Monitoring the RPC Gas Mixture with a Gas Chromatograph

C. Lu

Princeton University

Basic Working Principle of Gas Chromatography

A **gas chromatograph** is used for identifying constituents in a gas mixture.

It contains a narrow tube (*column*), through which different constituents (gases) of a sample gas mixture flow at different rates depending on their various chemical and physical properties.

The differential flow rate is increased by specific column filling, called the *stationary phase*.

The gas mixture to be tested is injected at time *t = 0* into the *carrier gas* stream, typically helium.

As the gases exit the end of the column, they are detected and identified by their exit time (*retention time*).

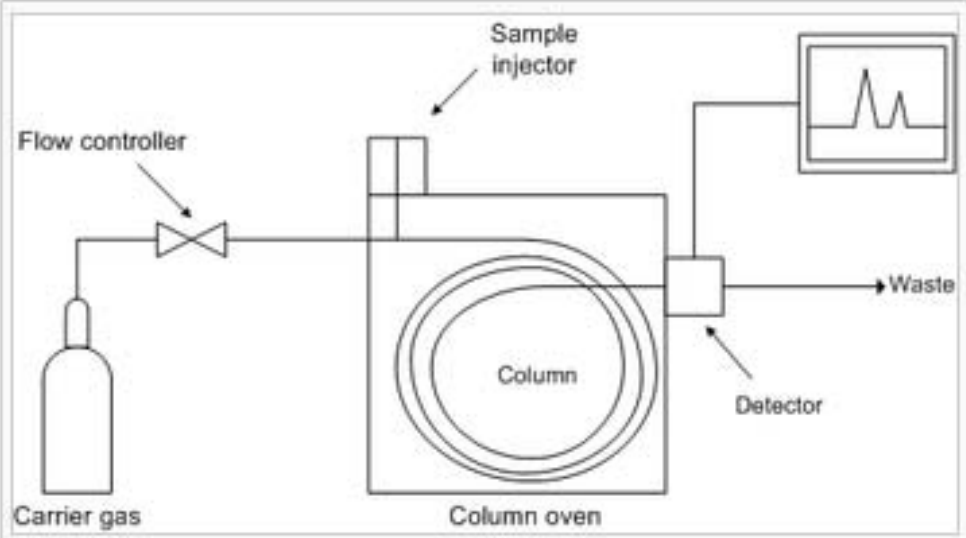


Diagram of a gas chromatograph.

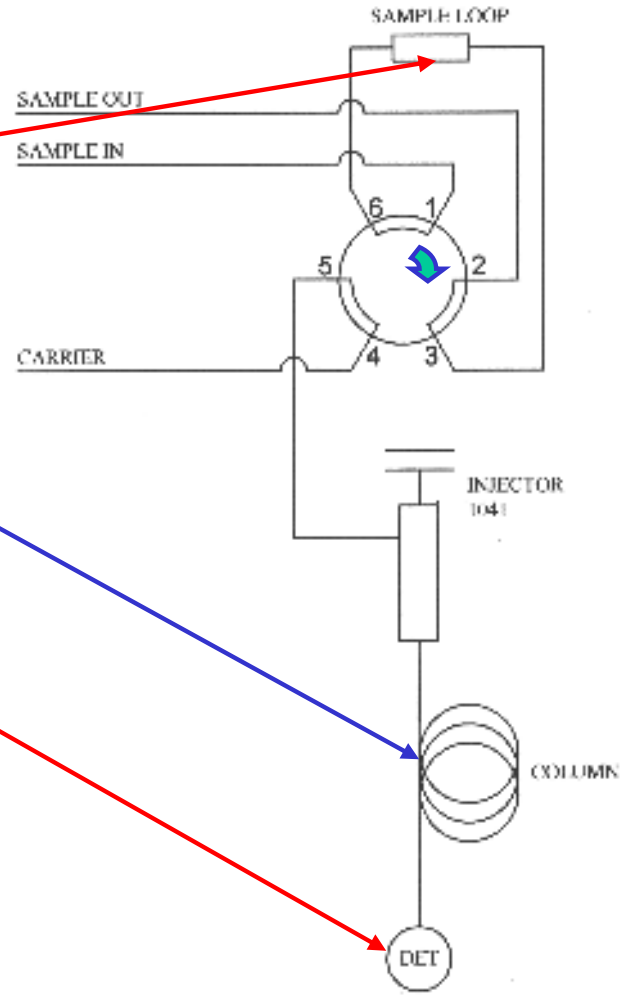
Varian 3900GC Gas Chromatograph

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.

The sampling loop on its sample valve has 5μL volume.

