Development of a System to Measure Trace Amounts of Helium in Air, Nitrogen, Hydrogen, or Deuterium

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What is the Problem?

- Need to be able to separate and then quantify ppb amounts of He from essentially pure D_2.

Why is this Difficult?

- Most mass spectrometers do not have sufficient mass resolution to separate He^4 from D_2. ($\Delta M=0.025$ AMU)
- Most measuring instruments do not have sufficient dynamic range (need > 10^9)
- Ambient air is 5 ppm He^4.

Masses of light ions

	atomic mass	MKS mass #	positive ion mass	MKS mass #
atomic hydrogen	1.007825	50.39	1.00727	50.36
molecular hydrogen	2.01565	100.78	2.01510	100.76
atomic deuterium	2.014102	100.71	2.01355	100.68
molecular deuterium	4.028204	201.41	4.02765	201.38
tritium	3.016049	150.80	3.01550	150.78
He-3	3.016029	150.80	3.01548	150.77
HD	3.02193	151.096	3.02083	151.04
He-4	4.002603	200.13	4.00205	200.10

Mass of electron = 0.00054858 amu = $9.10940*10^{-31}$ kg

Mass of neutron = 1.008665 amu = $1.67495*10^{-27}$ kg

Mass of proton = 1.007277 amu = $1.6726*10^{-27}$ kg

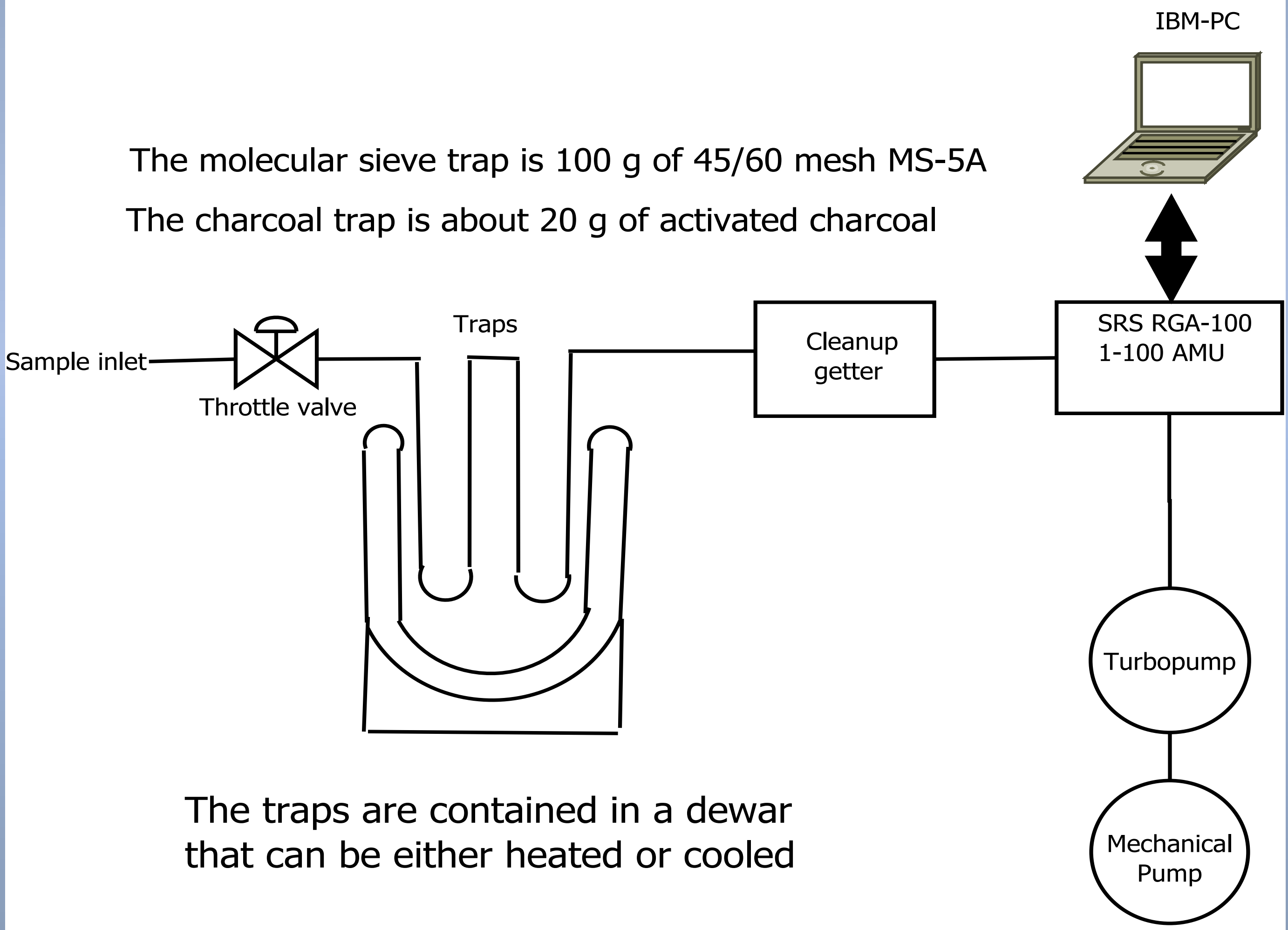
Atomic mass unit= 1.0000 amu

Concept of system for analysis of helium in air samples

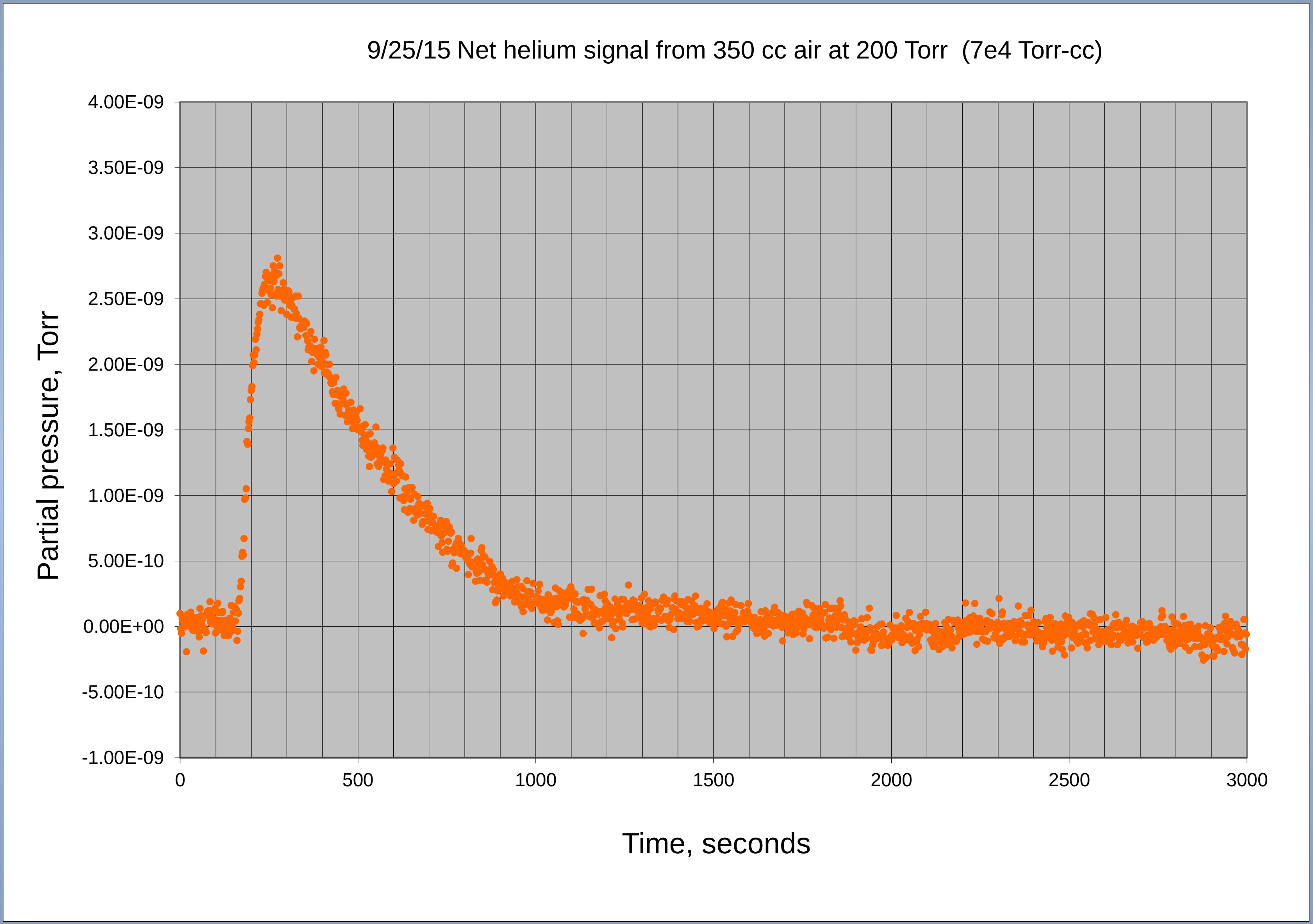
There are two traps proposed, one is activated charcoal and the other is MS-5A

