Monitor the Gas Mixture with Gas Chromatograph

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Basic working principle of GC

A **gas chromatograph** is used for separating constituents in a gas mixture. It uses a flow-through narrow tube (*column)*, through which different constituents (gases) of a sample gas mixture in a gas stream (carrier gas, *such as Helium*) at different rates depending on their various chemical and physical properties and their interaction with a specific column filling, called the *stationary phase*. As the gases exit the end of the column, they are detected and identified. The function of the stationary phase in the column is to separate different components, causing each one to exit the column at a different time (*retention time*).

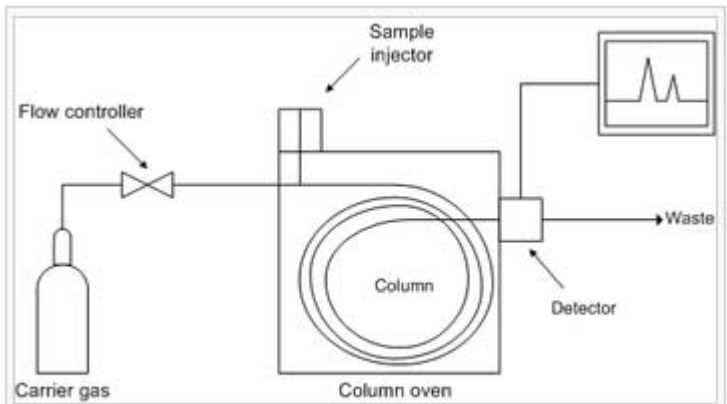
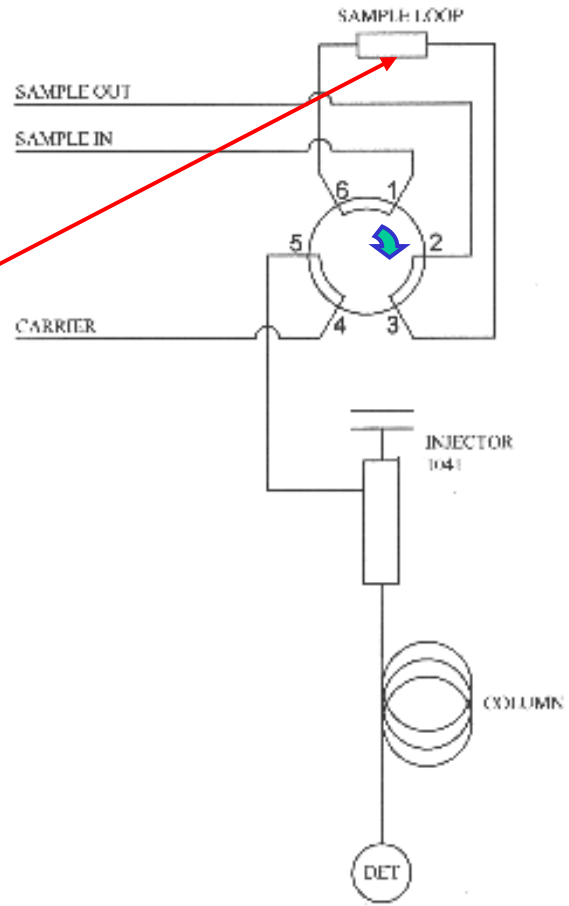


Diagram of a gas chromatograph.