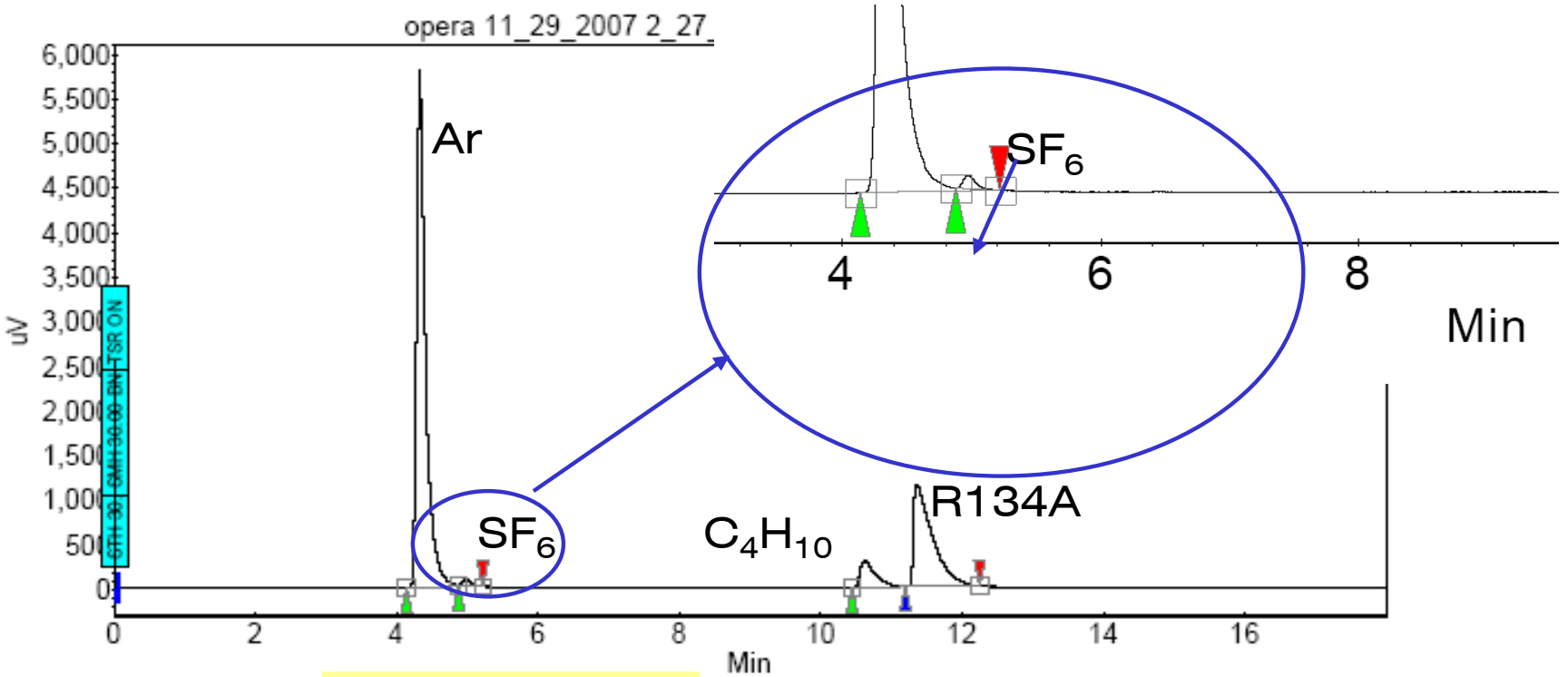
It uses a fused silica capillary column, 30m x 0.53mm ID, coating Silicaplot.

At the end of the column is a TCD (Thermal Conductivity Detector).



