

CTR1 Replacement Column for Fixed Gas analysis

May 2013

Separation of Oxygen, Nitrogen, Methane, CO and CO₂ has been difficult since there is no single column material which is good for all these molecules at room temperature or above. In the past, one column vendor sold a “column in a column” called a CTR1 which is shown at right. This is a large bulky column consisting of an outer 1/4 od column and an inner 1/8 od column.

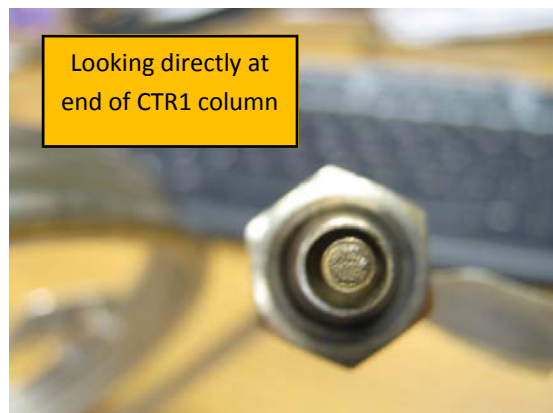
This column is no longer manufactured, or is not easily available, so SRI offers an equivalent or better column for the convenience of our customers.

8600– PKC7 “Fixed Gas Column”

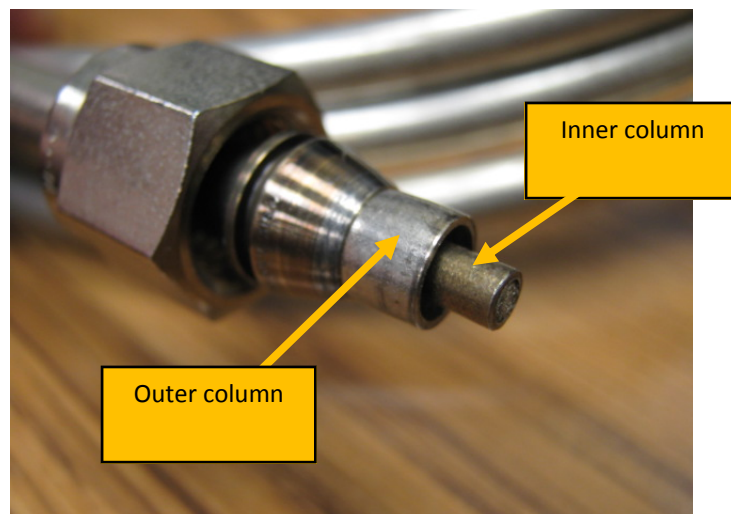
US\$ 659.00 August 2013 (price may change)

For a more robust method of separating these molecules as well as others like propane, propylene, butanes, pentane etc. Please see the MultipleGas#3 document on www.srigc.com

