TESTS OF THE ACTIVATED-CHARCOAL GETTER

Spartan IR Camera for the SOAR Telescope

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Activated coconut charcoal will be used in the Spartan IR Camera as a getter to maintain the vacuum for a period of a year. Two experiments were conducted to test the charcoal getter. The first tested the effectiveness of low temperature epoxy for holding charcoal on aluminum. The second determined how efficiently nitrogen gas and air are absorbed by the charcoal getter. We find that the getter can keep the pressure low for a year and that hydrogen is the dominant gas in the dewar.

1 Test of the Low-Temperature Epoxy

The activated coconut charcoal will be glued to the wall of the liquid nitrogen dewar inside the cryo-optic box. This test was designed to determine whether a particular brand of low temperature epoxy will retain the charcoal at liquid nitrogen temperatures.

1.1 Apparatus

Two 30×180×12-mm aluminum blanks were used as the surfaces to bond the activated charcoal to. A hole was drilled in the end of each blank so that the samples may be suspended by a string. The charcoal used in all three of the experiments was an activated coconut charcoal with a mesh size of 8-30 made by Spectrum Inc. The low temperature epoxy used in these trials was the 5-Minute Epoxy manufactured by ITW Devcon.

1.2 Experimental Details

The surface of the aluminum blank was cleaned using denatured alcohol. A 2mm thick layer of epoxy was then spread over one surface of the aluminum. Charcoal was sprinkled onto the layer of epoxy and pressed down using a sheet of paper as a barrier. Once the functional curing time of the epoxy was reached (1 hour), excess charcoal was removed by inverting the blank and shaking it.

The prepared sample was then wrapped in a plastic bag and the bag was sealed with masking tape. The entire apparatus was then lowered into a vat of liquid nitrogen via a string tied through a hole drilled in one end of the aluminum. When violent boiling ceased the aluminum was removed from the vat and allowed to return to room temperature. The plastic bag was then pulled away from the blank and its contents were inspected.

1.3 Results

Upon examining the contents of the plastic bag following the first trial it was clear that a thicker layer of epoxy would be needed to ensure adequate adhesion. Even after the epoxy layer thickness was increased to 2mm some charcoal was found in the bottom of the bag. Flaking was significantly reduced but not eliminated by increasing the amount of epoxy used.

1.4 Conclusions

The layer of epoxy bonding the charcoal to the wall of the dewar must be at least 2mm thick. A PTFE filter membrane with 0.2 micrometer pores will be used to prevent charcoal flakes from contaminating the camera. The filter holder will be designed in such a way as to allow for the use of these filter membranes.

2 Residual Gas Analyzer

The mass spectra of the residual gas analyzer (RGA) are difficult to interpret, because the RGA breaks apart and ionizes gas molecules with efficiencies that depend on the gas species and on the details of the particular RGA. For example, one RGA ionizes water 11 times and H_2 0.44 times as efficiently as N_2.[1]

We use an RGA100 residual gas analyzer built by Stanford Research Systems. It detects molecules with masses 1–100 amu.

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[1] Umrath, W., ed., 1998, *Fundamentals of Vacuum Technology*, Schoonover Inc, Atlanta, GA, www.schoonoverinc.com, p. 104.

We measured the mass spectrum with the RGA open to the system and also with the RGA closed off from the system (Figure 1). The pressure of two species with mass 12 and 18 is *higher* when the system is shut off. Carbon and water are primarily due to the RGA itself. (The identifications are unique for these masses.) The pressure of the species at 14, 16, and 44 amu is slightly lower when the system is shut off. The RGA produces almost as much of these as does the system. Finally, H2 and the species at 28 amu are caused by the system.