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

Analysis of the OPERA Gas Mixture at Princeton



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

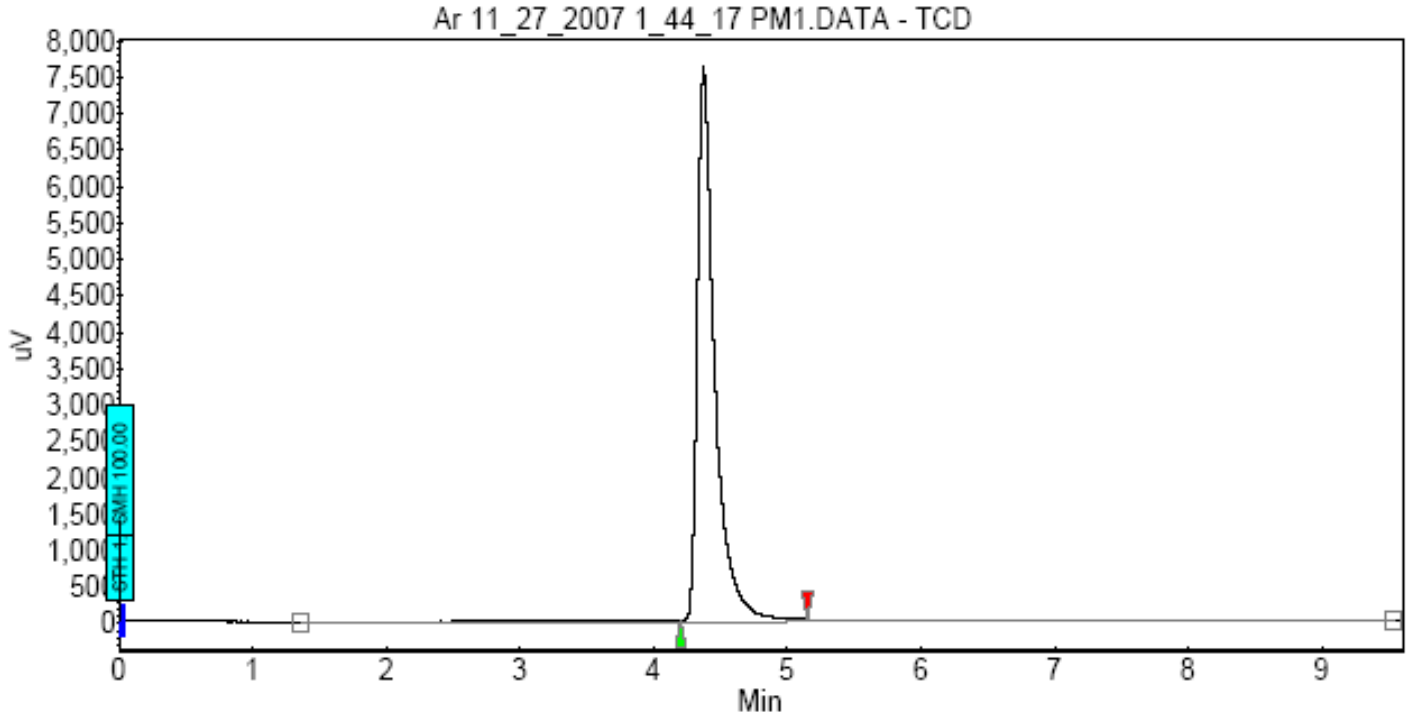
Four peaks can be clearly seen in the analysis of the OPERA gas mixture: Ar, SF6, C4H10 and R134A. The relation between the area under a peak and gas concentration is obtained by calibration with pure gases.

Argon 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 :

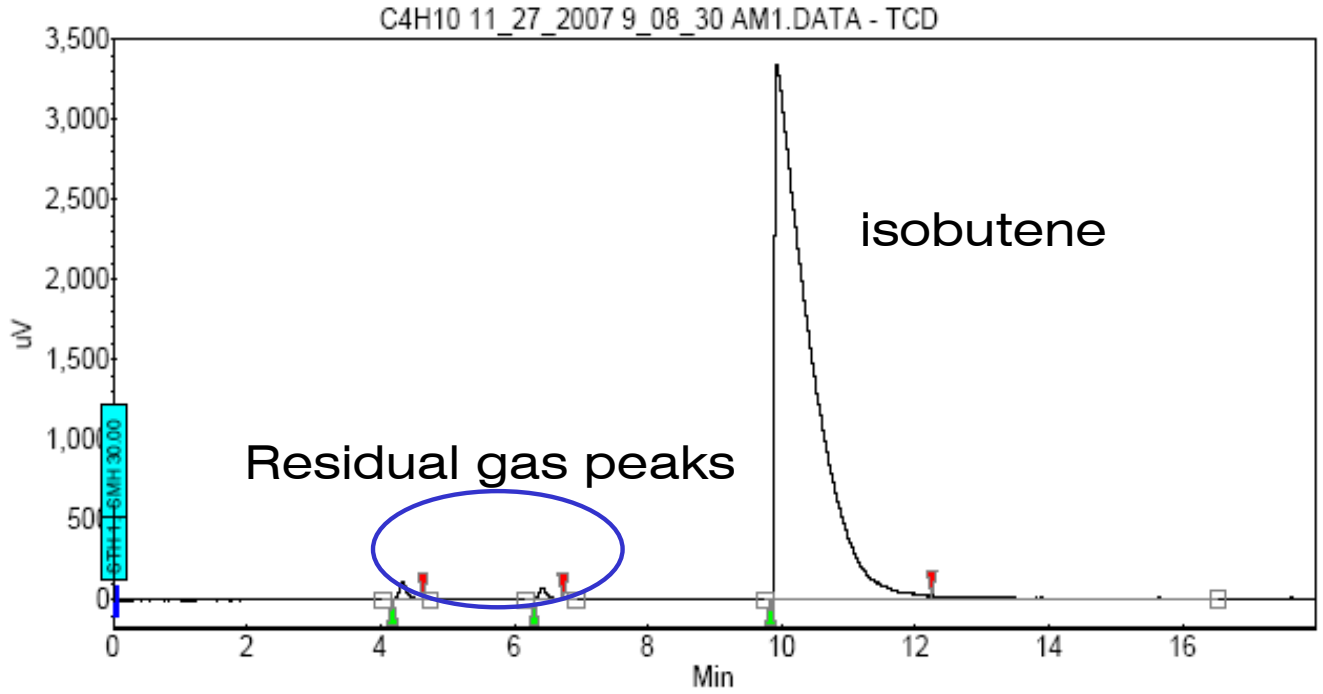
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.37	100.00	7809.8	1163.3	100.000
Total			100.00	7809.8	1163.3	100.000

