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CALIFORNIA INSTITUTE OF TECHNOLOGY  
MASSACHUSETTS INSTITUTE OF TECHNOLOGY

<b>Document Type</b>	<b>LIGO T990186-00-E</b>	08/22/99
<b>Technical Note</b>		
<b>Water Load on the Beam Tubes from Detector Components</b>		
Rainer Weiss		

*Distribution of this draft:*

Vacuum Review Board

This is an internal working note  
of the LIGO Project..

**California Institute of Technology**  
**LIGO Project - MS 51-33**  
**Pasadena CA 91125**  
Phone (818) 395-2129  
Fax (818) 304-9834  
E-mail: info@ligo.caltech.edu

**Massachusetts Institute of Technology**  
**LIGO Project - NW 17 - 161**  
**Cambridge, MA 01239**  
Phone (617) 253-4824  
Fax (617) 253-7014  
E-mail: info@ligo.mit.edu

WWW: <http://www.ligo.caltech.edu/>

### Summary and Conclusion

The water outgassing by the seismic isolation stacks is sufficiently large that we need to take actions to reduce the risk of contaminating the beam tube for future LIGO detectors. The most likely source is water in the Fluorel spring seats that have been treated by the Walker Flourine cleaning process. There are many ways to cure the problem. I favor the following steps but you may not agree, this is the reason for a meeting.

1) **For new installation:** continue the Walker process to remove Flourine from the surface of the Fluorel but subject the seats to a vacuum bake as the final step. The specific temperature and time needs to be determined with the aim to reduce the stored water in the rings below 1mg and also to minimize the amount of new Flourine brought to the surface. Experiments are needed to establish the temperature and time to remove the water with the minimum production of Flourine.

2) **For existing installation in the midstation in Washington and the LVEA in Livingston** : remove the seats and replace with baked seats.

3) **For existing installation in the LVEA:** a gentle heating (50 to 125C) in vacuum with Cal-rods mounted on the isolation system metal masses in contact with the spring seats . To avoid contamination on the optics, use clean aluminium foil covers around the test masses to reduce the chance of direct deposition. It is not necessary to heat the entire system since water that has been transported to the stainless steel surfaces will pump out much more rapidly than that stored in the Fluorel.

### Introduction

The question raised for the Vacuum Review Board is what conditions must be satisfied in the LVEA vacuum system to allow opening the gate valves to the baked beam tubes. This note addresses the question in the context of the current state of the residual gas in the LVEA vacuum system and evaluates various alternatives opened to the project.

The topics discussed are:

- 1) A review of the outgassing requirements in the beam tubes to accomodate the performance of advanced detectors - the goal pressure and associated outgassing rate.
- 2) The relation between surface loading of the beam tube and the outgassing rate.
- 3) The transmission of condensible gas by the liquid nitrogen isolation traps.
- 4) Measurements of the detector components gas load and the rate of change of the load with time and temperature.

#### 1) Review of the requirements

The goal for the residual gas in the LIGO beam tubes was set at a phase noise equivalent to a strain amplitude spectral density of  $h(f) \leq 1.5 \times 10^{-25} 1/\sqrt{\text{Hz}}$  @ 100 Hz

which corresponds to 1/2 of the noise associated with the quantum limit of a 1 ton mass in a search for periodic sources. Using the molecular polarizability and thermal velocity of the residual gas, the strain noise specification leads to permitted molecular column densities in the beam tube expressed as an average pressure and outgassing rate given in **Table 1**.

**Table 1: Goal average pressure and outgassing rates in the beamtube @ 300K**

<i>constituent</i>	<i>average pressure</i>	<i>end pump outgassing rate</i>	<i>nine pump/module outgassing rate</i>
	<i>torr</i>	<i>torr liters/cm<sup>2</sup>sec</i>	<i>torr liters/cm<sup>2</sup>sec</i>
amu 18 (H <sub>2</sub> O)	$1 \times 10^{-10}$	$2 \times 10^{-15}$	$2 \times 10^{-14}$
amu 100	$6 \times 10^{-13}$	$5 \times 10^{-18}$	$4 \times 10^{-16}$
amu 300	$5 \times 10^{-14}$	$2 \times 10^{-19}$	$1 \times 10^{-17}$
amu 600	$8 \times 10^{-15}$	$3 \times 10^{-20}$	$2 \times 10^{-18}$

The pumping speed assumed is the largest value possible consistent with the port size or chosen to have the length dependent pressure term dominate; for example, for water 4000 liters/sec/port.