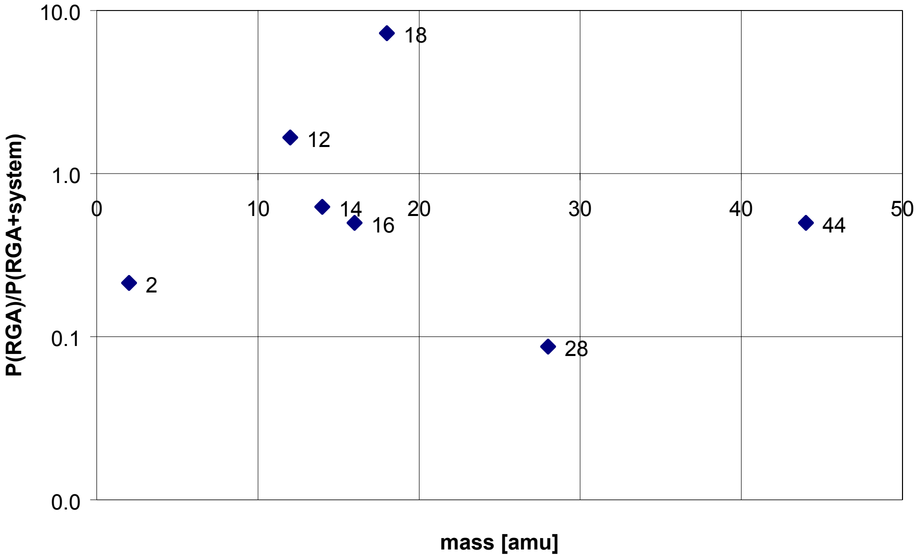

Figure 1 Ratio of the pressure with the RGA shut from the system to the pressure with the system open

3 Adsorbance of Nitrogen and Air

This test evaluated the ability of a sample of charcoal to soak-up trace amounts of nitrogen and air under conditions approximating those that will be found inside the camera box. Over time trace amounts of air will permeate through the o-ring seal.

3.1 Apparatus

The apparatus for the nitrogen absorbance test consisted of a Precision Cryogenic Systems, Inc. liquid nitrogen dewar containing a 10 gram sample of charcoal epoxied to an aluminum disk and covered by a Teflon membrane. The disk and an aluminum ring used to hold down the membrane were bolted to the underneath side of the liquid nitrogen can using a single 4-40 stainless steel bolt. The dewar was evacuated by means of ¼” Swagelok connections to a Varian Triscroll mechanical roughing pump. Internal pressures were monitored by means of Granville-Phillips Convectron and Ion Gauges. The compositions of residual gases present in the dewar were deduced using an SRS RGA 100 Residual Gas Analyzer.

3.2 Experimental Details

10 grams of activated coconut charcoal were epoxied to an aluminum disk, using the method described in the above section, covered with a 0.2 μm PTFE Filter Membrane manufactured by the Cole-Parmer Instrument Company, and fastened underneath the liquid nitrogen dewar using a stainless steel bolt. Upon sealing the dewar and connecting, via the Swagelok valves, to the vacuum pumps, the dewar was evacuated. The mechanical roughing pump was used to bring the pressure down from atmospheric to 2.8 mTorr. Cryogenic pumping by means of liquid nitrogen addition was used to bring the pressure down to the maximum operating pressure of the RGA (0.1 mTorr).

Ten “charges” of nitrogen gas collected at the exhaust port of the dewar were then added to the system. Each charge was approximately 3 cc in volume. The Swagelok valves were used to accurately administer these charges of nitrogen. The charcoal was allowed to absorb gases undisturbed overnight. Pressure within the dewar was measured before the addition of the 10 nitrogen charges and then again the following day.

For the tests with air, ten charges of air were added.

3.3 Results

Before and after addition of the 10 charges of nitrogen, the pressure was 0.078 mTorr. Therefore the pressure rise is less than 1μTorr.

We determined the composition of the gas with the RGA (Fig. 2, Fig. 4, and Table 1). To interpret the spectra, we use Table 4.5 of reference 1.

- Neon, argon, and helium are not detected.

Table 1 Masses present before and after addition of nitrogen and of air.

Mass	ID	Pressure [nTorr]			
[amu]		Addition of N2		Addition of Air	
		Before	After	Before	After
2	H2	484	176	480	155
12	C	53	47	48	40
14	N	48	41	62	39
16	O	33	34	38	32
17	H2O	2	4	7	3
18	H2O	10	13	23	11
28	N2, CO	16	17	33	18
44	CO2	6	8	20	9
Total		652	340	711	307

- Hydrogen is the most abundant species.