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 :

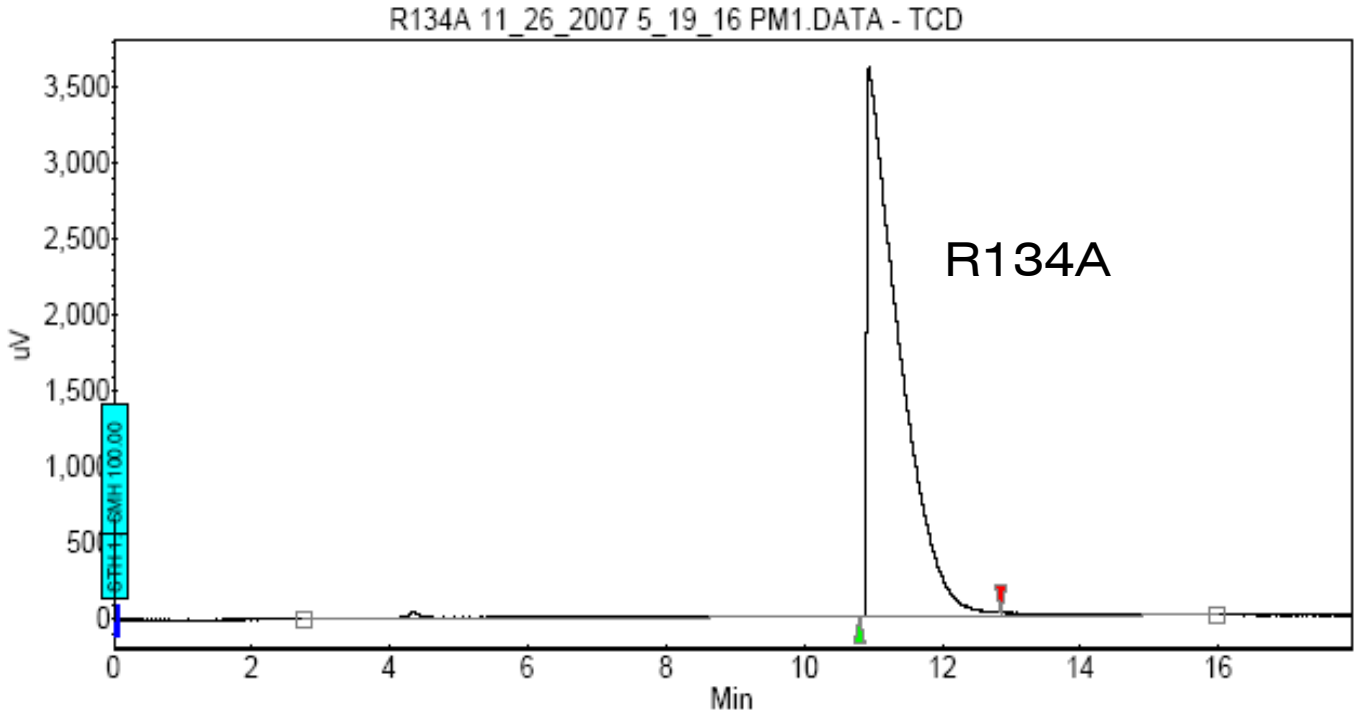
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.32	0.77	109.9	15.6	0.773
2	UNKNOWN	6.42	0.49	73.6	9.9	0.490
3	UNKNOWN	9.93	98.74	3337.7	1988.0	98.737
Total			100.00	3521.2	2013.4	100.000

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 :