## 2) Relation between surface loading of the beam tube and the outgassing rate of water

In lieu of actually doing the experiment of dumping a known amount of water back into the beam tube and measuring the change of equilibrium pressure and outgassing rate (an experiment we should have carried out in the beam tube bakeout demonstrations), the approach is to use the reasonably successful theory that has been developed to describe the adsorption on the surface presented in. “*Outgassing Documents from 1988 - 1992*” LIGO T920009-00-R based on the Dubinin-Radushkevich energy distribution of adsorption sites and the Langmuir theory of surface adsorption.

The water removed from a two km beam tube module during initial pump down and the bakeout is measured to be close to  $5 \times 10^5$  torr liters (25 moles = 450 grams). The initial surface coverage of water is 190 monolayers or about  $1.9 \times 10^{17}$  molecules/cm<sup>2</sup>. This becomes one of the model parameters, the saturated surface density - the number of binding sites for water on the surface. The bakeout was carried out after about 1 month of active pumping. The bakeout is measured to remove about  $3 \times 10^4$  torr liters or the equivalent of 11 monolayers. The model gives that 6.4 monolayers of tightly bound water still remain on the surface after the bakeout, all with binding energies greater than 13000K. The average binding energy of the skewed Gaussian distribution of binding sites is  $T_0 = 10000K$  which is determined from the pressure vs temperature data at the beginning of the bake. The measured outgassing rate of water at 300K after the bake is  $J < 2 \times 10^{-17}$  torr liters/sec cm<sup>2</sup>. The model gives  $J = 4 \times 10^{-17}$  torr liters/sec cm<sup>2</sup>, which is reasonable agreement for such calculations (the model could be further refined to adjust the repulsive part of the surface potential to give better agreement; the value currently being used is a barrier height of 0.33 of the binding energy.)

To answer the question of how much water can be injected into the beam tube from the detector components, the model was run with the best estimates of the three model parameters determined in the pump down and bake: the number of sites / area, the average binding energy and the magnitude of the repulsive potential at the surface. Varying amounts of water vapor were introduced into the tube through an increase in the pressure. The increase in surface coverage and the change in the outgassing rate was determined from the model and is given in **Table 2**.

**Table 2: Model results: increase in outgassing rate with water injected into the baked beam tube**

<i>injected water</i>	<i>surface load</i>	<i>water outgassing rate @ 300K</i>
<i>torr liters</i>	<i>monolayers</i>	<i>torr liters/sec cm<sup>2</sup></i>
0	6.4138	$4 \times 10^{-17}$
25	6.4233	$2 \times 10^{-16}$
225	6.50	$1.3 \times 10^{-15}$
485	6.60	$2.6 \times 10^{-15}$
1500	7.0	$8.1 \times 10^{-15}$

The results of the modeling indicate that we cannot introduce more than 400 torr liters of water into the beam tube before hitting the goal for end pumping only and approximately 4000 torr liters if we utilize all 9 ports at maximum pumping capacity.

### 3) Transmission by the Liquid Nitrogen Isolation traps

The traps at the two ends of the beam tube are designed to provide a large pumping speed for condensable gases. They have a pumping speed, determined by the geometry, of about  $10^5$  liters per second at their openings for water and hydrocarbons. Molecules destined to the trap wall stick without bouncing when the trap is clean. Some molecules not headed to the trap surface will be transmitted from the detector into the beam tube. To estimate the amount of gas transmitted, one establishes the pressure at the plane entering the trap at the detector side and calculates the one way flux of molecules per solid angle. The flux is given by

$$\frac{d\dot{Q}}{d\Omega} = \frac{\rho v A}{4\pi}$$

where  $\rho$  is the molecular particle density at the entrance of the trap,  $v$  the molecular speed and  $A$  the cross section of the trap entrance. (Note: this method of calculation takes into account the multiple encounters with the various surface between the detector components and the trap.) The transmitted molecular flow is the flux times the solid angle transmitted - the cross sectional area of the trap divided by the cold surface trap length.