- The peaks at 12, 14, and 16 are probably due to the RGA. These are seen even when the RGA is isolated from the system.

    - The peak at 14 amu could be atomic nitrogen. In that case, the peak at 28 amu corresponding to diatomic nitrogen should be 14 times higher. The other possibility is a fragment of CH4, but then peak at 16 and 15 should be 5 times larger.

    - The peak at 16 amu is atomic oxygen. However, there should be a peak at 32 amu (diatomic oxygen) that is 9 times larger, but no peak is visible in that region of the spectra.

    - The carbon peak at 12 amu is too high. This peak should correspond to carbon fractionated by the RGA from carbon monoxide and carbon dioxide. If this were the case, the carbon peak should only be a fraction (1/25th and 1/16th respectively) of the height of the peaks corresponding to these two compounds. It is, in reality, much higher than either of these peaks.

- Water is present in the spectrum both at 18 and 17 amu. The peak at 17 amu due to OH is 25% as high as the peak at 18 amu.

- CO2 is present.

- The peak at 32 amu could be either CO or water.

The sum of the partial pressures is a factor of 110 and 230 lower than the pressure measured by the ion gauge. Perhaps the RGA calibration is wrong; we did not calibrate it. Perhaps the pressure is not at equilibrium, and the pressures at the gauge and at the RGA are different. The fact that the pressure on the ion gauge is the same for all four cases in Table 1, and yet the pressures on the RGA are different supports the hypothesis.