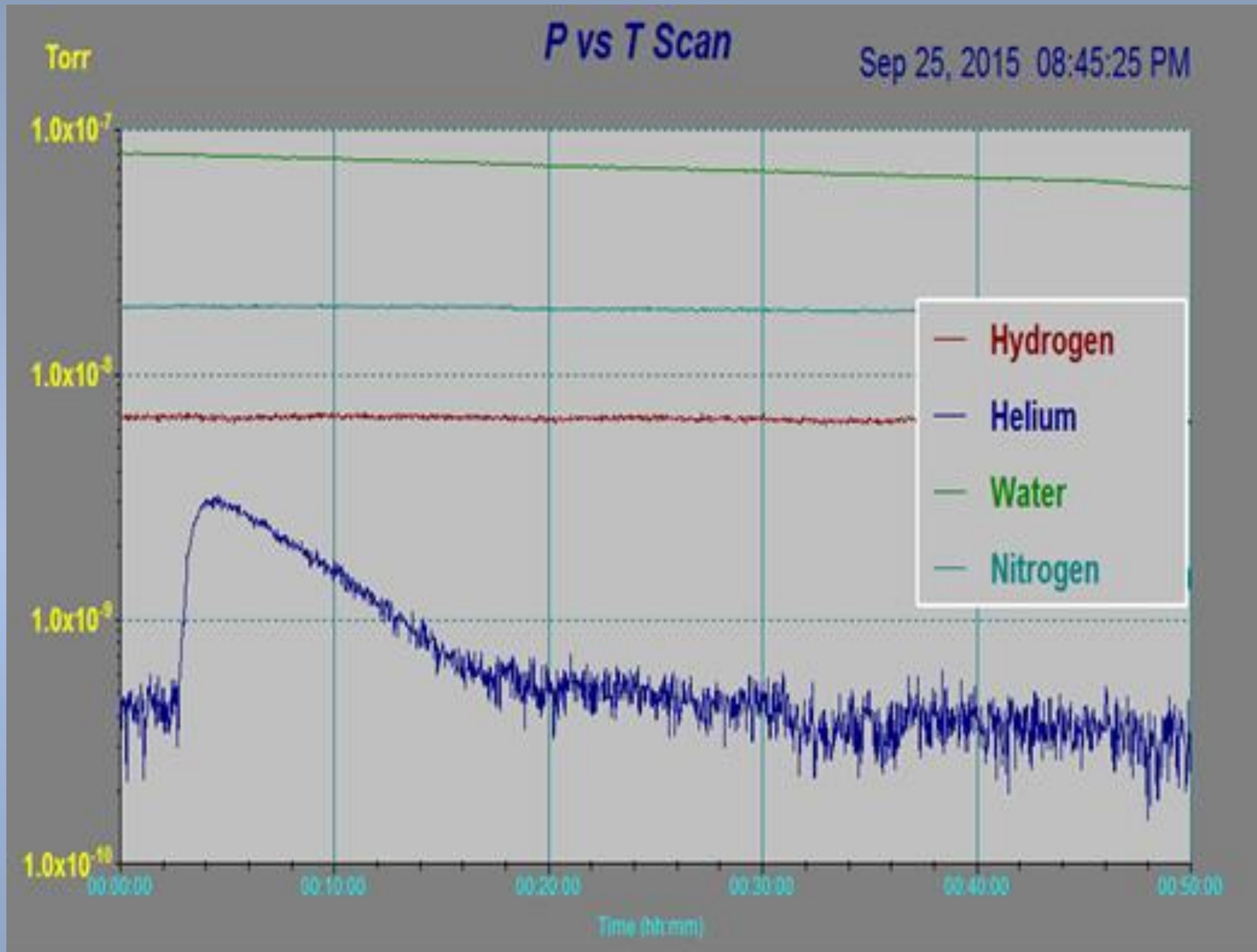
The molecular sieve trap is 100 g of 45/60 mesh MS-5A

The charcoal trap is about 20 g of activated charcoal



The traps are contained in a dewar that can be either heated or cooled


9/25/15 Net helium signal from 350 cc air at 200 Torr (7e4 Torr-cc)
Partial pressure, Torr
4.00E-09
3.50E-09
3.00E-09
2.50E-09
2.00E-09
1.50E-09
1.00E-09
5.00E-10
0.00E+00
-5.00E-10
-1.00E-09
0
500
1000
1500
2000
2500
3000
Time, seconds


P vs T Scan
Torr
Sep 25, 2015 08:45:25 PM
1.0x10-7
1.0x10-8
1.0x10-9
1.0x10-10
Hydrogen
Helium
Water
Nitrogen
00:00:00
00:10:00
00:20:00
00:30:00
00:40:00
00:50:00
Time (hh:mm)

Next, we made several modifications to improve the overall efficiency

- Added a magnetic sector helium leak detector.
  - This instrument has quite a bit more sensitivity for mass-4 helium relative to that for the RGA.
  - The effective background and variability is also much better than that for the RGA.

- Added a TDS-744A digital oscilloscope to log data from the leak detector.
  - This device has only 8 bit resolution, but can be operated in "high resolution" mode that averages the signal between samples yielding 11-12 bit effective resolution.

- Added several pressure gauges and vacuum pumps to facilitate the sample gas handling

- The SRS-100 RGA was retained to verify that there were no gasses other than mass=4 passing through the traps.