A subtle issue is the longitudinal distribution of the transmitted gas adsorbed on the beamtube surface. Initially when the tube is still uncontaminated, most of the low energy binding sites are

unpopulated and most of the water molecules hitting the beam tube wall will stick to it. Under these conditions the rate of increase in surface coverage on the tube wall will vary with distance into the tube,  $s$ , as

$$J = \frac{\rho v a^3}{4s^3}$$

There is a great deal more coverage at the tube entrance than further down the tube. The uneven coverage is not relevant to the phase noise calculation since it is the total column density that forward scatters that makes the phase noise (and we are not in a situation where absorption by the gas is a factor). It is, however, an issue when estimating the column density from the outgassing rate. Adsorbed gas at the tube end will be more readily pumped than gas near the middle of the tube. So it might appear there is a big factor to be gained by this consideration. I currently believe it is no more than a factor of two increase in the allowed beamtube contamination because of the way the molecular diffusion works - on desorption from the surface the gas is as likely to go toward the pump and populated sites (which don't stick) as it is to go further into the tube to binding sites that do stick. The outgassing models that have been developed all assume spatially uniform surfaces. The properties of the outgassing of an inhomogeneous surface would be a good problem for an undergraduate interested in statistical mechanics.

**Table 3: Isolation trap properties**

<i>property</i>	<i>LVEA trap</i>	<i>Mid and end station trap</i>
length	371 cm	122 cm
full aperture	134 cm	134 cm
vignetted aperture	112 cm	112 cm
full transmission	$3.3 \times 10^{-2}$	$3.0 \times 10^{-1}$
vignetted transmission	$2.3 \times 10^{-2}$	$2.1 \times 10^{-1}$
Lazzarini cold aperture	30 cm	30 cm
Lazzarini transmission	$1.7 \times 10^{-3}$	$1.5 \times 10^{-2}$

#### 4) Estimate of the gas load into the beam tube

The gas load is estimated from the pumpdown of the midstation, while the time dependence of the outgassing is determined from both the pumpdown of the mid station as well as the LVEA consisting of BSC4+HAM7+HAM8+HAM9+HAM10.

The trigger that led to this document is the observation at both Washington and Livingston that the chambers once loaded with the seismic isolation stacks seemed to be taking an inordinately long time to pumpdown. Simple estimates showed that the gas loads were around  $10^{-2}$  torr liters/sec after some 100's of hours of pumping and that there was little change in time. The estimates for

the constituents of the gas load are given in **Table 4**

**Table 4: Outgassing parameters for mid station**

<i>item</i>	<i>area</i>	<i>outgassing rate</i>	<i>gas load</i>
	$cm^2$	$torr\ liters/cm^2sec$	$torr\ liters/sec$
<b>Data</b> at 294K after 310 hrs			$1.2 \times 10^{-2}$
stainless steel	$1.8 \times 10^6$	$4 \times 10^{-9} /t(hr)$	$7.2 \times 10^{-3} /t(hr)$
viton O rings (PSI values)	$6.3 \times 10^3$	$3 \times 10^{-8} / \sqrt{t(hr)}$	$1.9 \times 10^{-4} / \sqrt{t(hr)}$
flourel seats (current)	$2.4 \times 10^4$	$7.2 \times 10^{-6} / \sqrt{t(hr)}$	$1.7 \times 10^{-1} / \sqrt{t(hr)}$
flourel seats (best prior)	$2.4 \times 10^4$	$6 \times 10^{-9} / \sqrt{t(hr)}$	
flourel seats (worst prior)	$2.4 \times 10^4$	$6 \times 10^{-8} / \sqrt{t(hr)}$	
wiring	$1.2 \times 10^4$		

The Flourel water outgassing listed in the table is assumed to vary as  $1/\sqrt{t(hr)}$  for the sake of consistency with the rest of the data on diffusion from Fluorocarbon rubbers. The measured time dependence varies as  $1/t^n$  with  $n$  between 0.8 to 0.5. This will be discussed below.

The Walker @ Sons treated Flourel seats are the most likely source of the excessive water load. Some indication of this comes from weight loss measurements performed on the seats after a 250C bake in vacuum. The weight loss for two treated seats was 213 and 222 milligrams out of total weight of 38 gm, a fractional loss of about 1/2 % which is an excessive amount for flourocabons such as Flourel which have weight loss listed as less than 0.1%. Another indication comes from the paper by Kemo Welch et al (Vacuum **41**,1924 (1990)) in which unbaked Viton is listed as having an outgassing rate  $2 \times 10^{-5}/t(hr)^{0.6}$  torr liters/sec  $cm^2$ , a value comparable with what is now being measured from the seats.

The total amount of water in a seismic isolation stack assembly is about 3.4 moles or about  $7 \times 10^4$  torr liters, so that even without detailed calculations, it is easy to see that with a trap transmission of 20% , the amount of water injected into the tube eventually will exceed the allowed amount by a factor of 30.