Varian 3900GC Gas Chromatograph

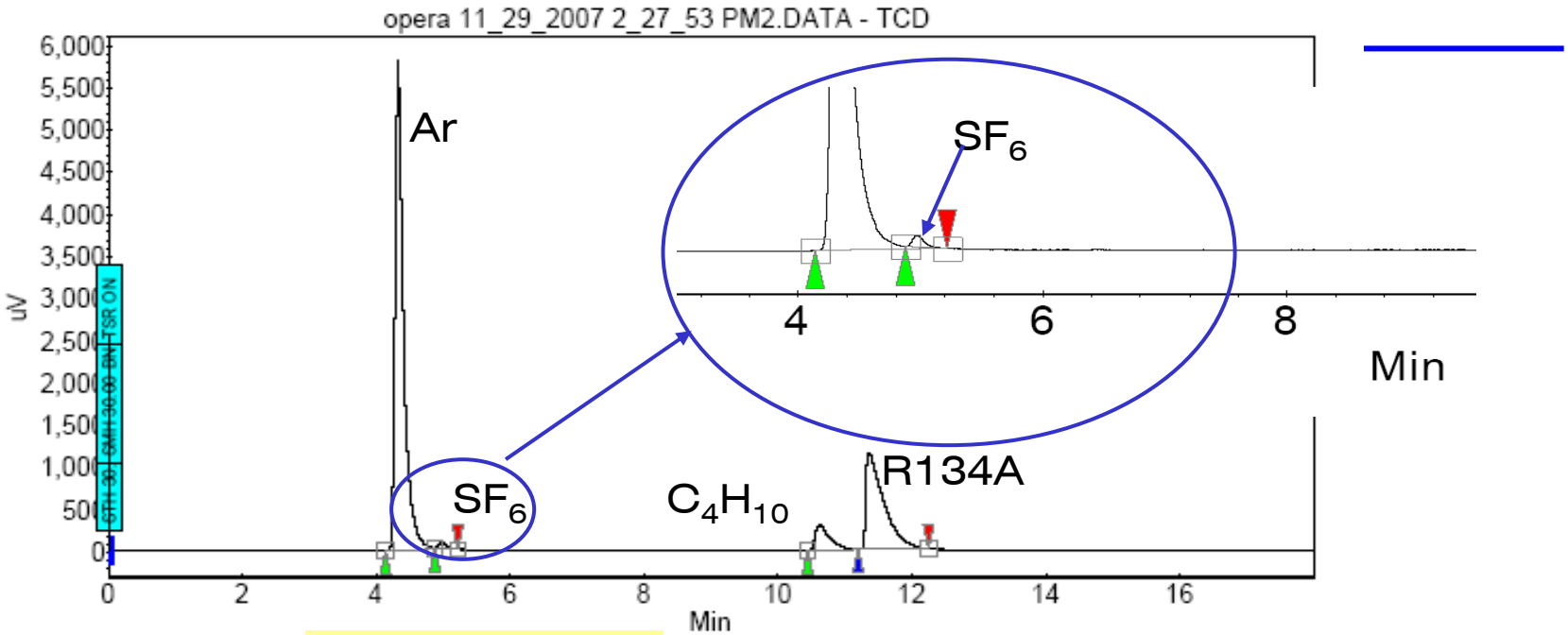
The GC system used in Princeton is Varian 3900GC, which is a single channel all electronic gas chromatograph, equipped with a 1041 on-column injector and a TCD (Thermal Conductivity) detector. It uses a fused silica capillary column, 30m x 0.53mm ID, coating Silicaplot. The sampling loop on its sample valve has 5μL volume.

(Xiaolan Luo of IHEP provided a lot of useful information on running their HP GC system, it was very helpful for setting up 3900GC system at Princeton.)



System : System_1
Method : opera-gas-11282007
User : Administrator

Acquired : 11/29/2007 2:29:16 PM
Processed : 11/29/2007 3:12:22 PM
Printed : 12/8/2007 11:04:56 AM



Area under peak

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.32	65.95	5786.4	848.1	65.946
2	UNKNOWN	4.97	0.71	86.3	9.1	0.709
3	UNKNOWN	10.62	5.89	293.3	75.8	5.893
4	UNKNOWN	11.36	27.45	1148.0	353.0	27.452
Total			100.00	7314.0	1286.0	100.000

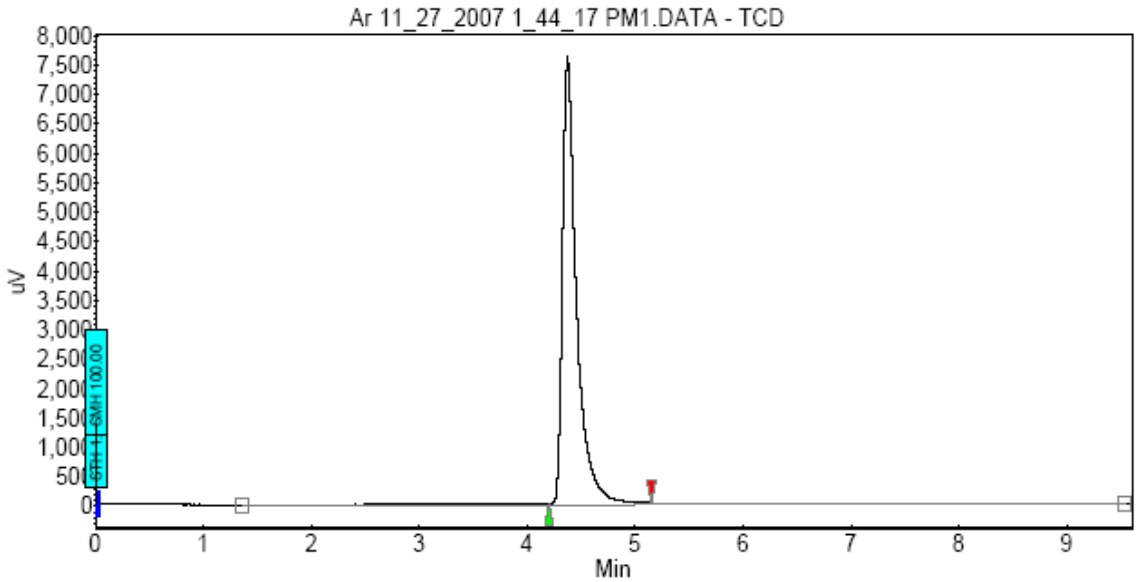
OPERA gas mixture, four peaks can be clearly seen: Ar, SF6, C4H10 and R134A. The different gas has different peak area, use pure gas to calibrate each peak.