<http://www.srigc.com/MG3+SulfurFeb2013.pdf>



Looking directly at end of CTR1 column



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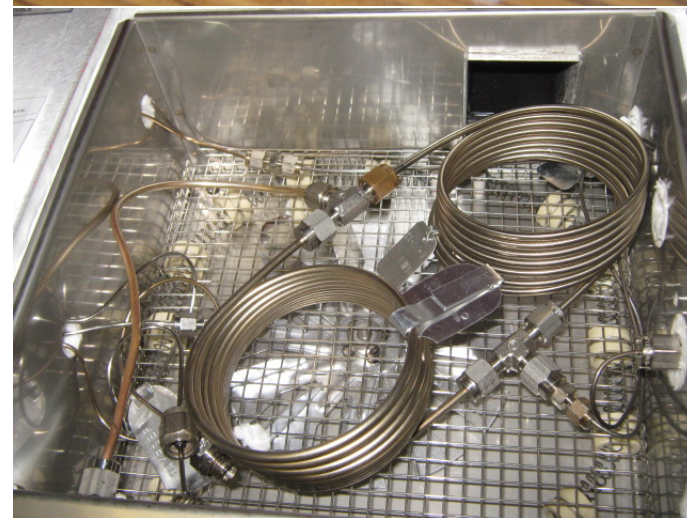
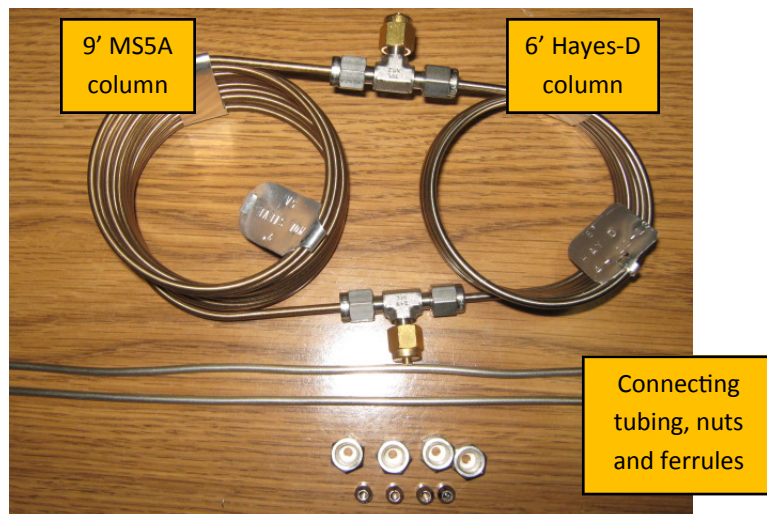
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The SRI “ Fixed Gas Column” (FG) consists of two side by side columns rather than one column inside another. This arrangement is superior because the columns have different bake out temperatures and having them separable makes it easier to bake out the Molecular Sieve column (300C+)without damaging the lower temperature Hayesep-D (270C max) column.

The column is supplied with two 12” lengths of flexible 1/8”od nickel tubing to make connecting it easier and extra nuts and ferrules. We like the soft graphite ferrules for this application because they seal well and do not deteriorate at the 300C bake-out temperature. However metal ferrules can also be used.

The “ Fixed Gas Column” is shown installed in an SRI 8610C GC column oven. There is still room for other columns.