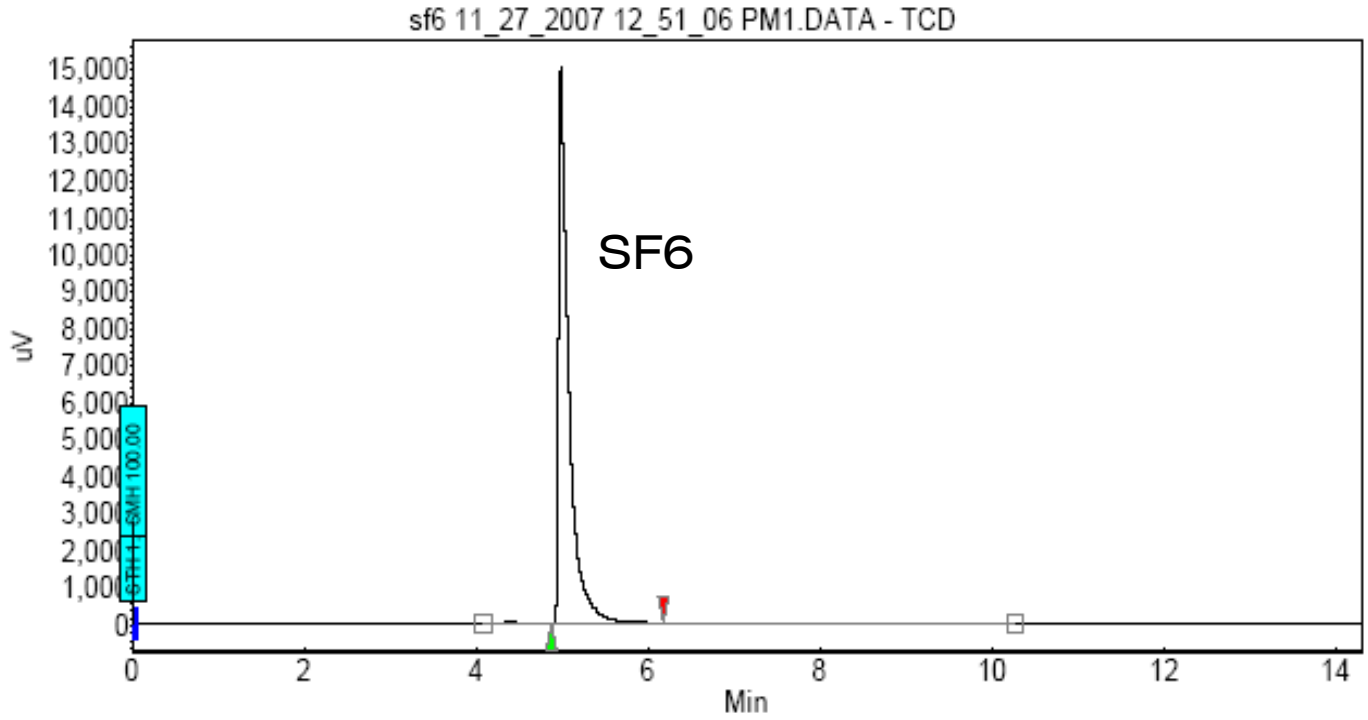
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	10.94	100.00	3809.6	1927.5	100.000
Total			100.00	3809.6	1927.5	100.000

SF6 Calibration Chromatogram

Chromatogram : sf6 11_27_2007 12_51_06 PM1_channel1

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

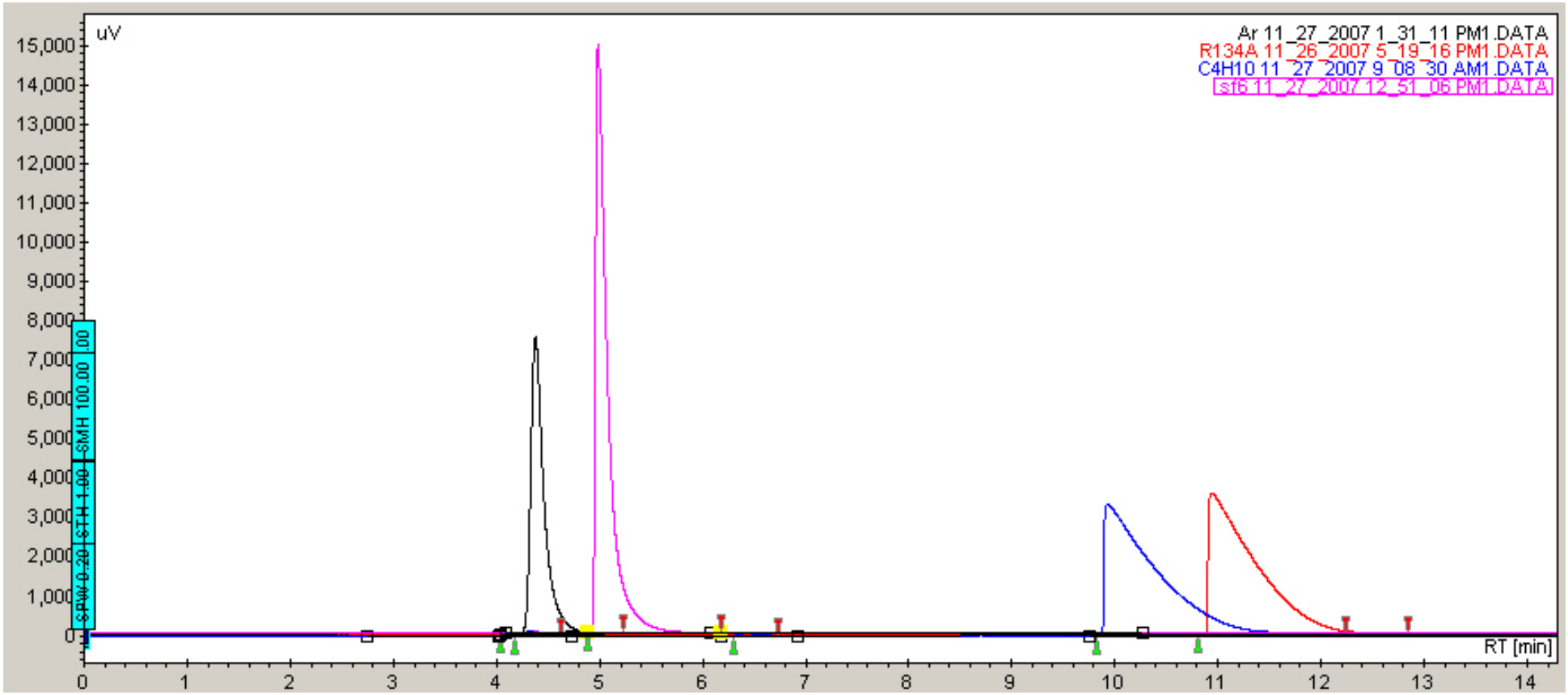
Acquired : 11/27/2007 12:52:27 PM
Processed : 11/27/2007 1:29:10 PM
Printed : 12/8/2007 11:25:54 AM