Ar calibration chromatogram

Chromatogram : Ar 11_27_2007 1_44_17 PM1_channel1

System : System_1
Method : test-11192007B
User : Administrator

Acquired : 11/27/2007 1:46:09 PM
Processed : 11/27/2007 1:57:22 PM
Printed : 12/8/2007 11:26:54 AM



Peak results :

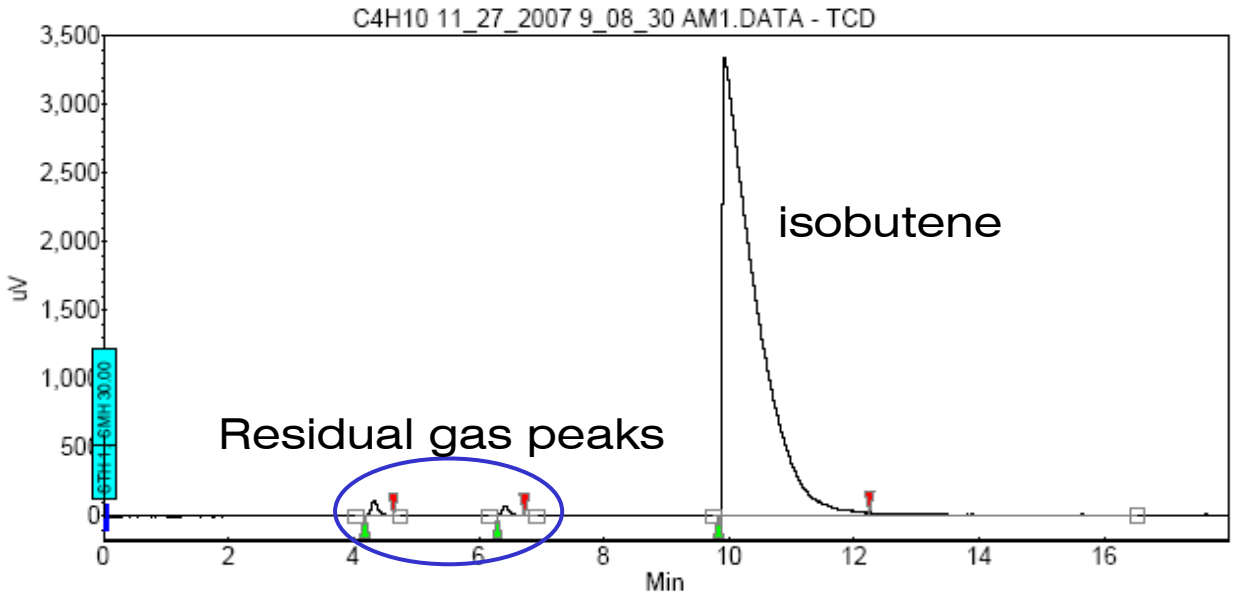
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.37	100.00	7609.8	1163.3	100.000
Total			100.00	7609.8	1163.3	100.000

C4H10 calibration chromatogram

Chromatogram : C4H10 11_27_2007 9_08_30 AM1_channel1

System : System_1
Method : test-11192007B
User : Administrator

Acquired : 11/27/2007 9:18:14 AM
Processed : 11/27/2007 10:38:20 AM
Printed : 12/8/2007 11:39:15 AM



Peak results :