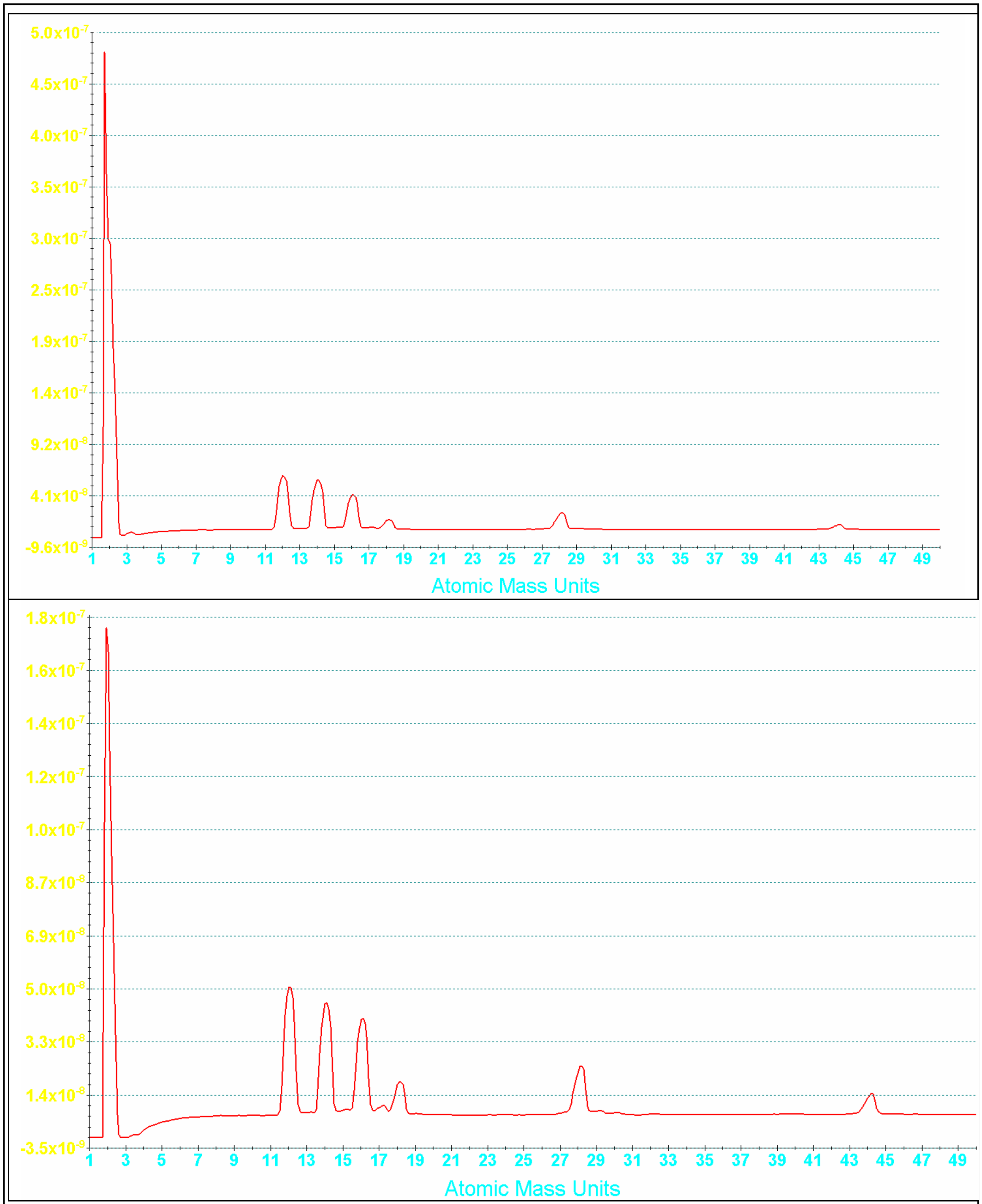

Figure 2 Mass spectrum taken before (upper panel) and after (lower panel) addition of nitrogen. The vertical axis is in Torr.

3.4 Discussion

The results may depend more on the history of the dewar than on the addition of nitrogen or air. The final mass spectrum is the same after addition of air or nitrogen ("postAir/postN2" in Figure 3). Immediately after the spectrum "postN2", the spectrum "preAir" was taken. However the two do not agree. A possibility is that the RGA was not allowed enough time to stabilize. The pressure in the ion gauge rose when the RGA was turned on, and the spectrum was taken when the pressure on the ion gauge ceased to change. Since the RGA is about 6" from the ion gauge, its influence on the pressure on the ion gauge is smaller than the remainder of the system.

In spite of these problems, we can conclude that H_2 dominates and He, O_2, Ar, and Ne are not seen.