Peak results :

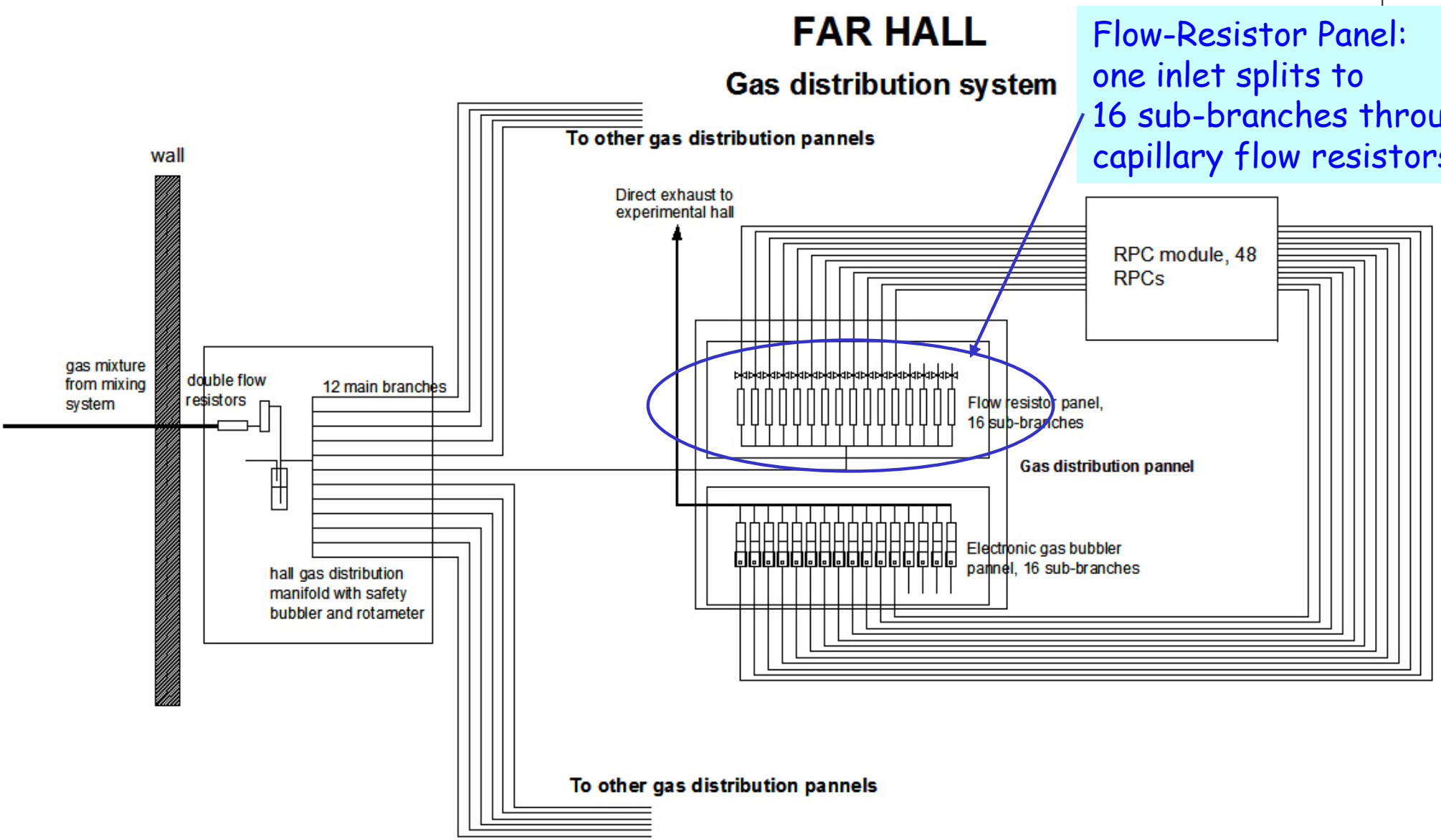
Index	Name	Time [Min]	Quantity [% Area]	Height [uV]	Area [uV.Min]	Area % [%]
1	UNKNOWN	4.99	100.00	15005.1	2048.9	100.000
Total			100.00	15005.1	2048.9	100.000

Four Calibration Peaks



The areas under four peaks are different, so use their ratios as the calibration constants.