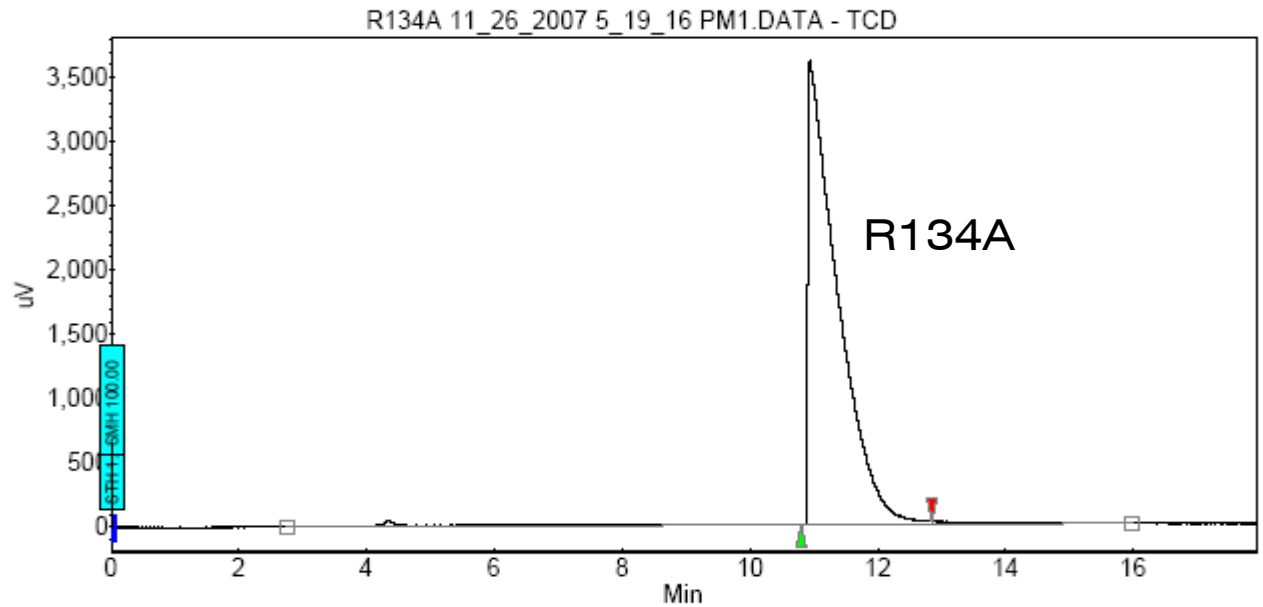
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.32	0.77	109.9	15.6	0.773
2	UNKNOWN	6.42	0.49	73.6	9.9	0.490
3	UNKNOWN	9.93	98.74	3337.7	1988.0	98.737
Total			100.00	3521.2	2013.4	100.000

R134A calibration chromatogram

Chromatogram : R134A 11_26_2007 5_19_16 PM1_channel1

System : System_1
Method : test-11192007B
User : Administrator

Acquired : 11/26/2007 5:20:32 PM
Processed : 11/26/2007 5:42:45 PM
Printed : 12/8/2007 11:28:03 AM



Peak results :

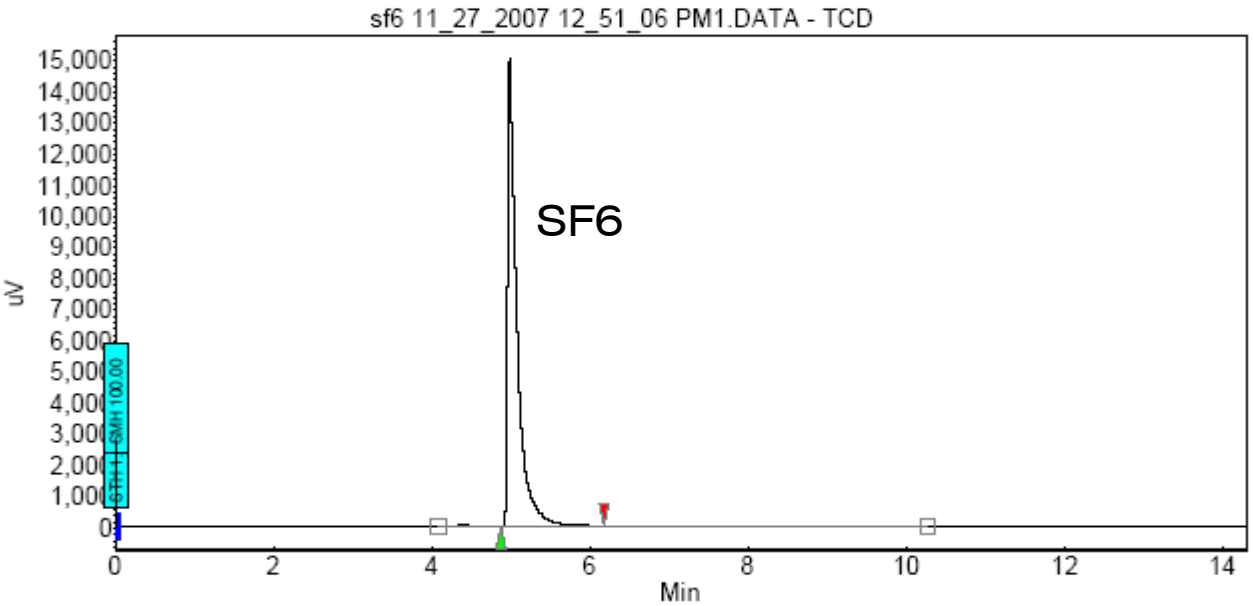
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	10.94	100.00	3609.6	1927.5	100.000
Total			100.00	3609.6	1927.5	100.000