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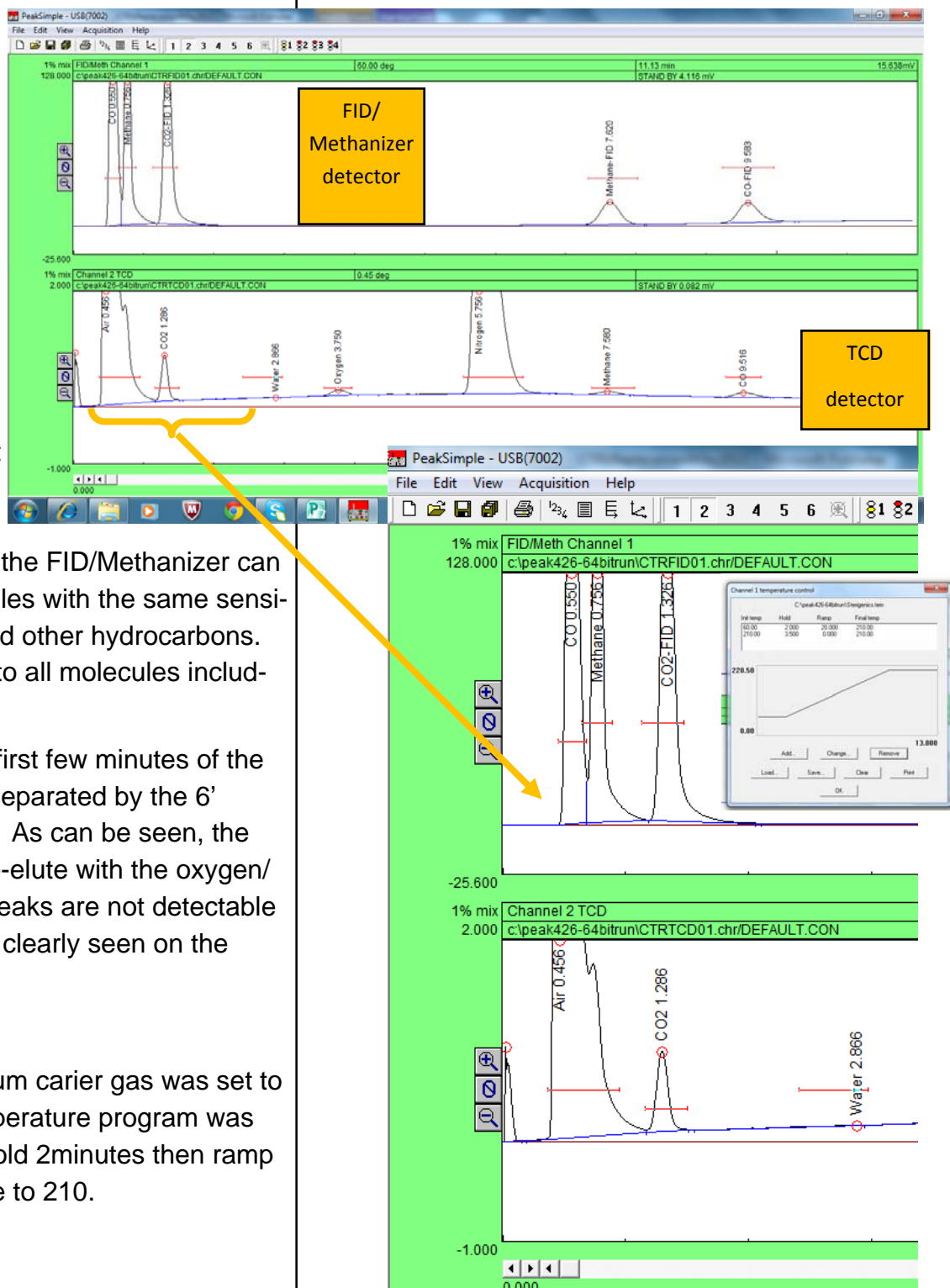
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The FG column was installed in an SRI 8610C GC equipped with a TCD detector and FID/Methanizer (FIDM) detector. The two detectors were plumbed in series so some peaks are detected by both detectors. The methanizer part of the FID detector converts CO and CO₂ to methane so the FID/Methanizer can detect those molecules with the same sensitivity as methane and other hydrocarbons. The TCD responds to all molecules including water.

All the peaks in the first few minutes of the chromatogram are separated by the 6' Hayesep-D column. As can be seen, the CO and Methane co-elute with the oxygen/nitrogen, so these peaks are not detectable by the TCD, but are clearly seen on the FIDM.

In this case the helium carrier gas was set to 20 PSI and the temperature program was set to start @60C hold 2minutes then ramp at 20degrees/minute to 210.