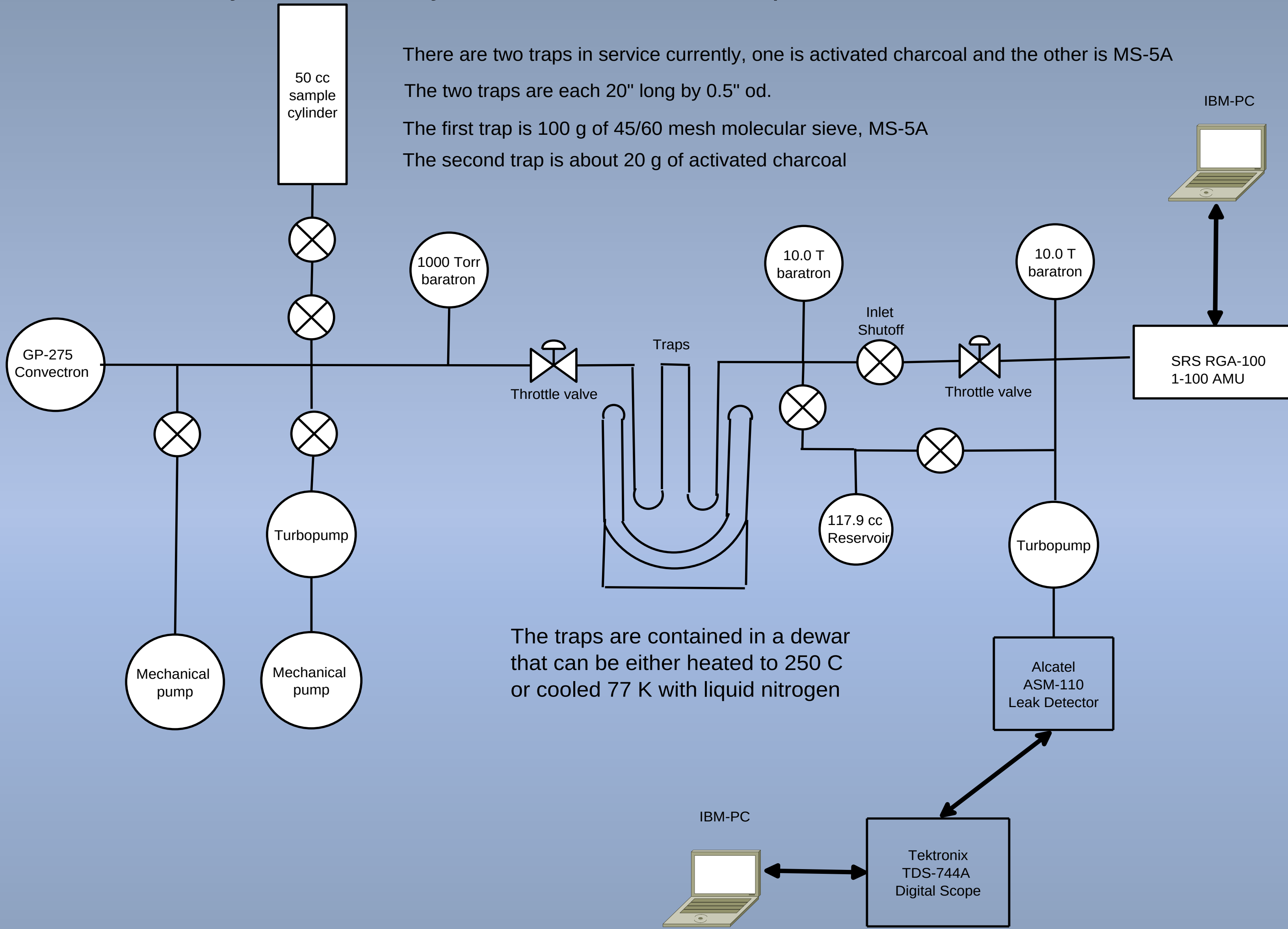
Gas system for analysis of helium in air samples as of 10/15/2015

There are two traps in service currently, one is activated charcoal and the other is MS-5A

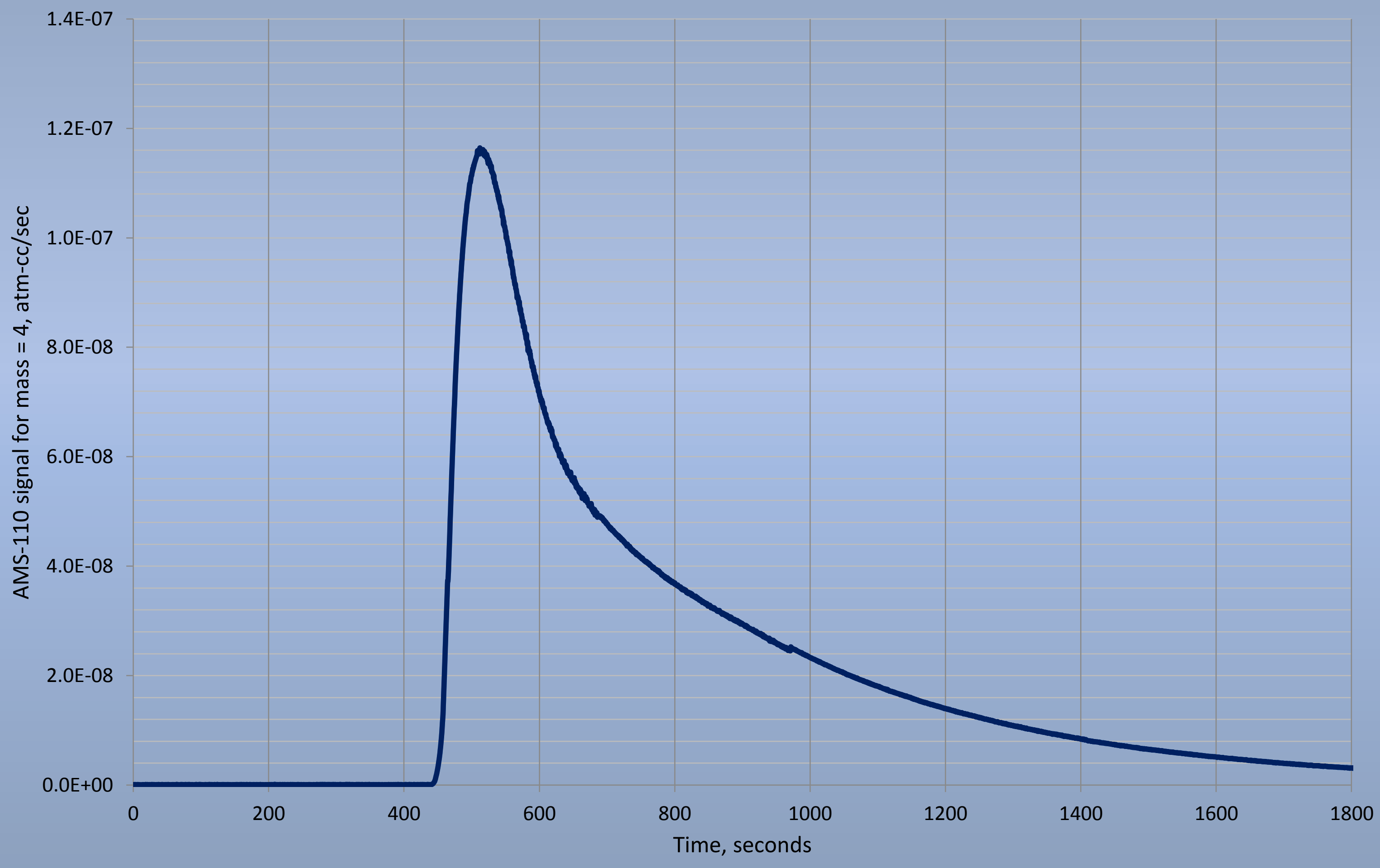
The two traps are each 20" long by 0.5" od.

The first trap is 100 g of 45/60 mesh molecular sieve, MS-5A

The second trap is about 20 g of activated charcoal



ASM-110 signal for mass=4 from 10 cc of air at 2585 Torr (2.6e4 Torr-cc) 9-30-15
This sample was about1/3 the size of that shown on the previous plot.

