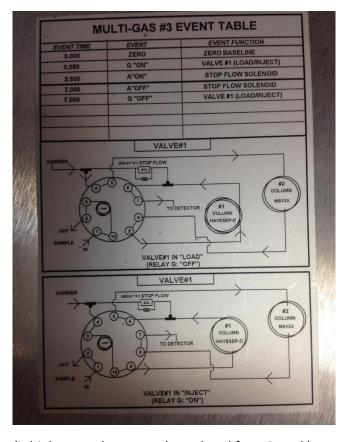
SRI 8610C GC Ann's Notes 11-17-2014

#### Introduction

This is the GC as it came from the manufacturer



This GC has two columns (I think new columns can be ordered from Restek)

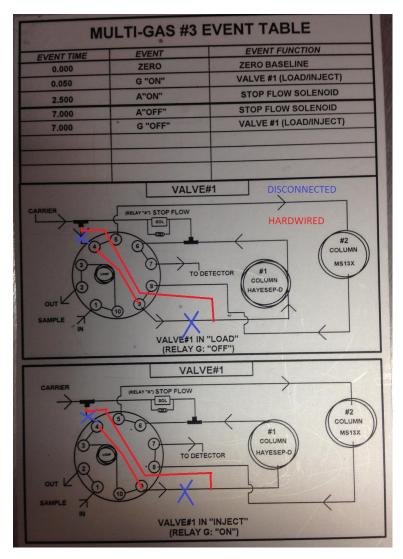
- 1. Hayesep D: Column 1
  - a. Separating CO, CO<sub>2</sub> and other gaseous carbon compounds ex. acetylene
- 2. Molecular sieve: Column 2
  - a. Separating extremely small gases
  - b. IMPORTANT: CO<sub>2</sub> and H<sub>2</sub>O cannot enter this column because the pore size is not big enough to allow them to pass through. They must be burned off the column at 200°C (limit of stability for molecular sieve) if they enter.

Because CO<sub>2</sub> cannot enter into the molecular sieve column, there is a switching mechanism between the columns to only allow certain gases to pass through the molecular sieve column. This switching mechanism is designated G "on" or G "off". When G is off the gas flows through only the hayesep D column. When G is on the sample flows through both columns.

In addition there is a solenoid valve that stop flow through the hayesep D column to allow the separation to occur on the molecular sieve column. When the solenoid valve is open, it allows the

carrier gas to pressurize both the inlet in the outlet of column A stopping the flow through this column. When A is off the solenoid is closed and gas flows through the hayesep D column. When A is on there is no flow through the hayesep D column.

The GC was intended to be operated in the mode where you constantly have sample flowing in and out. This does not work for us since we don't have enough sample and we want to use manual injection. The manual injection in deposited straight into the carrier gas. However, even when using manual injection you have to flow carrier gas through the sample loop to flush the remaining sample out. This had been verified now twice by Joerg that you get double peaks if you switch the G valve while injecting sample manually because there is still sample in the loop that then re-elutes on the next run. We didn't want to run carrier gas through the sample loop since it is expensive helium (Mass spec grade) so we rewired the GC. We rewired it to not have the sample loop in the flow. I relabeled the diagram on the GC below to detail how Joerg and I rewired the GC.



### The rewiring involved

1. Hardwiring the carrier gas to the inlet of column 1.

- 2. Hardwiring connection 4 to connection 9
  - a. In G off position column 2 is connected to itself so nothing will flow
  - b. In G on position the sample loop is connected to itself

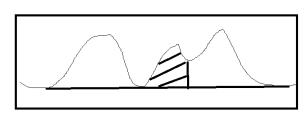
Based on this rewiring we have created two programs to measure gases. Ann's program is intended to measure  $CH_4$ ,  $CO_2$  and  $CO_3$ . Joreg's method is to measure  $CH_4$  and  $CO_3$ .