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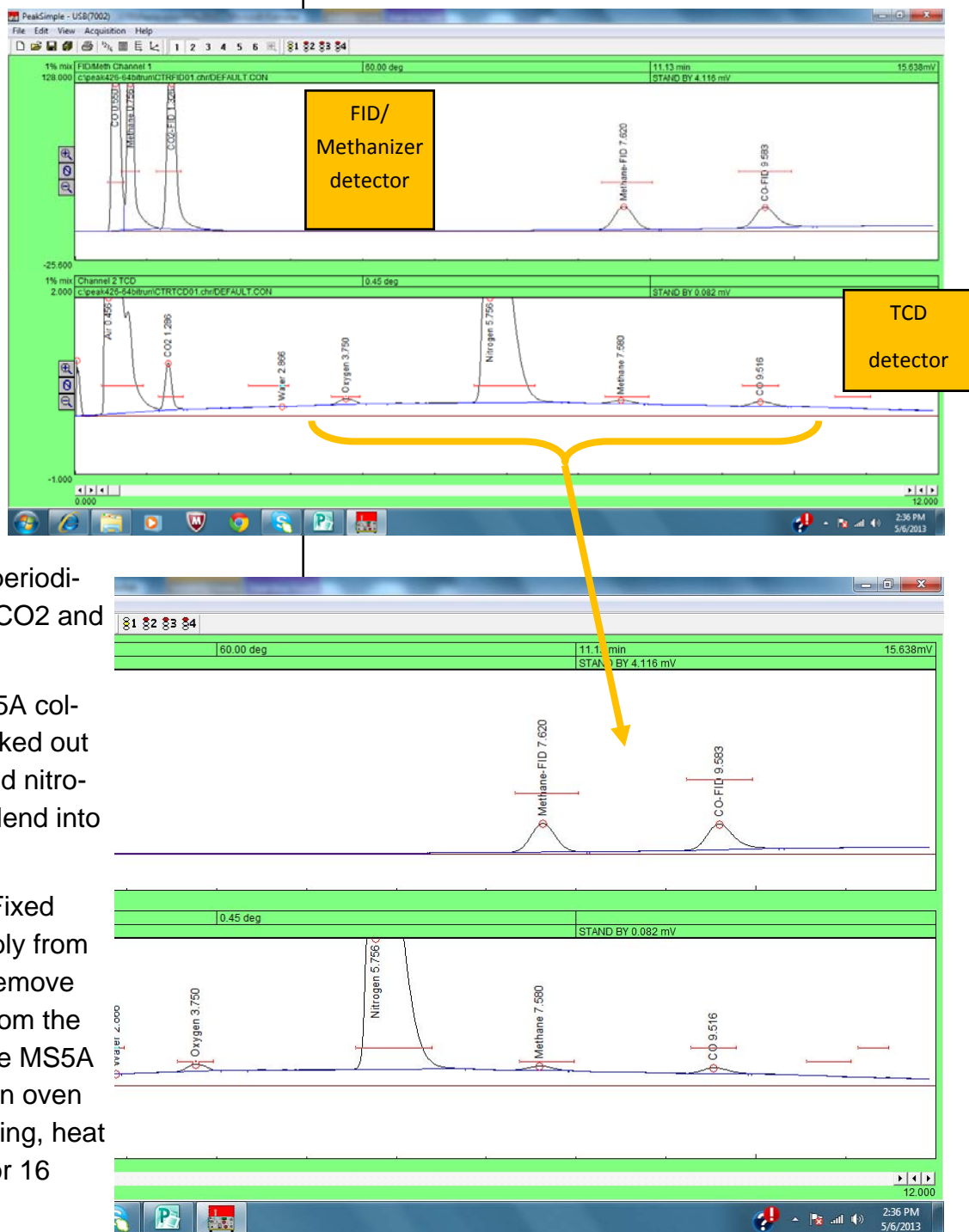
The peaks in the last minutes of the chromatogram are from the 9' Mole-Sieve 5A column.

This column separates oxygen from Nitrogen as well as methane and CO.

CO₂ however and water are permanently absorbed by this column which must be baked out periodically to remove the CO₂ and water.

You can tell the MS5A column needs to be baked out when the oxygen and nitrogen peaks start to blend into each other.

Remove the entire Fixed Gas column assembly from the oven and then remove the MS5A column from the assembly. Install the MS5A column in the column oven and with carrier flowing, heat the MS5A column for 16 hours at 300C.