The time dependence of the outgassing is critical in devising a strategy. The best data we currently have comes from the discharge gauge readings in the LVEA and in mid station at Hanford. RGA spectra from the mid station show that the water in the residual gas is about 4 times larger than all other peaks in the spectrum. It is assumed that the LVEA data is also dominated by the water but I have not seen RGA spectra from this system. **Figure 1** shows the pumpdown of the LVEA system

which has a mixture of treated and untreated seats. The system is pumped by a 1800 liters/sec turbo pump. The absolute value of the pressure is not as useful as the data from the mid station because of the mixture of seats but the outgassing rate of change with time is useful.

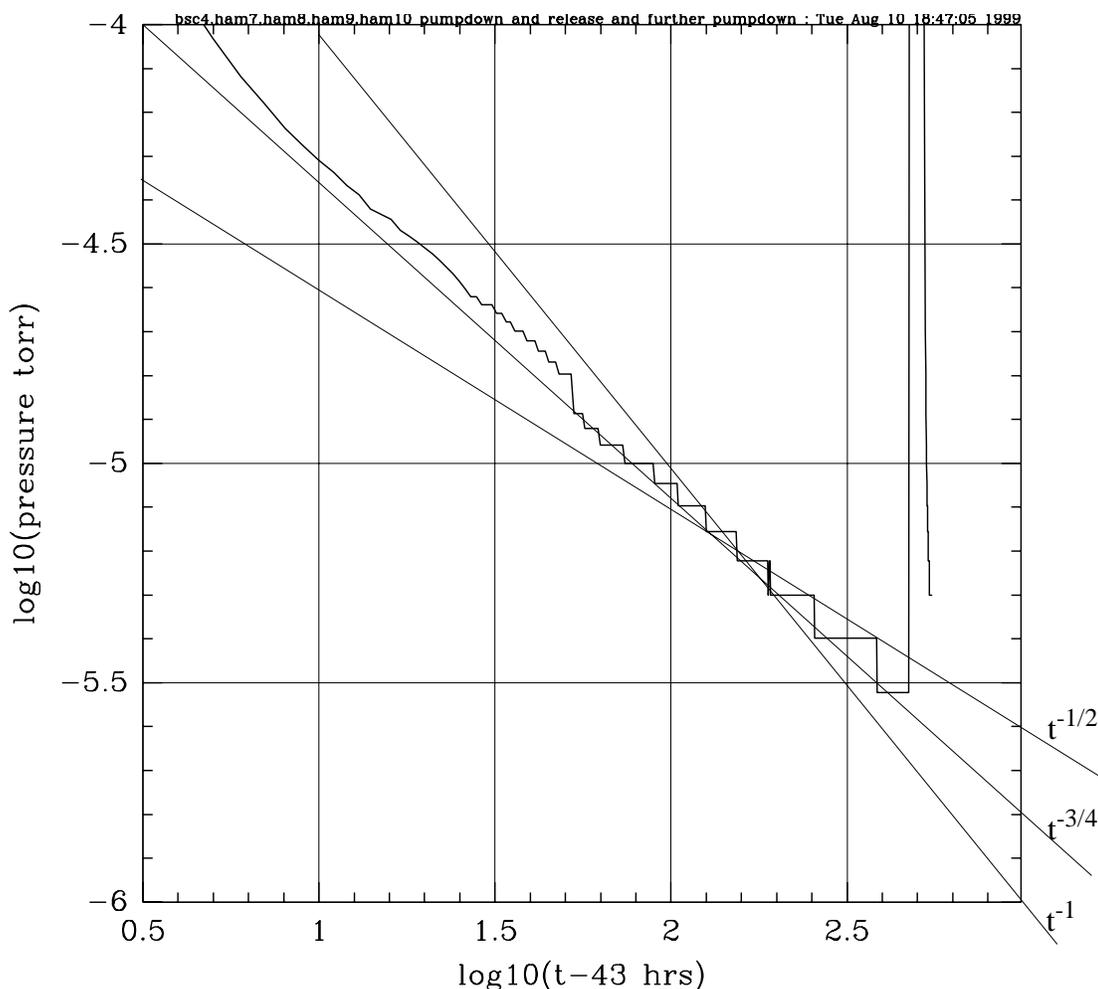
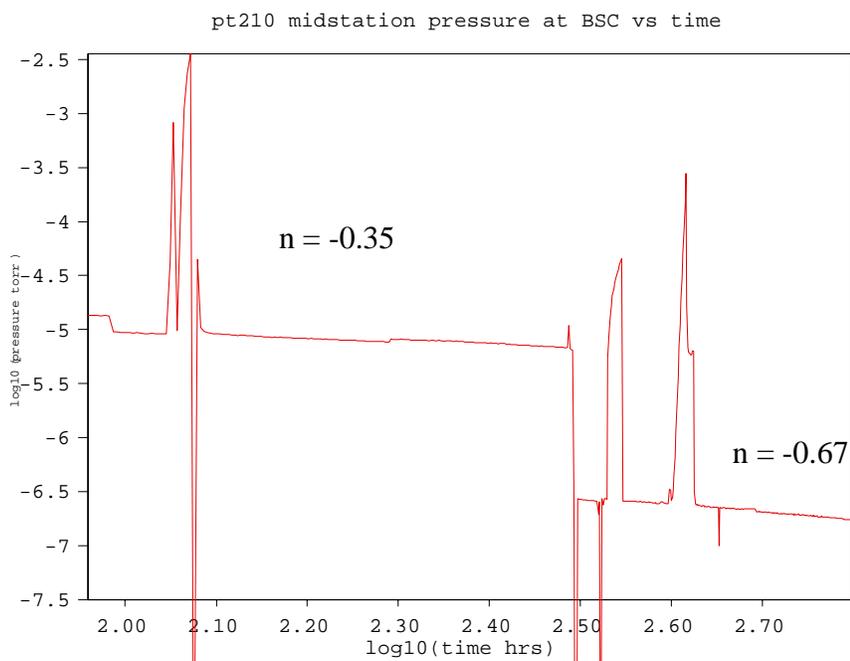
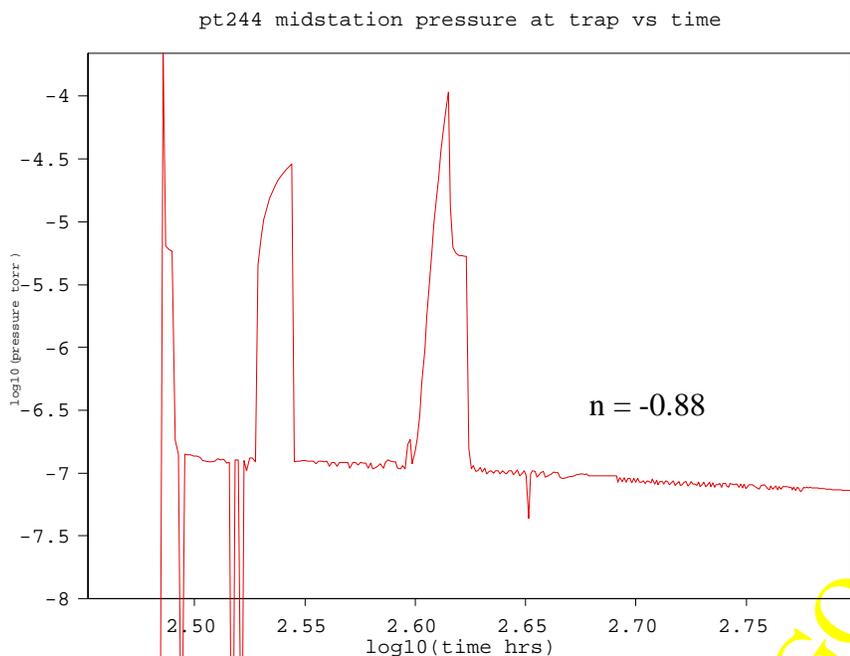