We edit the method every time because the software sucks. To edit the method go to Edit/Channel. Then click on the appropriate box to edit what you want. It should be noted you should change the name where the file will save by clicking the postrun box. You need to make sure the postrun box for channel 1 and 2 have different names or one will overwrite the other when saving. Also, the change the carrier flow you must manually do this by changing the value for the carrier 1 panel on the front of the GC.

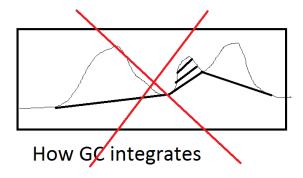
#### Random GC comments

When switching carrier gas make sure the run the new carrier gas through both columns and make sure all of the previous carrier gas has been purged before making measurements.

When integrating make sure to ingrate from the baseline and draw peaks as designated below. This involves manual integration because the GC is not programed to integrate this way.



How to properly integrate



The gas flows through the TCD detector followed by the FID since the sample is destructively measured in the FID. Therefore the peak for the TCD is 0.1 min earlier than the FID peak

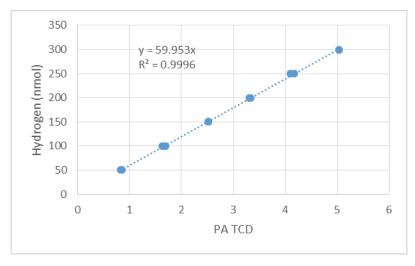
### Joerg's method

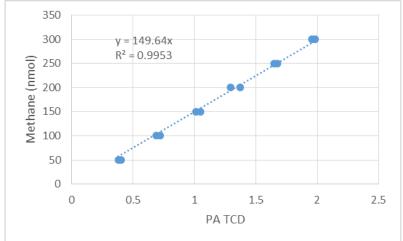
Carrier Gas: N₂ Carrier Flow: 13 Temperature: 40°C Run Time: 3 minutes FID sensitivity: medium Event File: G off entire time

**Elution Time** 

Compound	Elution Time TCD (min)
H <sub>2</sub>	0.516
CH <sub>4</sub>	1.130
CO <sub>2</sub>	2.553

# 1% CH<sub>4</sub>/1% H<sub>2</sub> Combined Standard Curve





## Ann's method

Carrier Gas: He
Carrier Flow: 13
Temperature: 40°C
Run Time: 10 minutes
FID sensitivity: medium

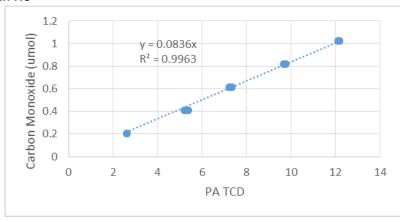
Detection: TCD works for high concentrations (in the percent range).

**Event File** 

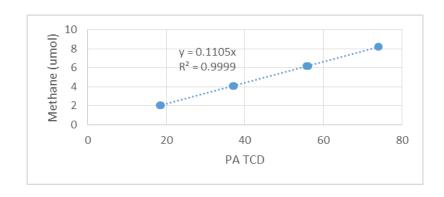
Time (min)	Event	Flow
0	G on	Flow through both columns
0	A off	
3.4	A on	Stop flow Hayesep D, flow through molecular sieve
6.7	G off	Flow through Hayesep D only
6.75	A off	Resume flow through Hayesep D

Compound	<b>Elution Time TCD</b>
CO	4.1
CH <sub>4</sub>	5.1
CO <sub>2</sub>	8.1

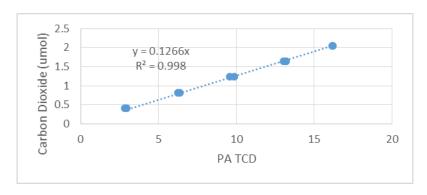
## 10% CO Standard in He



## 100% CH4



# 20% CO2 in N2



More information in the  $\underline{\text{manual}}$