SF6 calibration chromatogram

Chromatogram : sf6 11_27_2007 12_51_06 PM1_channel1

System : System_1
Method : test-11192007B
User : Administrator

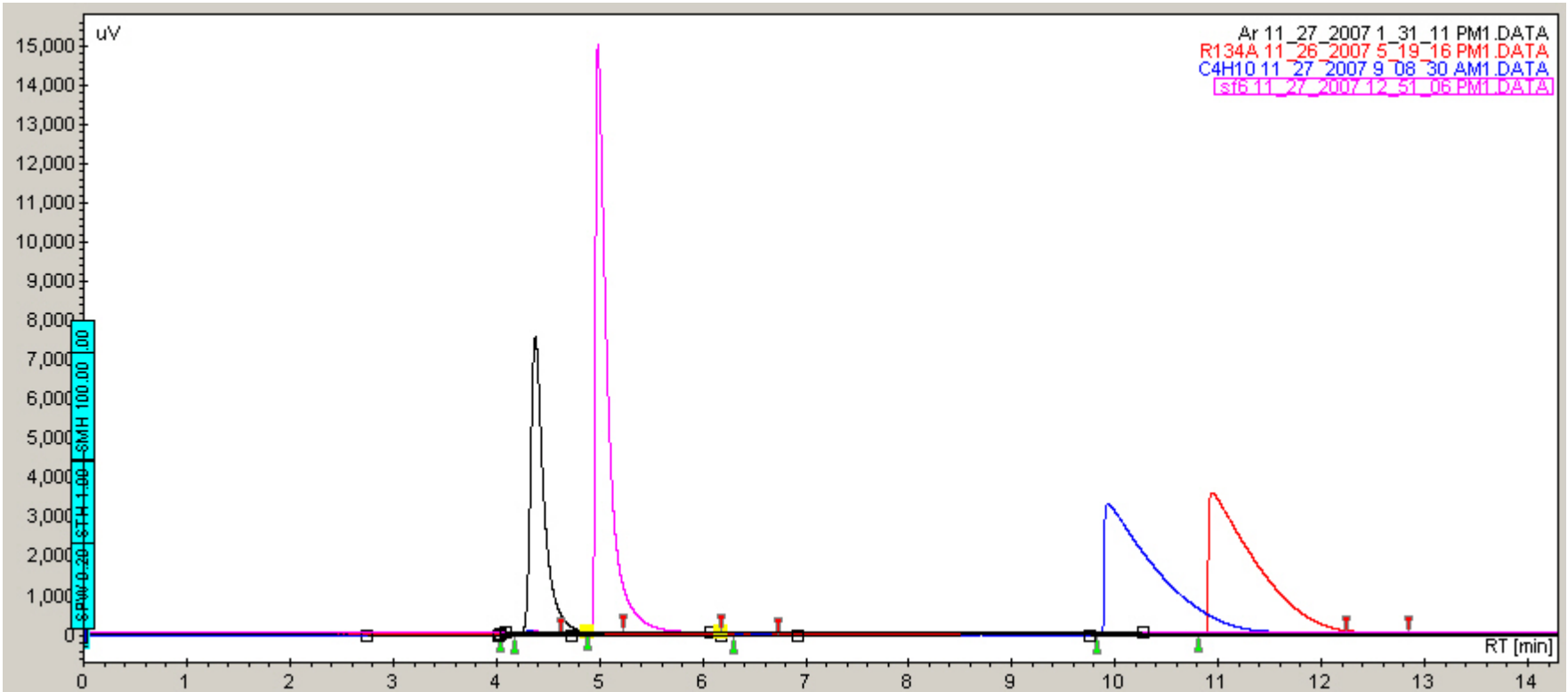
Acquired : 11/27/2007 12:52:27 PM
Processed : 11/27/2007 1:29:10 PM
Printed : 12/8/2007 11:25:54 AM