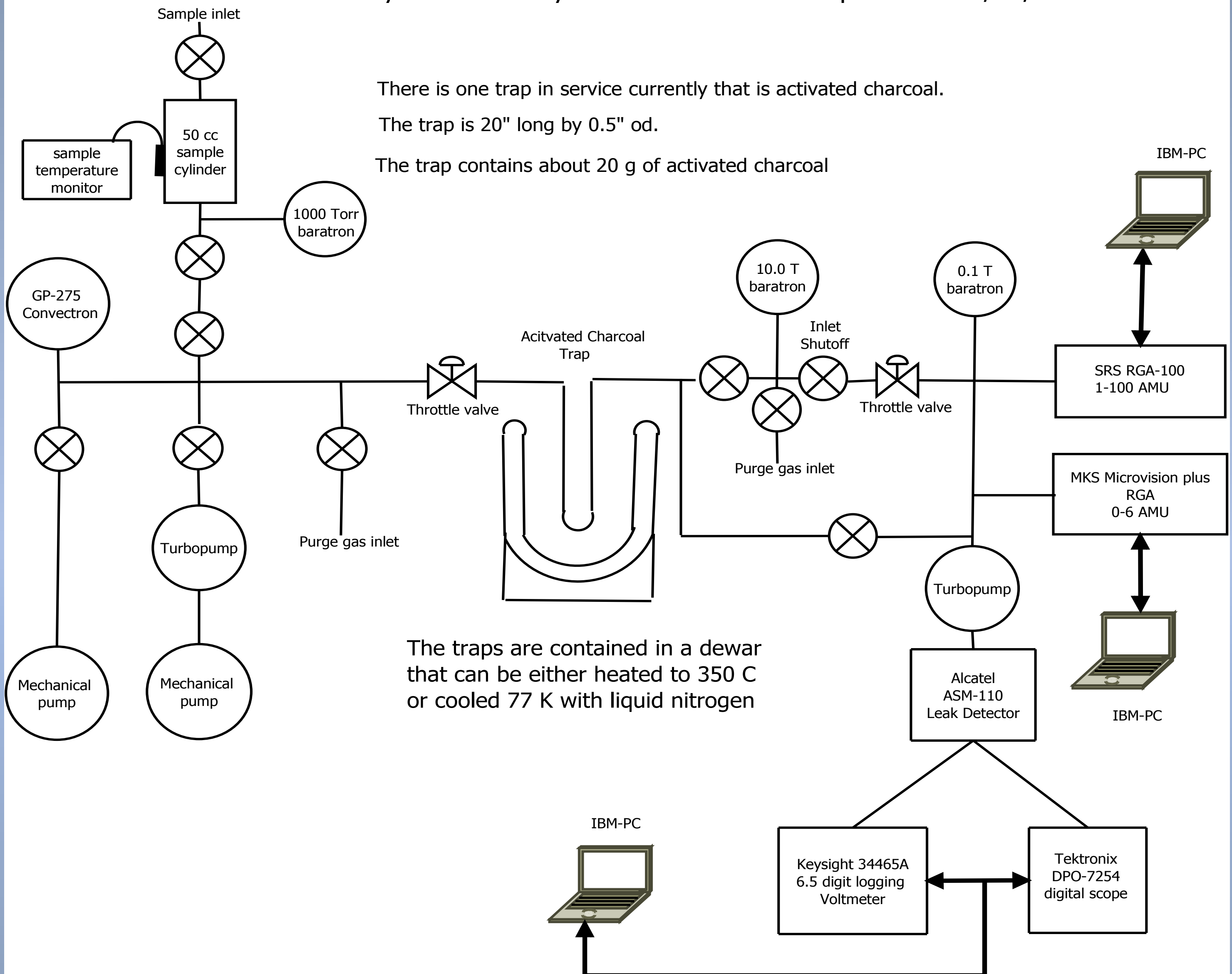


Gas system for analysis of helium in air samples as of 3/17/2017

There is one trap in service currently that is activated charcoal.

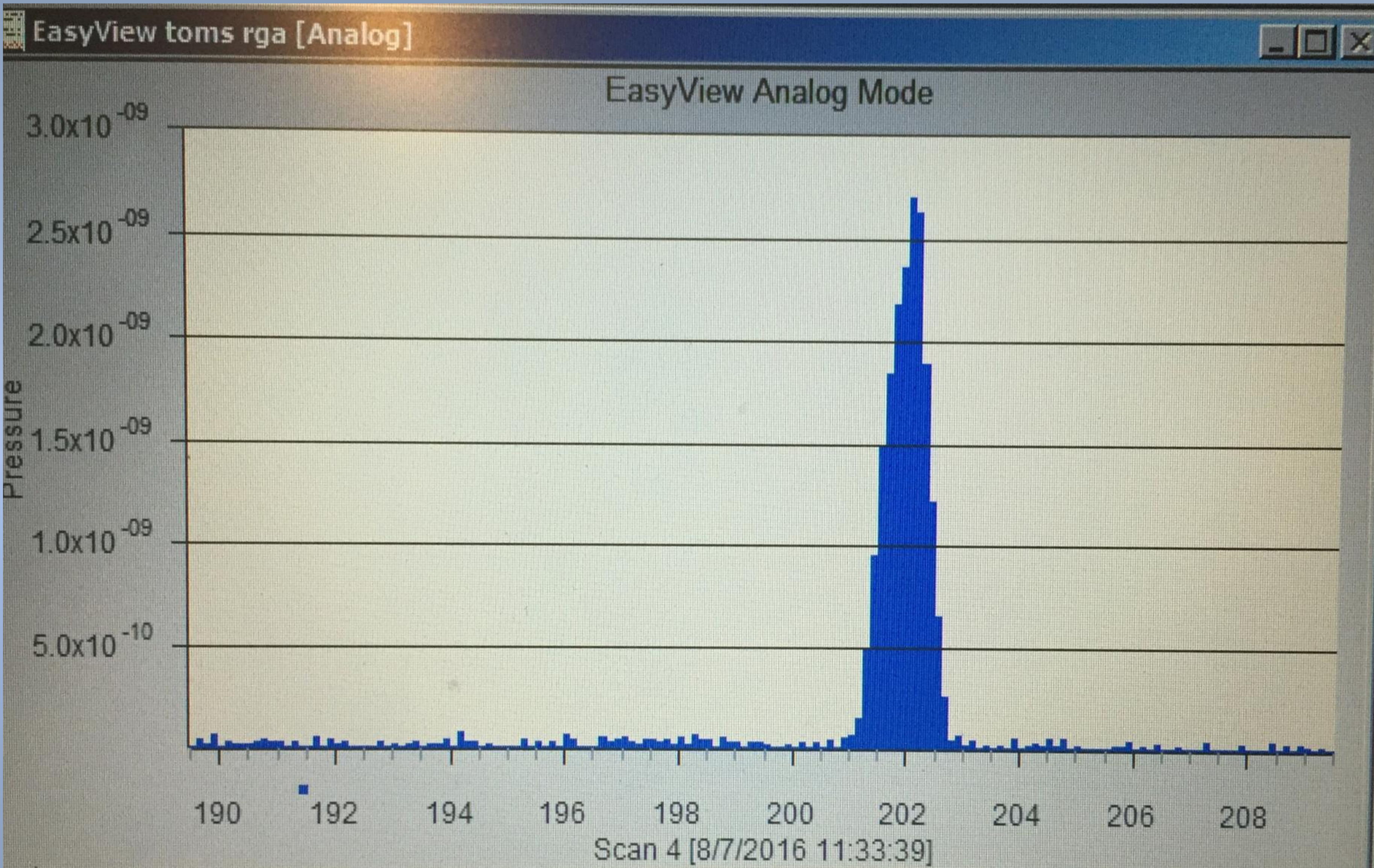
The trap is 20" long by 0.5" od.