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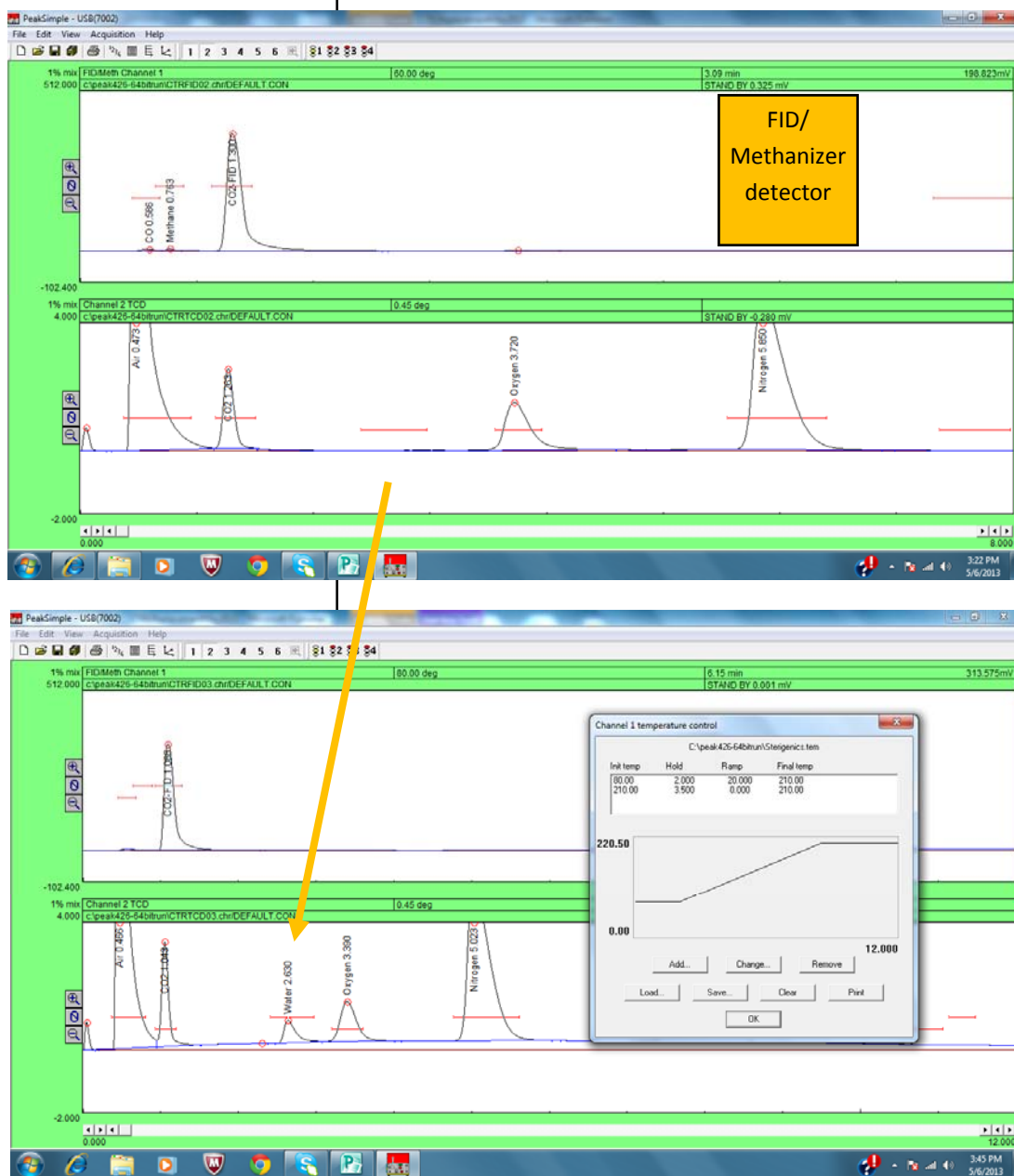
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Shown at right is a chromatogram of 1ml exhaled breath using the 60C starting temperature.

Compare this chromatogram to the one below at a 80C starting temperature. The water peak in the top chromatogram co-elutes with the oxygen while at the 80C starting temperature, the lower chromatogram shows the water nicely separated from the oxygen.

There is less separation however between the CO2 and the air peak.