Peak results :

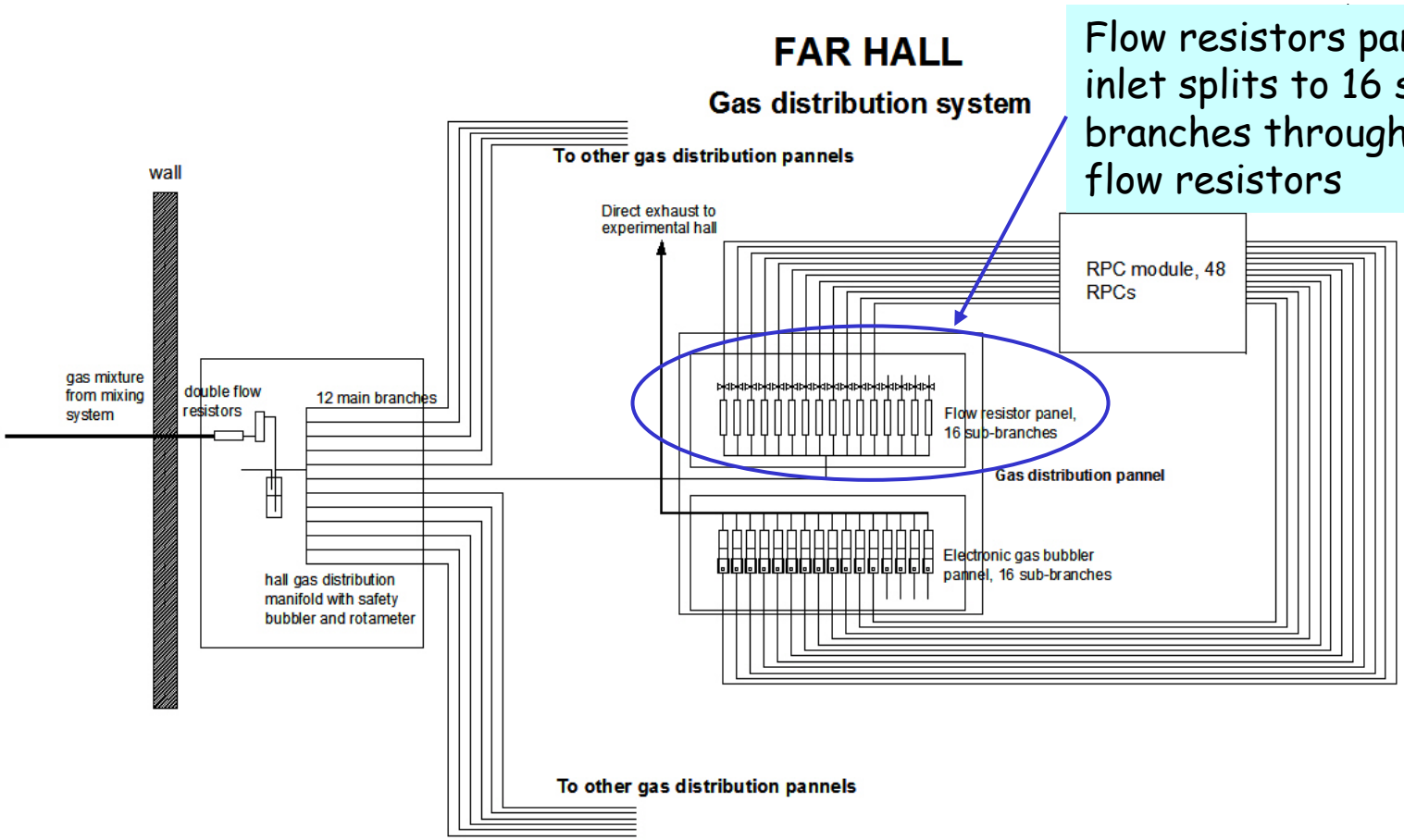
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.99	100.00	15005.1	2048.9	100.000
Total			100.00	15005.1	2048.9	100.000

Four calibration peaks



Apparently the areas under four peaks are different, use their ratio as the calibration constants.

Daya Bay RPC gas distribution panel



Flow resistors panel, one inlet splits to 16 sub-branches through capillary flow resistors

Gas flow resistor study

Does the capillary gas flow resistor affect the gas mixing ratio? We have studied the gas mixing ratio with GC for 1, 2 and 7 flow-resistor branches to see if the mixing ratio is stable.

