbons each) indicating the oxidation/ reduction reactions were working close to 100%.



Component	Retention	Area	Internal	Units
CO	0.860	2948.8120	10000.0000	ppm
Methane	1.210	2979.7696	10000.0000	ppm
CO2	2.180	2893.7534	10000.0000	ppm
Ethylene+Acetylene	3.886	11189.6760	20000.0000	ppm
Ethane	4.916	5676.9526	10000.0000	ppm
		25688.9636	60000.0000	

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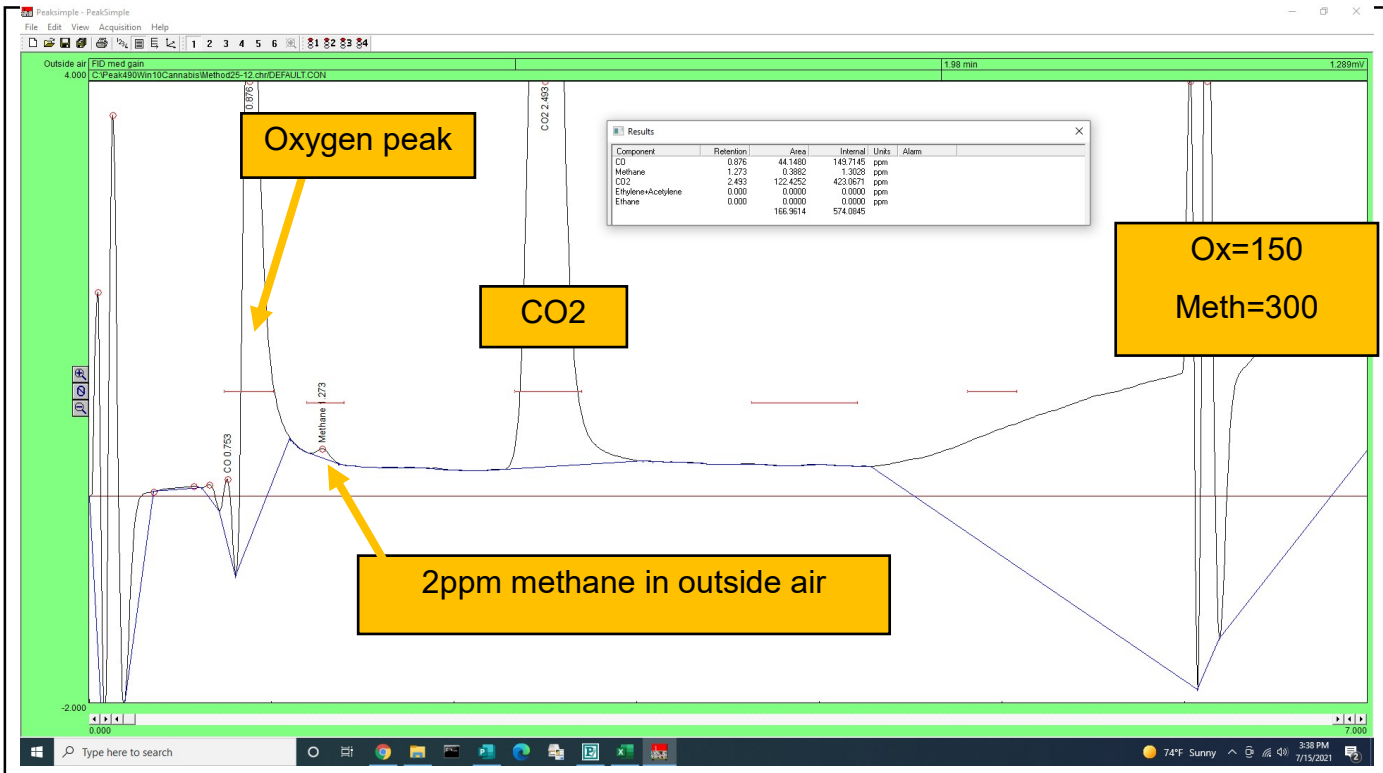
GC with Oxidizer



This time we injected outside air to demonstrate a detection limit for 1ppm or less of methane.

Notice the large peak just before methane. While oxygen should not in theory be detectible by the FID, it reacts with residual carbon in the methanizer to make CO and then with the nickel and hydrogen to make methane. The size of the oxygen peak gradually diminishes with time as the carbon is used up, but never goes away entirely.

This is a weakness in the method as it now stands, because low concentrations of CO will not be detected because the CO peak is obscured by the oxygen peak. Its is possible to rectify this deficiency by adding another valve and molesieve column to the GC. The Molesieve column provides complete separation of oxygen and CO, but adds to the cost of the GC and also makes the runtime a few minutes longer.