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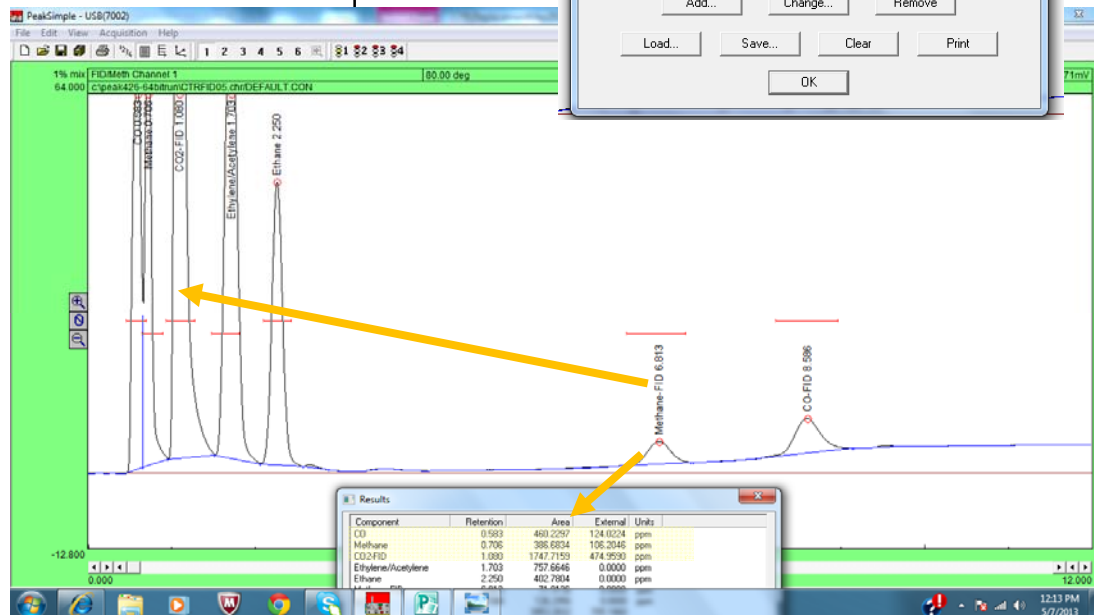
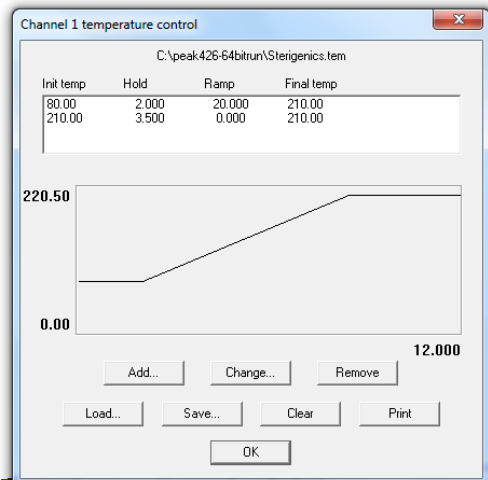
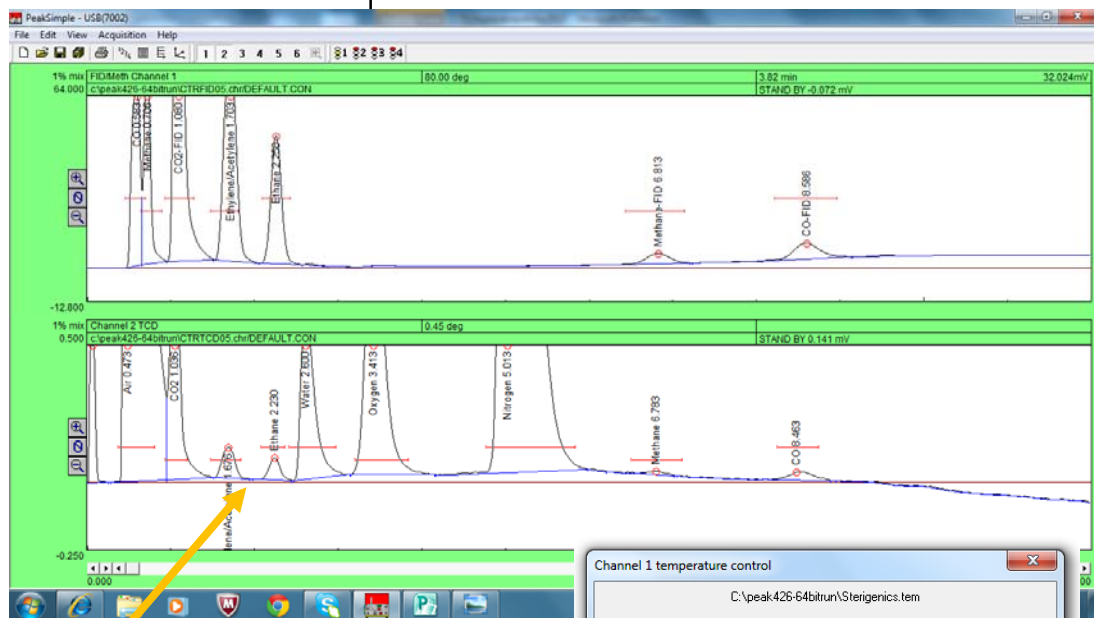
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Shown at right is a chromatogram of 1ml 1% gas mix including ethylene/acetylene and ethane using the 80C starting temperature oven program.