The trap contains about 20 g of activated charcoal

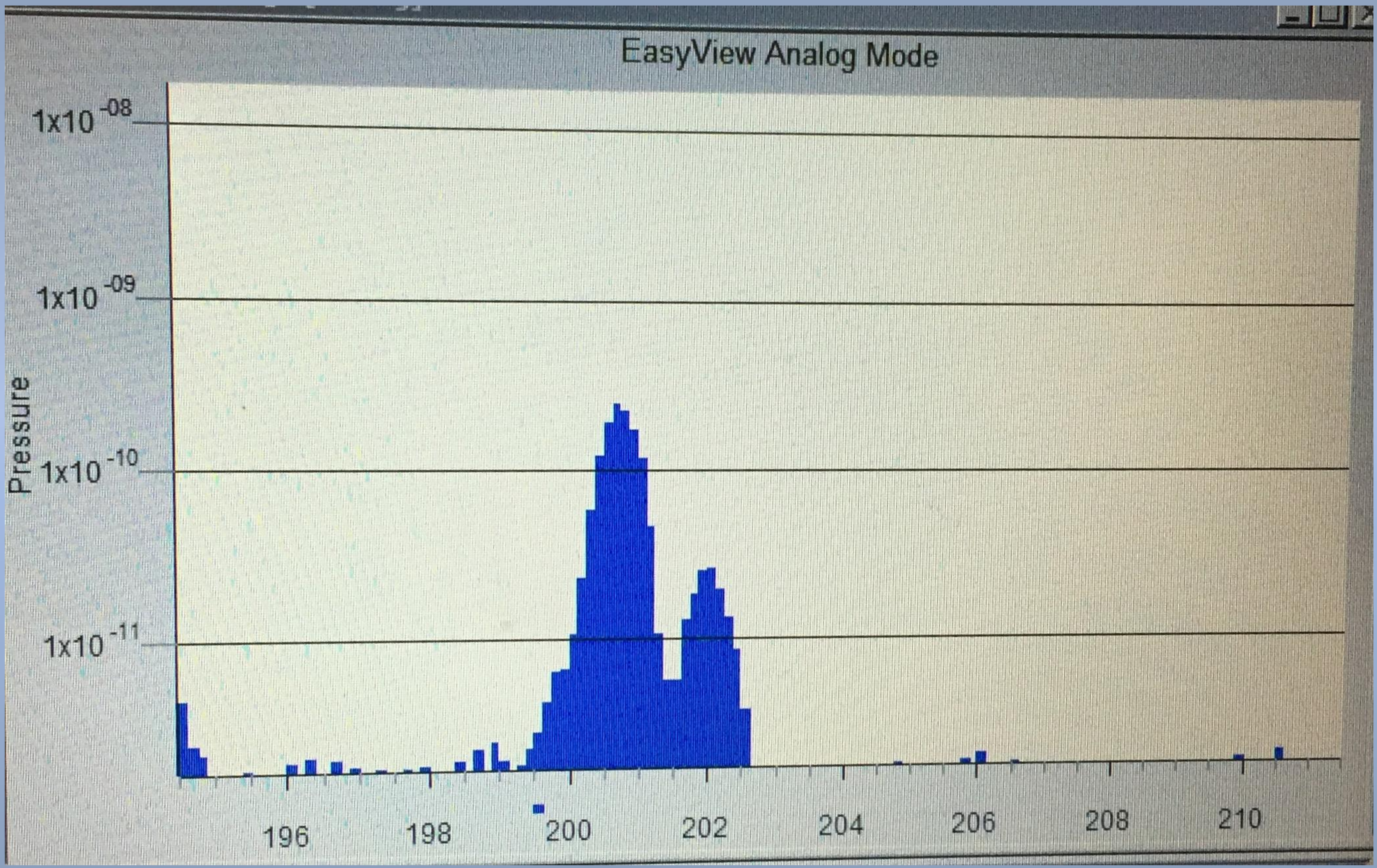


The traps are contained in a dewar that can be either heated to 350 C or cooled 77 K with liquid nitrogen

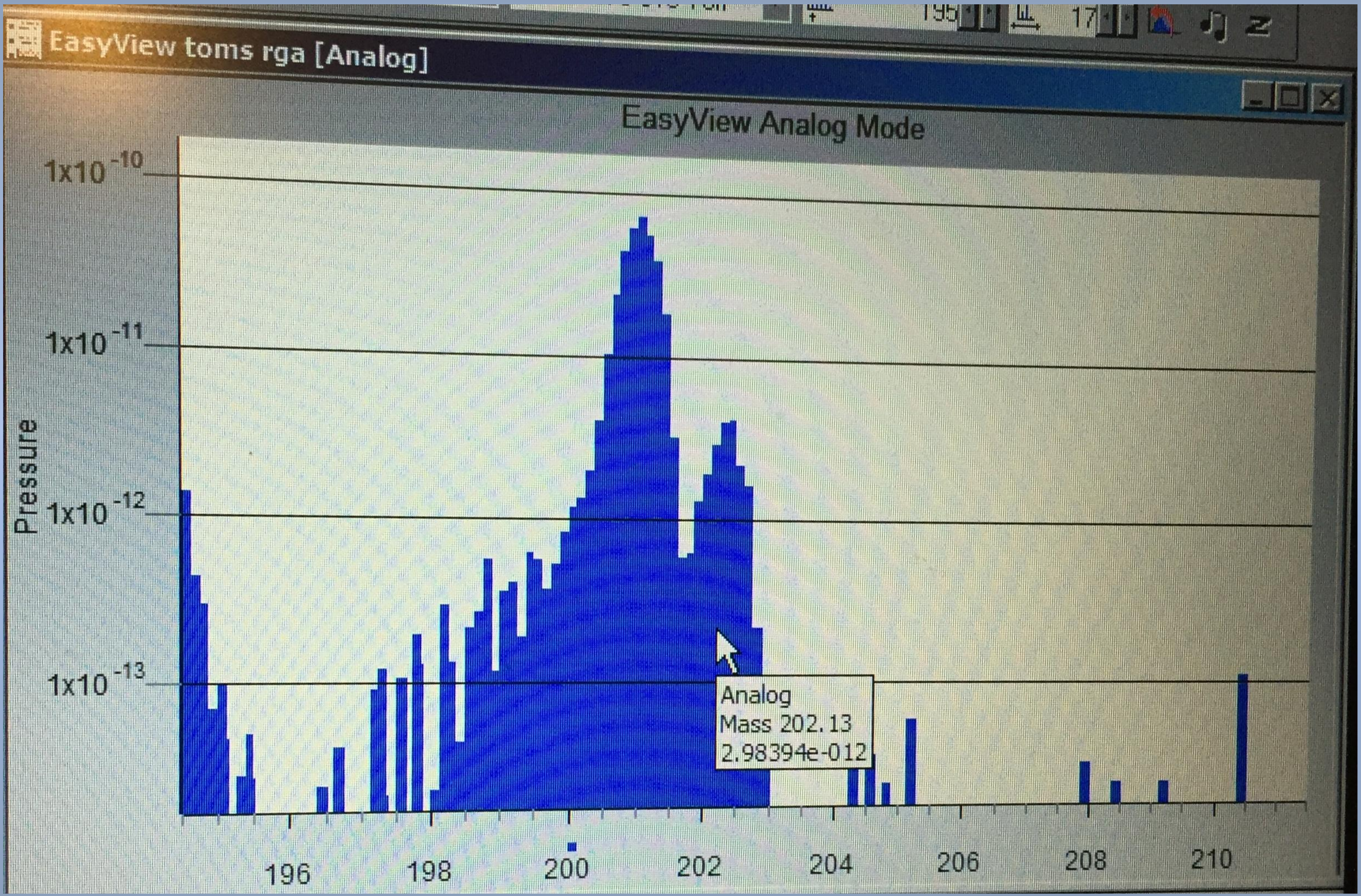
Deuterium Response using the MKS Microvision RGA