Daya Bay RPC Gas Distribution Panel



Flow-Resistor 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 differences were observed between one branch and 2 or 7 branches.

This could be due to uncertainties in the test procedure.

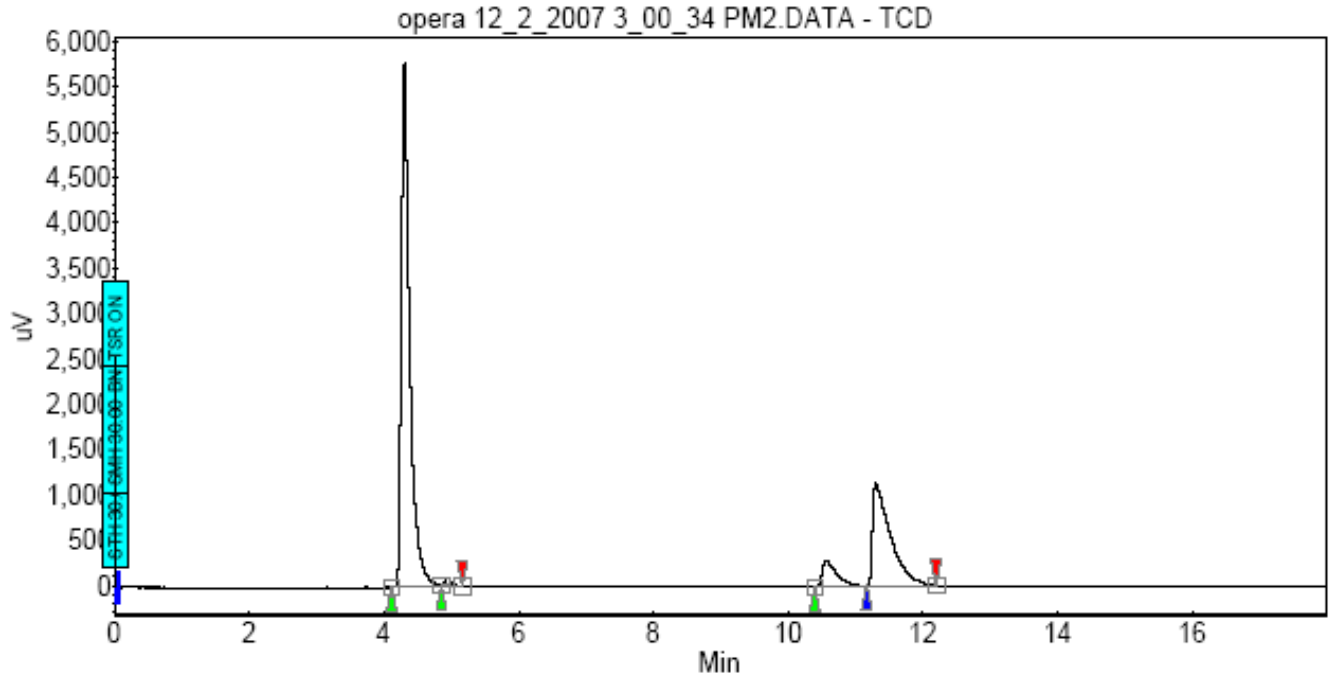
More test will be done before drawing any conclusion.

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



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, II

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 additional peaks are visible.