Between the TCD and FID/ Methanizer CO, CH₄, CO, Ethane, Ethylene/Acetylene, Water, Oxygen and Nitrogen are all resolved.

Ethylene and acetylene co-elute, but are separated from ethane and also water.

Note also that the split ratio between the columns is about 4:1 judging by the area counts of CO₂ (which elutes from the 6'Hayesep-D column) and the methane and CO (which elute from the 9'MS5A column).



Component	Retention	Area	External	Units
CO	0.583	460.2257	124.0224	ppm
Methane	0.705	385.6334	106.2045	ppm
CO2-FID	1.000	1747.7155	474.9530	ppm
Ethylene/Acetylene	1.703	757.6646	0.0000	ppm
Ethane	2.250	402.7884	0.0000	ppm



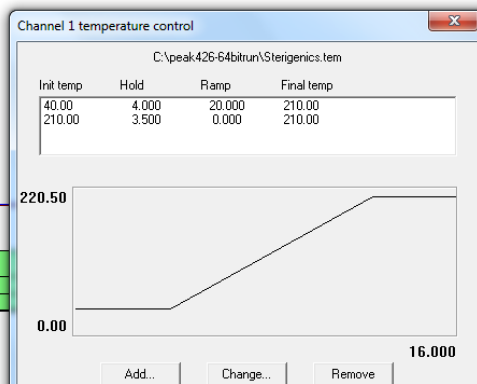
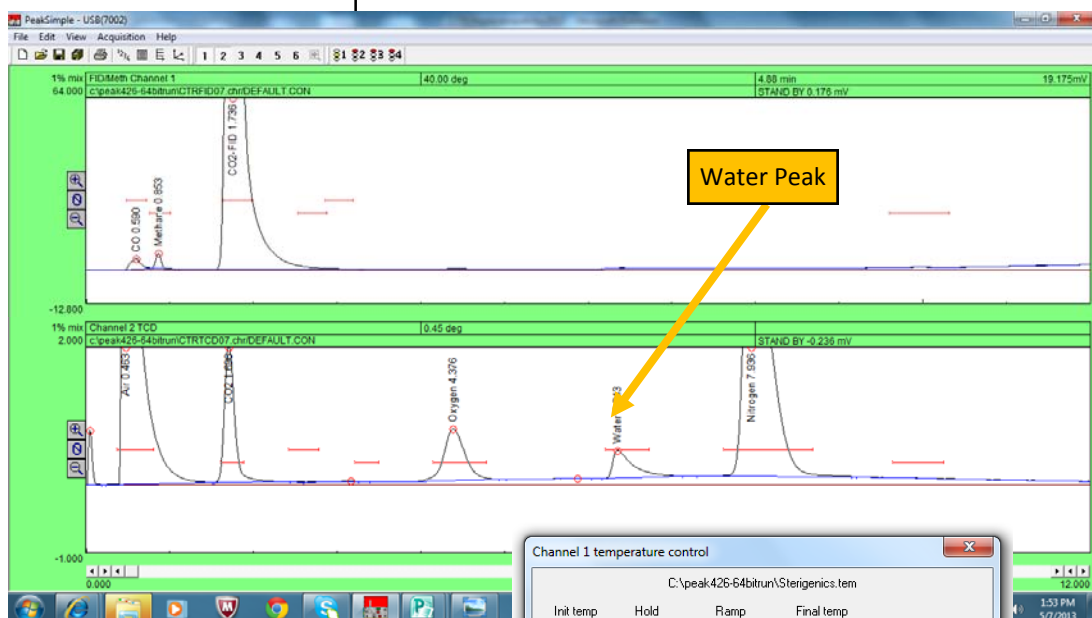
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Shown at right is a chromatogram of exhaled breath starting at 40C.