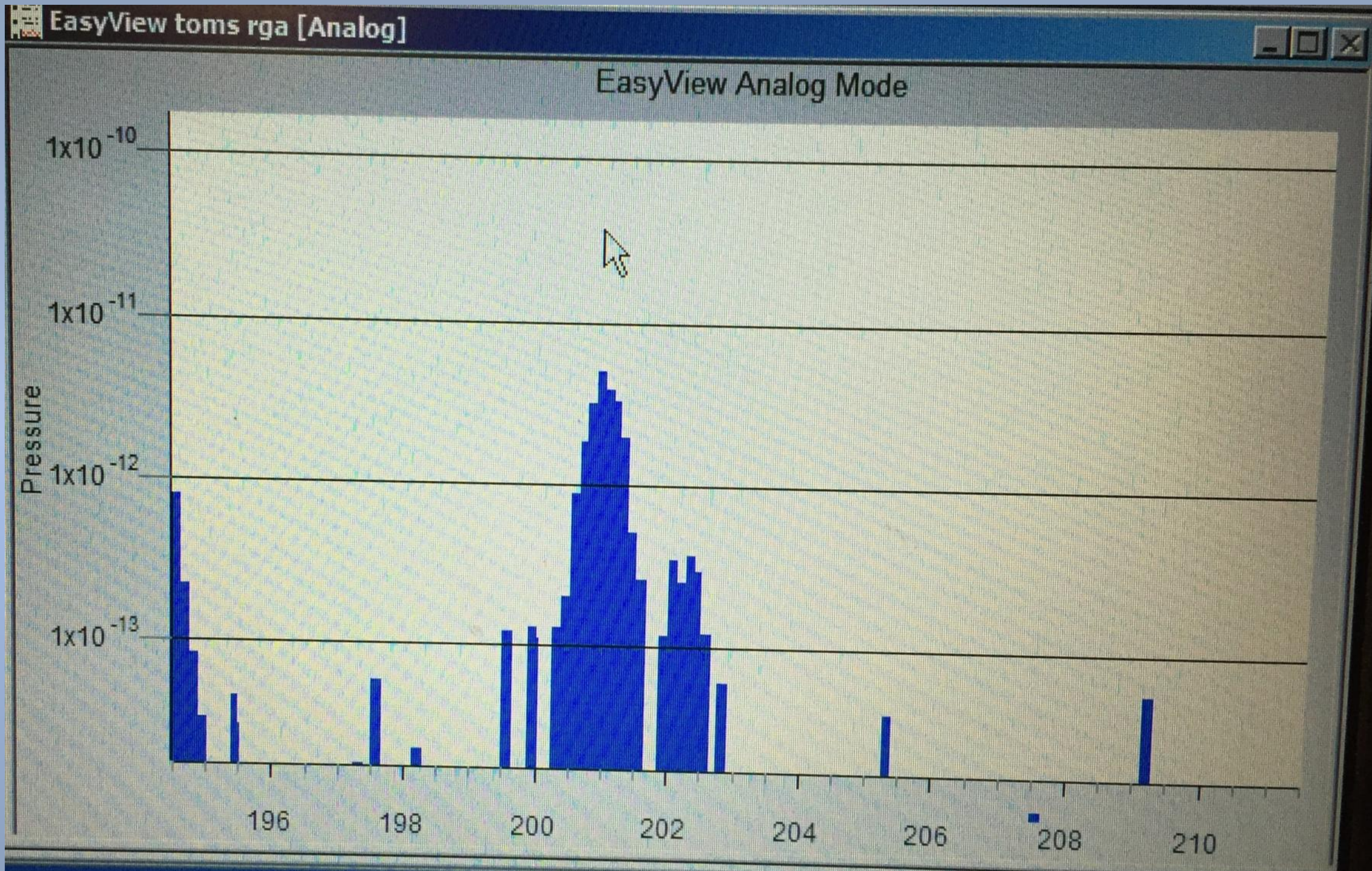
Mixture of He^4 and D_2



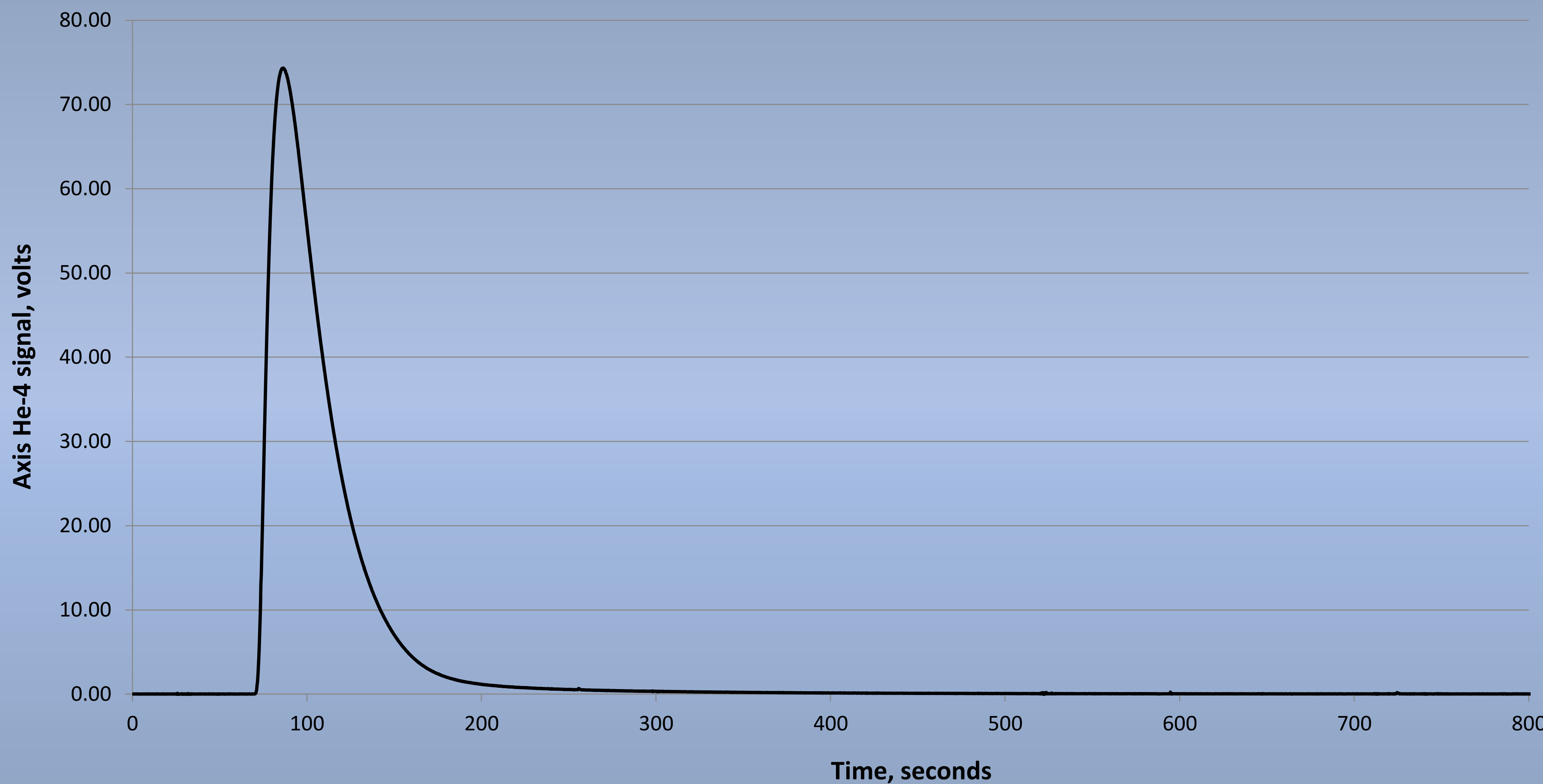
Same mixture at lower pressure showing low mass tailing



Again at even lower pressure



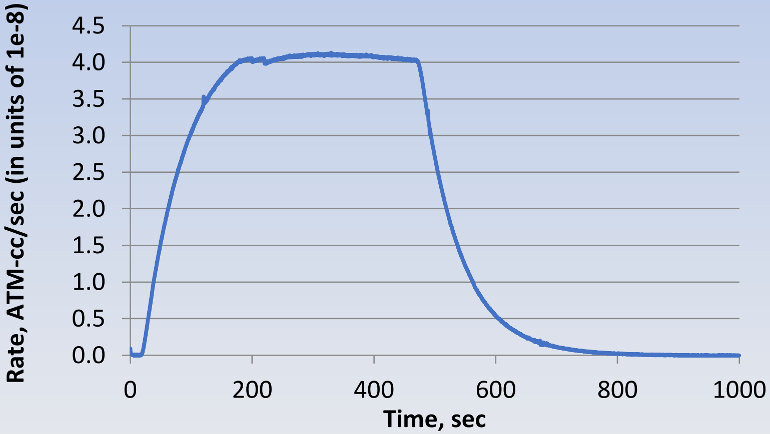
Run 127 50 cc @ 25 Torr 12.8 PPM He-4 (1250 Torr-cc)