File	Ar	R134A	C4H10	SF6	Sum	comment
opera 11_29_2007 1_00_39 PM2	872.1	331.4	78	7.8		1 branch
mixing ratio (opera)	77.70	17.82	4.07	0.42		
opera 11_29_2007 1_25_40 PM2	858.9	353.5	77.8	8.4		2 branches
mixing ratio (opera)	76.50	19.00	4.05	0.45		
opera 11_29_2007 2_27_52 PM2	848.1	353	75.8	9.1		7 branches
mixing ratio (opera)	76.26	19.16	4.10	0.49		

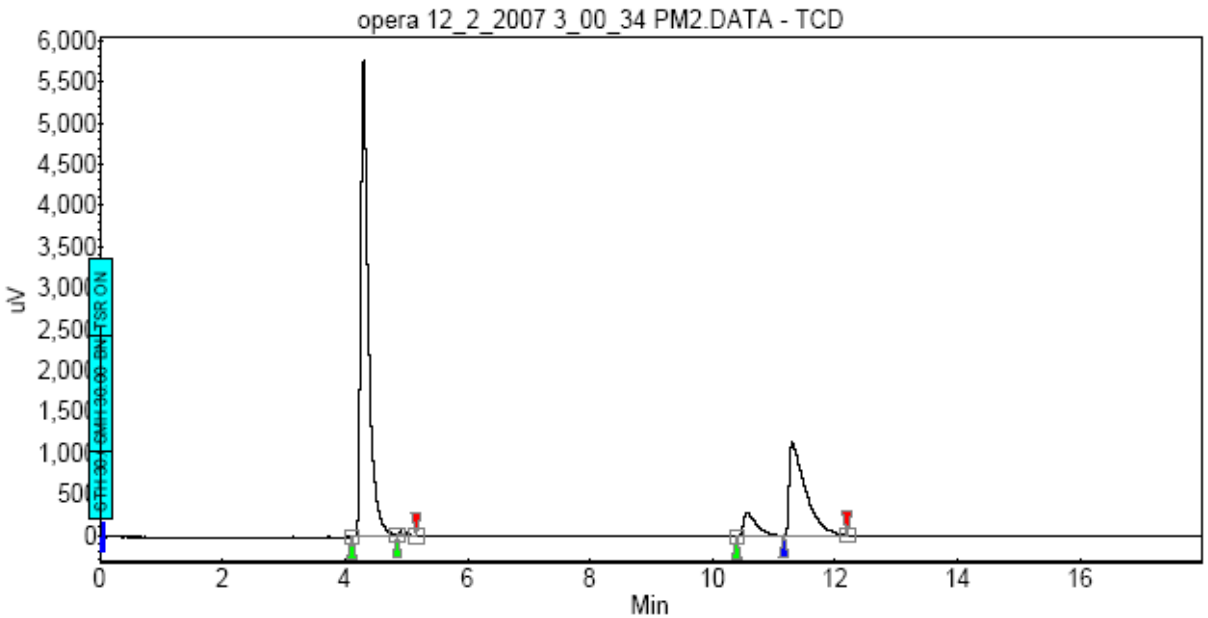
Slight difference between one branch and 2 or 7 branches, it could be due to the test error itself. More test needs to be done before drawing any conclusion.

Gas mix at the RPC gas outlet

Chromatogram : opera 12_2_2007 3_00_34 PM2_channel1

System : System_1
Method : analyze-11302007A
User : Administrator

Acquired : 12/2/2007 3:01:40 PM
Processed : 12/2/2007 3:33:54 PM
Printed : 12/8/2007 12:34:10 PM



Test the gas mixture at the gas outlet of two RPCs, HV (6000V) on.

Peak results :

Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.30	66.07	5763.7	837.0	66.070
2	UNKNOWN	4.94	0.63	77.9	8.0	0.635
3	UNKNOWN	10.57	5.76	289.5	73.0	5.762
4	UNKNOWN	11.30	27.53	1136.7	348.8	27.534
Total			100.00	7267.7	1266.9	100.000