You can see the water peak has shifted to the right and elutes between oxygen and nitrogen.

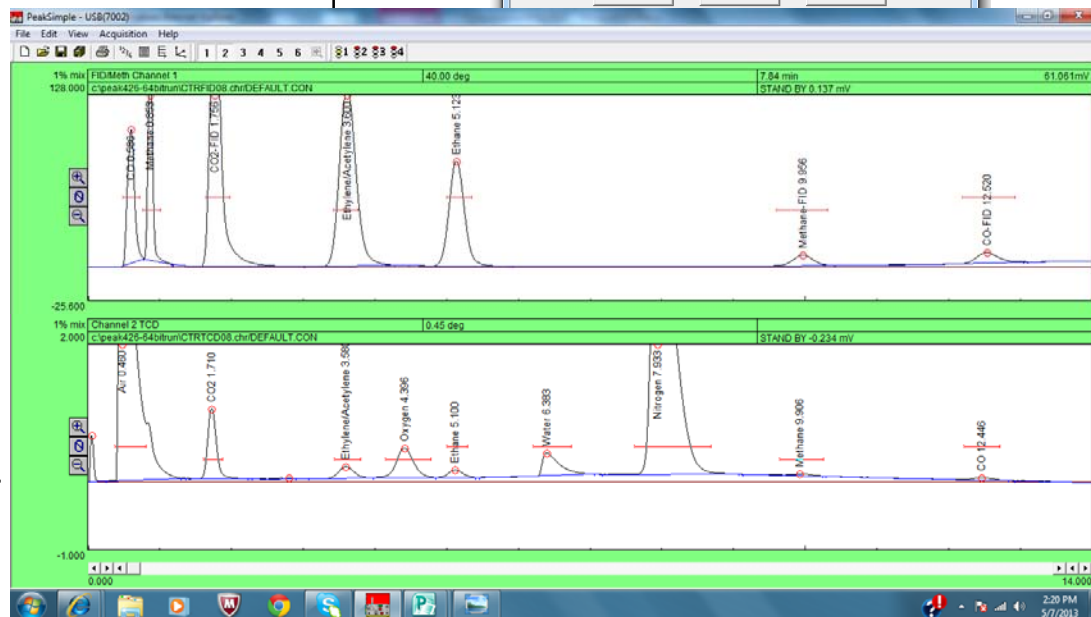


A mix of:

O2, N2, CO, CO2, methane, ethane and ethylene/acetylene plus water is shown starting at 40C. You

can see the peaks from the Haysep-D column are interspersed with the peaks from the MS5A column.

You can experiment with different temperature programs to best suit your particular mix of gases.



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