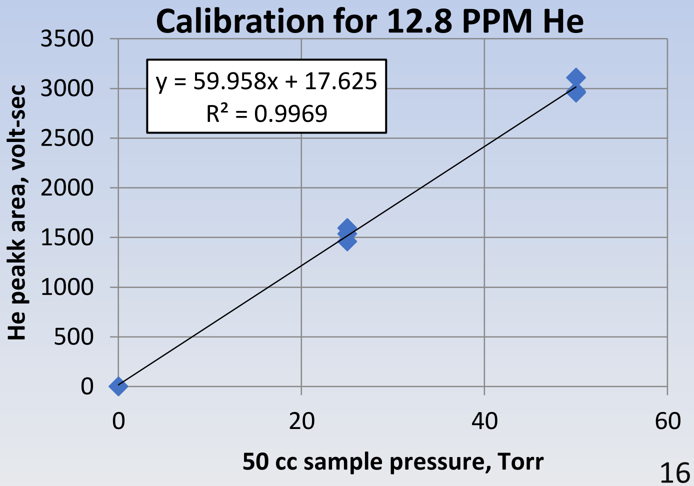


Calibration

We have two methods to calibrate the system:

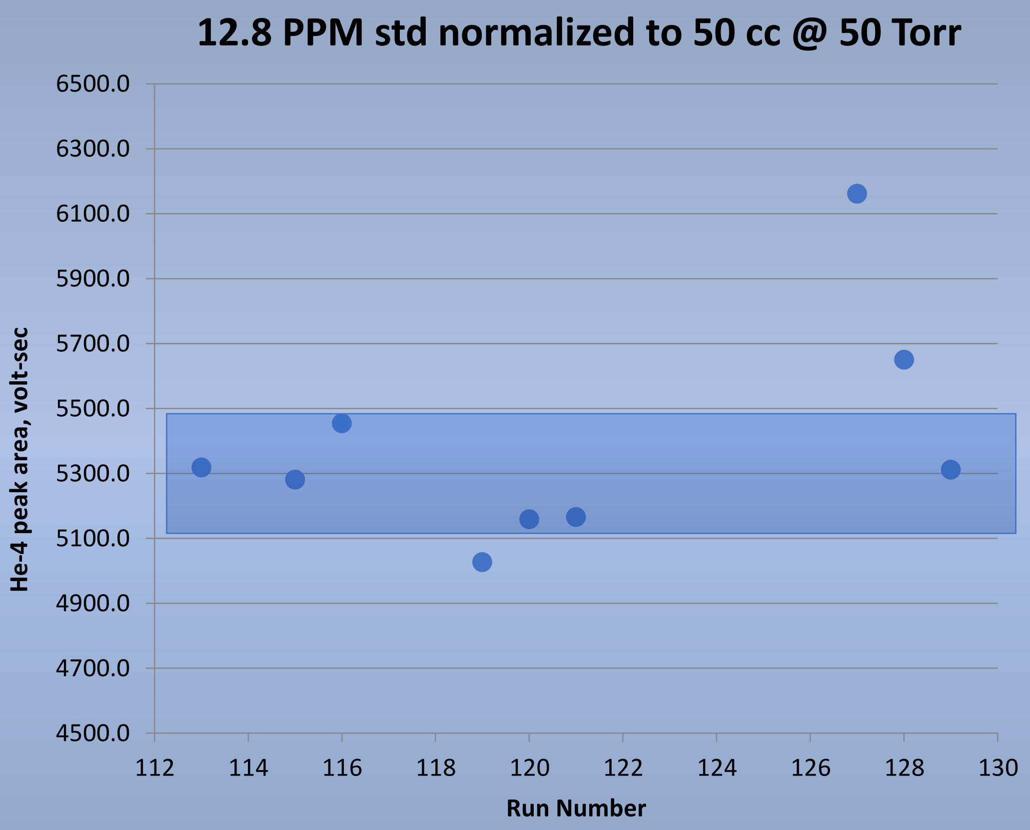
1. We can verify that the calibration of the helium leak detector is correct using a known calibrated leak. The integral, over time, of the measured sample "leak rate" gives the number of atm-cc of He in the sample. The ratio of this to the total number of atm-cc in the sample is the concentration of helium in the sample.
2. We can prepare known standards of helium that are similar in concentration to that expected in the samples and then analyze the sample and standard using the same volumes and pressures for both. The concentration of helium in the sample is given by the ratio of the integrated signal over time for the standard to that for the sample.





Date	pressure	area	norm area
3/25/17	25.00	2659.5	5319.0
3/29/17	25.00	2640.5	5281.0
3/31/17	25.01	2526.2	5454.4
4/2/17	24.96	2509.4	5026.8
4/7/17	25.01	2580.6	5159.2
4/7/17	25.03	2586.1	5166.0
4/29/17	25.00	3081.0	6162.0
4/30/17	25.01	2826.0	5650.5
4/30/17	25.00	2655.8	5311.6

avg	5296.1
stdev	192.7
rsd	3.6%
+1 stdev	5488.7
-1 stdev	5103.4



Gas system for analysis of helium in air samples as of 5/9/2017

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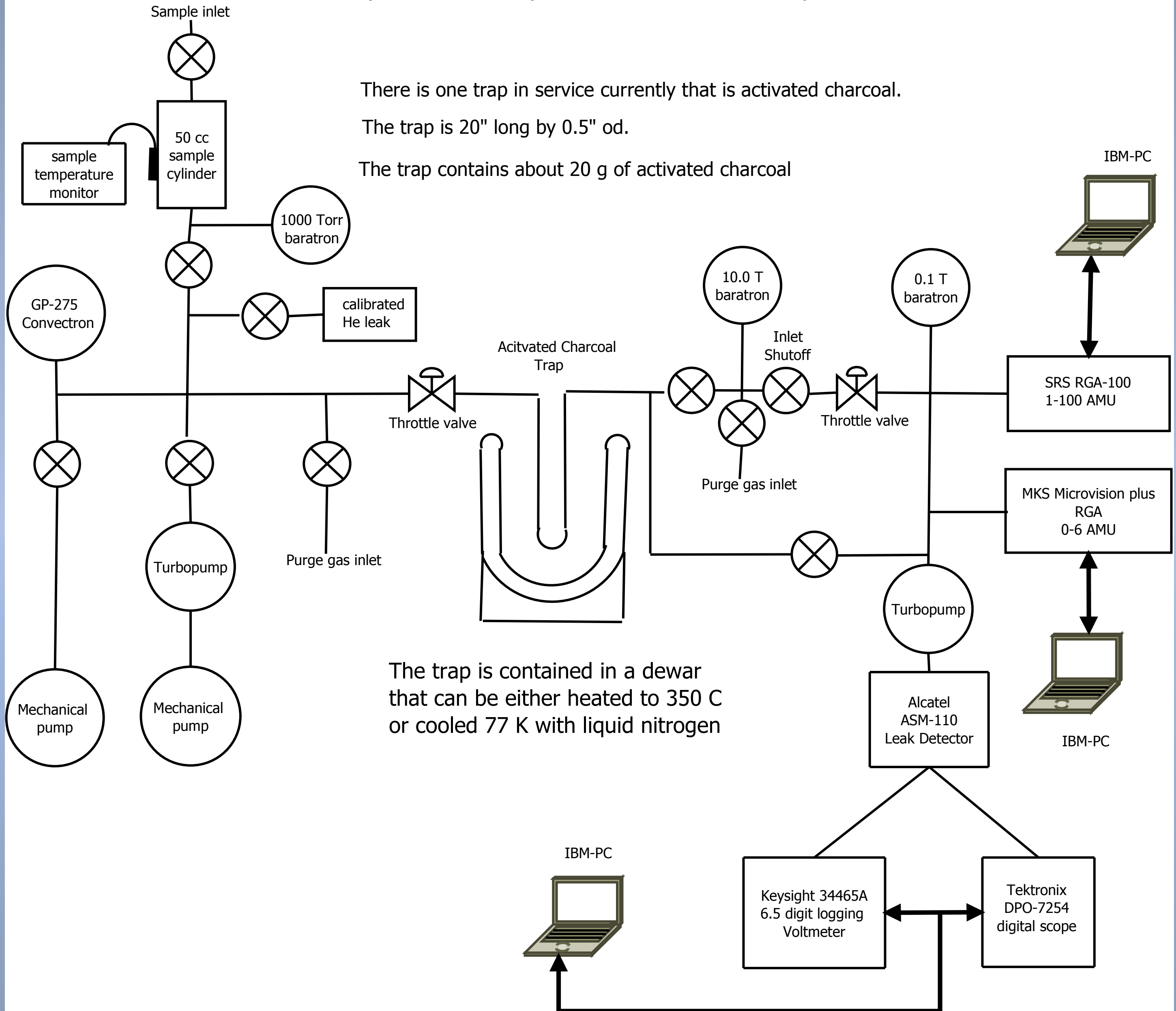

Estimation of the total amount of helium and heat produced in the recent gas discharge experiments

- Gas Discharge system contains about 2400 cc of D_2 at 350 Torr, or 8.4×10^5 Torr-cc.
- The analyzed samples contain 2500 Torr-cc, or about 0.3% of the total.
- The last 5 samples samples analyzed ranged from 20 to 70 ppb He with a fairly large uncertainty.
- This would correspond to 0.017 to 0.059 Torr-cc of He. or about 5.5×10^{14} to 1.9×10^{15} helium atoms.
- If the reaction was d+d ---> He, and the helium survived, the helium would represent 2100 to 7100 Joules of heat spread over 24 hours.
- This would correspond to 24-82 mW while the discharge is several watts.