Gas at the outlet of RPCs

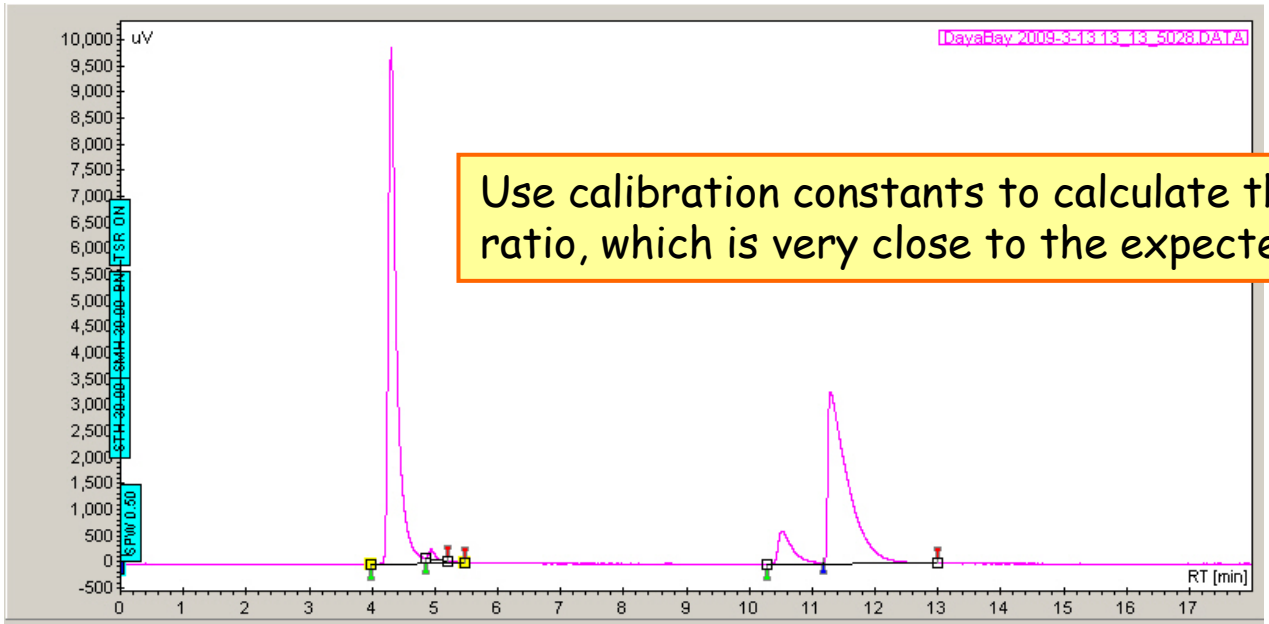
File	Ar	R134A	C4H10	SF6	Sum	comment
opera 12_2_2007 3_00_34 PM2	837	348.8	73	8		7 branches
	837	210.5105	42.71675	4.778885	1095.006	at outlet of 2
mixing ratio (opera)	76.44	19.22	3.90	0.44		
Calibration						
Ar 11_27_2007 1_44_17 PM1	1163.3					
R134A 11_26_2007 5_19_16 PM1		1927.5				
C4H10 11_27_2007 9_52_16 AM1			1988			
SF6 11_27_2007 12_51_06 PM1				1947.4		
	1	0.603528	0.585161	0.597361		

No extra peak is shown up, the mixing ratio is similar to original gas mixture.

HF production is not expected to be seen with such GC system, if we have GC-MS system, we might be able to see very low level of HF in the outlet gas mixture.

Daya Bay RPC gas mixture

Due to the gas safety consideration Daya Bay RPC has chosen a different gas mixing ratio as its baseline mixture: Ar/R134A/Isobutane/SF6(65.5/30/4/0.5), which is a non-flammable gas mix.



Use calibration constants to calculate the mixing ratio, which is very close to the expected value.

#	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.30	52.58	9914.9	1553.2	52.576
2	UNKNOWN	4.94	0.70	187.1	20.6	0.698
3	UNKNOWN	10.52	5.77	629.9	170.4	5.767
4	UNKNOWN	11.29	40.96	3289.8	1210.0	40.959
Total			100.00	14021.6	2954.1	100.000

Quote from Varian and Perkin Elmer

Varian's quote for 2 sets of 430-GC (newer version of 3900-GC) and one software license (can be used for two systems) is $23,389. Third 430-GC is adding additional $10,320. Total will be $33,709.

Perkin Elmer's quote for 3 sets of ARNEL-CLARUS 500 GC is $51,105.

The 3900-GC we purchased two years ago was $15,770. Varian is offering us deep discount indeed!

Conclusion

- The GC system mentioned here is a very good tool to check Daya Bay RPC gas system, it is a very reliable instrument we can count on;
- The gas mixing ratio before entering the gas flow resistor panel and after different number of flow resistors shows no unexpected difference;
- The gas mixture after two working 2x1m IHEP RPCs (OPERA gas mixture at 6000V) shows no unexpected extra peaks and different mixing ratio;
- Use the same GC to check mixing ratio for Daya Bay RPC gas mixture, the measured ratio is as expected;
- Under present financial situation Varian is offering deep discount for their new version 430-GC system, compare to Perkin Elmer's offer Varian is much more favorable.