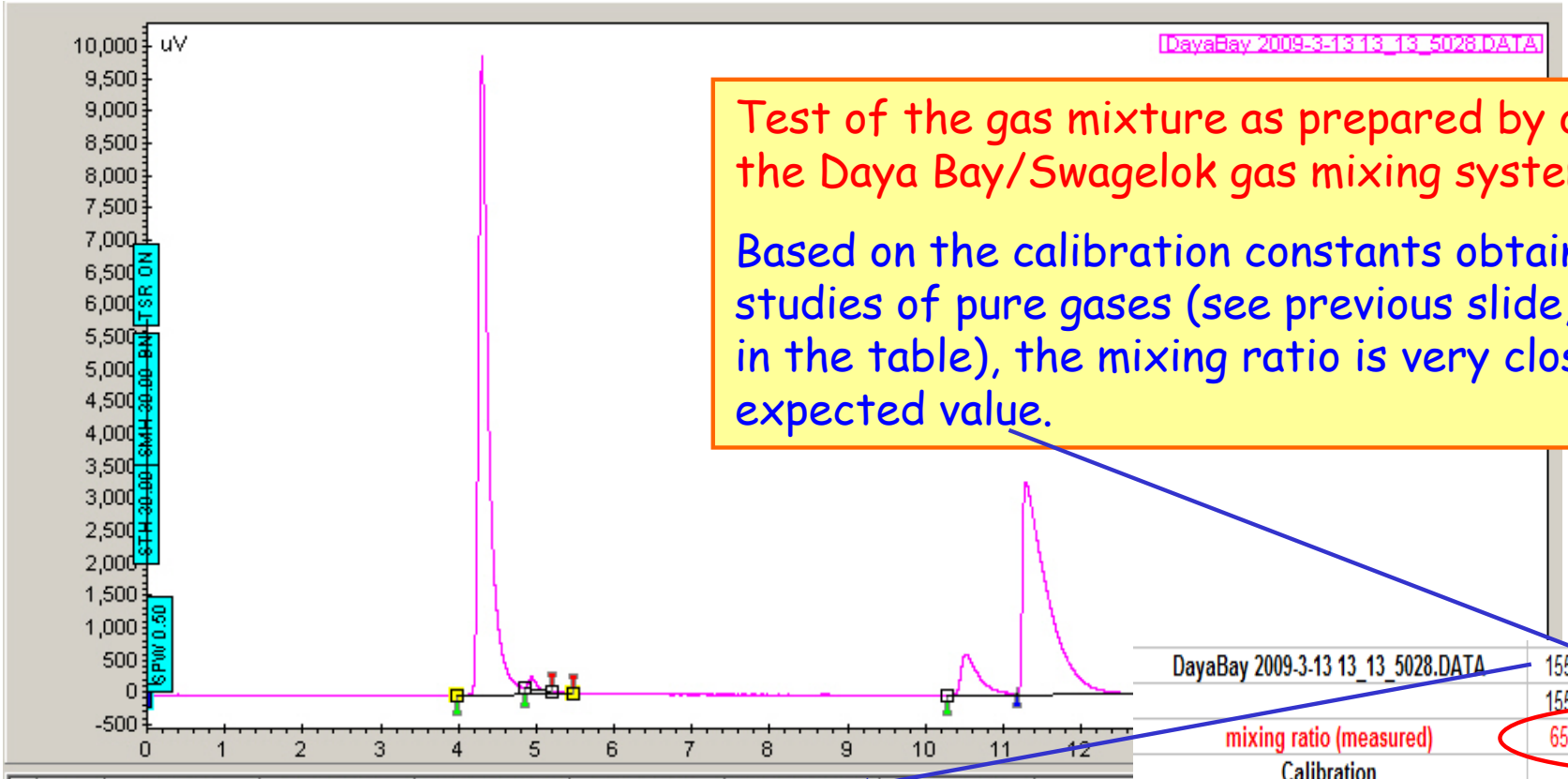
The mixing ratio is close to that of the original gas mixture.

HF production is not expected to be detectable with our GC system.

A GC-MS (Mass Spectrometer) system, might be able to see very low levels of HF in the outlet gas mixture.

Daya Bay RPC Gas Mixture

Due to gas safety considerations, Daya Bay RPC has chosen a different, non-flammable gas mixture has been chosen as the Daya Bay RPC baseline: Ar/R134A/Isobutane/SF6(65.5/30/4/0.5).



Test of the gas mixture as prepared by one of the Daya Bay/Swagelok gas mixing systems.

Based on the calibration constants obtained from studies of pure gases (see previous slide, last row in the table), the mixing ratio 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

DayaBay 2009-3-13 13_13_5028.DATA	1553.2	1210	170.4	20.6	
	1553.2	718.69	94.81	12.00	2378.71
mixing ratio (measured)	65.30	30.21	3.99	0.50	100.00
Calibration					
Ar 2_25_2008 2_12_24 PM20	1099.9				
R134A 2_25_2008 9_11_13 AM20		1851.8			
C4H10 2_26_2008 4_47_33 PM31			1976.8		
SF6 2_27_2008 10_12_19 AM31				1888	
	1	0.593963	0.556404	0.582574	

Gas calibration constants (see previous slide)

Quotes 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 2 or 3 systems) is $23,389.

A third 430-GC is adding additional $10,320.

Total cost for 3 systems 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 a deep discount!

Conclusion

- The Varian 3900GC system studied here is a reliable, high-quality tool to check Daya Bay RPC gas system, including the concentration of isobutane.
- Tests of gas mixing ratio before and after the gas flow-resistor panel show no unexpected differences.
- The gas mixture at the exit of two working 2 x 1 m^2 IHEP RPCs (OPERA gas mixture at 6000V) shows no unexpected extra peaks or difference between the input and output mixing ratio.
- The GC also verified that Daya Bay RPC gas as mixed by one of the Daya Bay/Swagelok mixing panels has the desired mixing ratio to better than 0.5% in all 4 component gases.
- Varian is offering a deep discount for 2 or 3 of their new version 430-GC systems.