Summary

- A system has been developed that can measure small amounts of helium in gas samples.
- Currently, we can measure the amount of helium in air at ambient levels (5.26 ppm) with an uncertainty of about 10% using a sample of 50 cc at 50 Torr pressure to make the measurement.
- The minimum detectable amount of helium is estimated to be about 3 ppb for the same size sample.
- The measurement requires about 1 hour to complete, but then requires several hours to warm and regenerate the traps to prepare for the next sample.
- We have run about 150 analyses to date with most of the samples being standards and blanks.
- We detect small amounts of helium in almost every gas sample we run.
- We may consider adding a getter in the future, although it does not seem to be necessary at this time.
- The most recent standard runs have a scatter of ~5% at the one standard deviation level.
- The the baseline measurement is equivalent to about $1x10^{-10}$ atm-cc/sec with an uncertainty of about 30%.
- This value integrated over 100 sec that the He signal is being measured corresponds to about $1x10^{-8}$ atm-cc or $7.6x10^{-6}$ torr-cc, equivalent to 3 ppb in our standard sample.

We would like to acknowledge the continued support and encouragement for this work by:

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Mr. Thomas Francis Darden II, J.D.
Manager, President, and Director

Mr. J.T. Vaughn
Vice President

Mr. Dewey Weaver

Questions?

Estimate the amount of He in various air samples and the energy release if it came from fusion

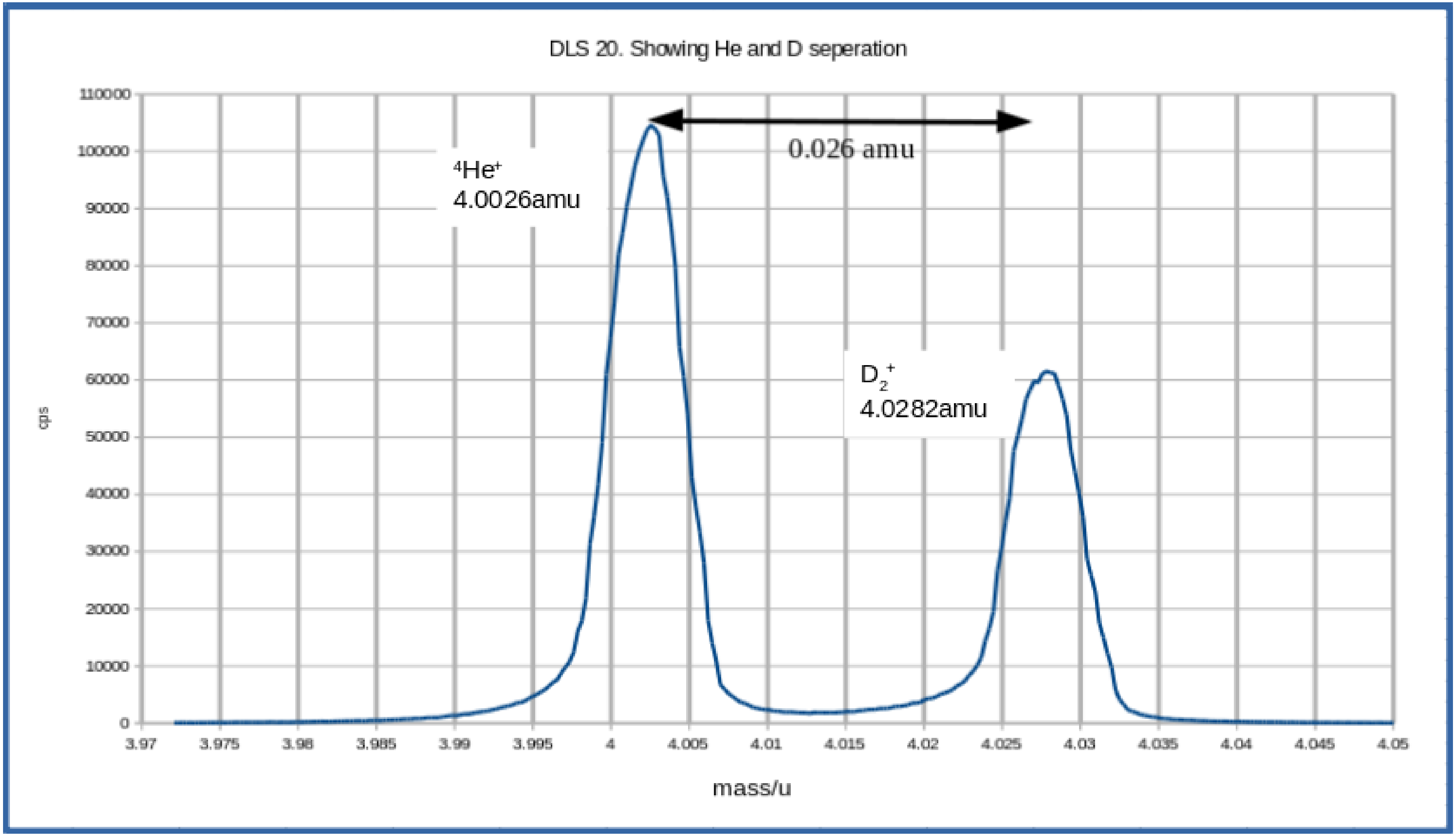
Assuming the concentration of He in the air samples is 5.26 ppmv

Malcolm Fowler, 10/7/2015

	volume	pressure			moles	moles of He	atoms of He	energy release, based on D+D→He	
	cc	PSIA	Torr	Atm				MeV	joules
			2585.0340						
	10	50.0	14	3.40136	0.00139	7.31718E-09	4.40714E+15	1.05771E+17	16.9
	350	3.9	200	0.26316	0.00377	1.98141E-08	1.19341E+16	2.86417E+17	45.9
	1725	19.4	1001	1.31711	0.09292	4.88765E-07	2.94383E+17	7.06520E+18	1131.8
	1725	12.3	636	0.83684	0.05904	3.10544E-07	1.87041E+17	4.48898E+18	719.1
	50	12.3	636	0.83684	0.00171	9.00128E-09	5.42147E+15	1.30115E+17	20.8
	100	12.3	636	0.83684	0.00342	1.80026E-08	1.08429E+16	2.60231E+17	41.7
	150	12.3	636	0.83684	0.00513	2.70038E-08	1.62644E+16	3.90346E+17	62.5
check:	24451	14.7	760	1.00000	1.00000	5.26002E-06	3.16811E+18	7.60347E+19	12180.8

DLS-20

Ultra high resolution quadrupole mass spectrometer featuring 20mm pole diameter – specifically designed for the analysis of hydrogen and Helium isotopes and light gases.


DLS 20. Showing He and D seperation
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
cps
3.97
3.975
3.98
3.985
3.99
3.995
4
4.005
4.01
4.015
4.02
4.025
4.03
4.035
4.04
4.045
4.05
mass/u
0.026 amu
$^4He^+$
4.0026amu
D_2^+
4.0282amu