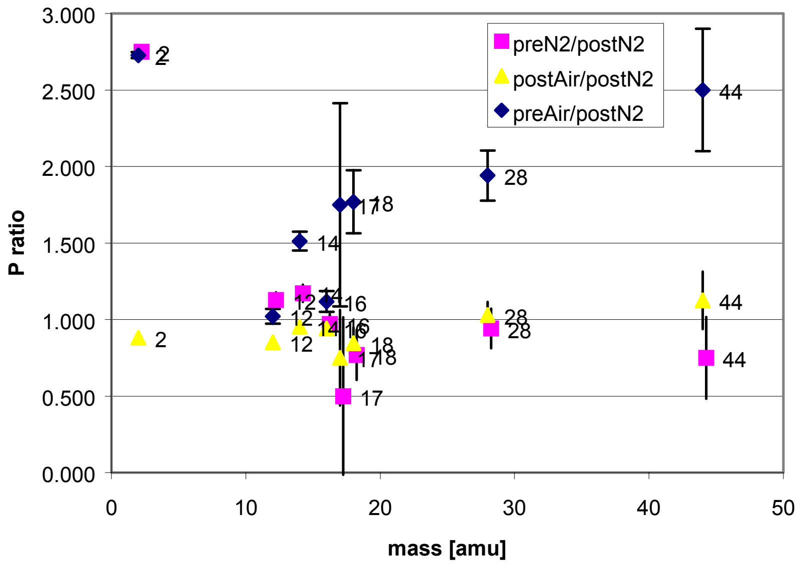

Figure 3 Pressure ratios for the addition of air or nitrogen

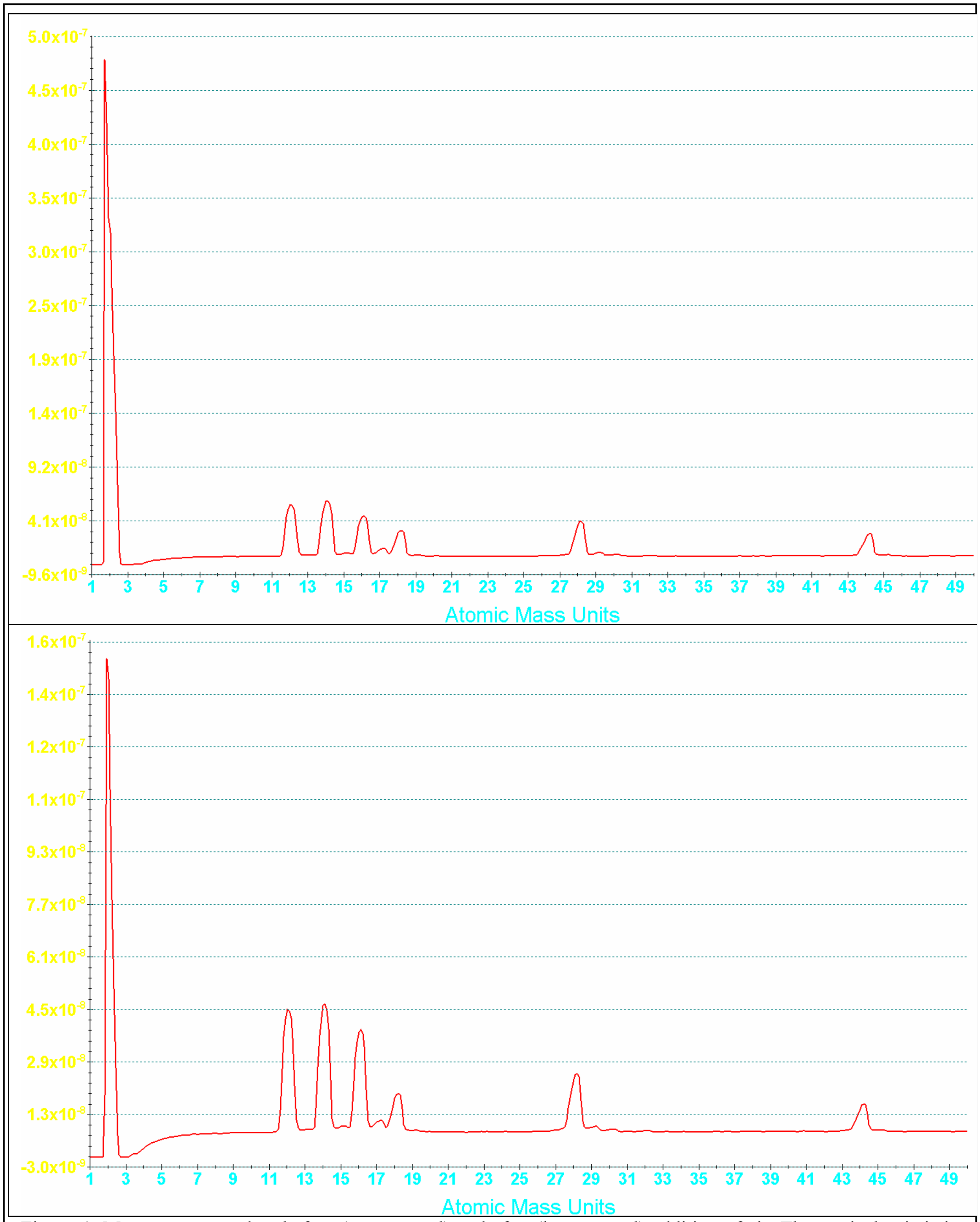

Figure 4 Mass spectrum taken before (upper panel) and after (lower panel) addition of air. The vertical axis is in Torr.

4 Conclusions

Gluing 10g of charcoal to the wall of the liquid nitrogen dewar using low-temp epoxy will ensure that the temperature of the charcoal is low enough for it to be an effective getter. Sealing the filter plate behind a Teflon filter membrane will prevent any of the charcoal from flaking off into the optical box.

From the test with the injection of air we draw several conclusions. The injected pressure of each gas species and the measured pressure are shown in Table 2. The remnant ratio, defined as the ratio between the remaining pressure and the injected pressure, is the key quantity.

- The hydrogen abundance changes even though the getter cannot absorb hydrogen. Some possibilities are that hydrogen is trapped in the getter, is in the metal, or is produced by a chemical reaction.

- The charcoal getter pumps oxygen, nitrogen, argon, and CO2 as expected, since these species have boiling or sublimation temperatures higher or equal to that of nitrogen. The remnant ratio for oxygen is less than 8×10^{-8}. The remnant ratio for nitrogen is less than 2×10^{-7}. We cannot separate N2 and CO. The higher remnant of CO2 indicates its source is not only the injection of air.

- Surprisingly, the charcoal getter pumps neon, which has a very low boiling point. There is some evidence that the getter even pumps helium, although we do not trust the calibration enough to make the statement firmly.

Table 2 Effectiveness of the charcoal getter. ratio between measured pressure and injected pressure. The calibrated pressure accounts for the ionizing efficiency and an overall calibration factor for the RGA, which is assumed to be the ratio of the sum of the partial pressures and the pressure on the ion gauge.

	% Air	T(boiling) [K]	P(injected) [μTorr]	P(meas,uncal) [μTorr]	Ionizing Eff.	P(meas,cal) [μTorr]	P(meas)/P(inj)	Notes
N2	78.1	77	3.6E+07	? 0.017	1	? 6.8	? 2.E-07	1
O2	20.93	90	9.5E+06	< 0.002	1	< 0.8	< 8.E-08	
Ar	0.93	87	4.2E+05	< 0.002	1.2	< 0.7	< 2.E-06	
CO2	0.03	195	1.4E+04	0.008	1.4	2.3	2.E-04	2
Ne	1.80E-03	27	8.2E+02	< 0.002	0.23	< 3.5	< 4.E-03	
He	7.00E-05	4	3.2E+01	< 0.002	0.14	< 5.7	< 2.E-01	
H2	5.00E-05	20	2.3E+01	0.176	0.44	160.0	7.E+00	3

Notes
1. Mass spectrum cannot distinguish between N2 & CO.
2. P(meas) is the total pressure; the pressure of CO2 decreased by a factor of 2 with the addition of air.
3. P(meas) is the total pressure; the pressure of H2 decreased by a factor of 3 with the addition of air.

Does the charcoal getter keep the pressure in the instrument below 30μTorr for a year?

We estimate the pressure of each gas species (Table 3). If the source is permeation through the o-rings, we assume the ratio of the pressure to the injected pressure is the same as our measurement, although this depends on geometry. We use the permeation of Viton o-rings in Appendix C6 of O'Hanlon, John F., 1980, A User's Guide to Vacuum Technology, Wiley, New York, 1980. (We assume the permeation of Ar and Ne is the same as that of H2.) For the species that are dominated by outgassing from surfaces, the amount of gas is proportional to $V^{2/3}$, where V is the volume of the dewar, and the pressure is proportional to $V^{-1/3}$. We scale our measurement by $500^{-1/3}=1/8$ (The vacuum enclosure of the camera is 500 times larger than that of the laboratory dewar.)

Table 3 Expected pressure after 1 year

	Source	Perm/ Perm(N2)	P(1yr) [μTorr]	
N2	o-ring	1	<	0.001
O2	o-ring	22	<	0.004
Ar	o-ring	44	<	0.006
CO2	wall	118		0.29
Ne	o-ring	44	<	0.03
He	o-ring	178	<	0.21
H2	wall	44		20
Total				21
Total permeation				0.3

The expected pressure in the camera after one year is lower than 30μTorr. Hydrogen and CO2, the two species caused by outgassing, dominate the pressure. If H2 and CO2 are removed (by different methods of cleaning or by a hydrogen-active getter), then the pressure after one year is better than the requirement by a factor of 1000.