Figure 1 Total pressure as read on a discharge gauge vs time in the LVEA system consisting of BSC 4, ham7,8,9 and 10. The lines show various power law outgassing functions, the closest to fitting (by eye) is the  $n = -3/4$  dependence which would be a mixture of diffusion with surface desorption. An interesting feature is the air refill which takes place at the right showing that there is little repopulation of the water into the slow processes which dominate the power law dependence - additional evidence that the metal walls desorb quickly and the Flourel is not repopulated with water. The initial charge of water dominates the rates.

The data from the midstation is shown in Figure 2a and 2b. The pumping speed for water of the 80K pump at the entry to the pump is  $1 \times 10^5$  liters/sec. Although it may be fortuitous, since the gauges have not been compared, the flow determined from the pressure gradient in the pipe also indicates a gas load of  $1 \times 10^{-2}$  torr liters/sec which agrees with the flow determined from the pressure measured under both turbo and 80K pumping conditions.



**Figure 2a** Pumpout of the mid station as measured on a discharge gauge mounted on the BSC. The initial pumpdown was carried out with a turbo pump. At about 300 hours the 80K isolation trap was filled. The ratio of the pressures before and after the filling is close to 60000/1800, the ratio of the pumping speeds for water at the BSC chamber under the two conditions.



**Figure 2b** Pumpout of the mid station as measured on a discharge gauge at the entry to the 80K isolation trap. Note the break in the power law at 400 hours. This is not understood.

As you can see from the data there is not a good single value for the power law, which make pre-

dictions for the pump out times uncertain.

The algebraic relations needed to make the outgassing vs time estimates knowing the pressure,  $P$ , at a time  $t_0$  and the pumping speed,  $F$ , at the location where the pressure is measured are given below.

The assumed outgassing relation is

$$J(t) = \frac{\alpha}{t^n} \quad \text{where} \quad \alpha = \frac{P(t_0)Ft_0^n}{A}$$

and  $A$  is the area of the outgassing material. The gas flow at  $t_0$  is  $\dot{Q}(t_0) = P(t_0)F$ . The total amount of water removed,  $Q_r$ , from the outgassing material in a time  $\tau$  becomes (approximately)

$$Q_r = \frac{FP(t_0)\tau^{1-n}t_0^n}{1-n}$$

Finally, the time to attain a desired pressure is given by

$$t = t_0 \left( \frac{P(t_0)}{P(t)} \right)^{\frac{1}{n}}$$

Table 6 gives a summary of the estimates associated with the band of power laws embraced by the data.

**Table 5: Outgassing dynamics as function of power law index**

<i>index</i>	<i>time to exhaust 220 mg/seat</i>	<i>time for 1.0x10<sup>-10</sup> torr</i>	<i>time for 1.0x10<sup>-9</sup> torr</i>	<i>time for 1.0x10<sup>-8</sup> torr</i>
<i>n</i>	<i>days</i>	<i>days</i>	<i>days</i>	<i>days</i>
-0.8	0.15	9.1 x 10 <sup>4</sup>	5.1 x 10 <sup>3</sup>	2.9 x 10 <sup>2</sup>
-0.75	0.7	1.6 x 10 <sup>5</sup>	7.6 x 10 <sup>3</sup>	3.6 x 10 <sup>2</sup>
-0.7	1.9	3.2 x 10 <sup>5</sup>	1.2 x 10 <sup>4</sup>	4.5 x 10 <sup>2</sup>
-0.6	6.9	1.8 x 10 <sup>6</sup>	3.8 x 10 <sup>4</sup>	8.1 x 10 <sup>2</sup>
-0.5	14.8	1.9 x 10 <sup>7</sup>	1.9 x 10 <sup>5</sup>	1.9 x 10 <sup>3</sup>

The time to exhaust 220mg/seat is determined from the integral  $Q_r$  by solving for the time. The outgassing will not stop abruptly at this time but a change in the outgassing power law (most likely to a slower rate) will occur.

The basis of the calculations used in the table is the pump down data from the mid station at  $t_0 = 310$  hours where an outgassing flow of  $\dot{Q} = P(t_0)F = 1.2 \times 10^{-2}$  torr liters/sec for water from the Flourel is measured at 294 K. The pressure of  $1 \times 10^{-10}$  torr or less of water at the entry to the 80K pump would give us 10 years before the goal water pressure is compromised in the tube. A water pressure of  $10^{-9}$  would be needed at the trap in the LVEA for the same condition. Now, having corrected the numerical error I made on Friday, waiting just for the pressure to drop does not look viable for any of the power law models

Heating the Flourel changes the outgassing rate and the estimated times required to achieve a given pressure. The rate limiting step in the Flourel outgassing is the diffusion from the interior of the material. A value of  $T_0 = 7800\text{K}$  is given as the activation temperature for diffusion of water in Viton in *Vacuum Engineering* D.H. Holkeboer, et al. The outgassing rate varies as

$$J(T) = Ae^{\frac{-T_0}{T}}$$

which gives a doubling of the outgassing for every 8 K temperature increase. For example, in going from 70F (21 C) to 120F (49 C) (a temperature increase that might be contemplated for the entire building), the outgassing increases by a factor of about 9.8. Raising the temperature to 80C, increases the outgassing rate by a factor of almost 100 while raising the temperature to 124C would provide a factor of 1000. The times to achieve a particular condition are reduced by the inverse of the increase in outgassing rate.

### *Appendices*

#### **Information on Fluorocarbon Processing and a recommended program for preparing the Flourel seats**

##### **Prior research on cleaning VITON**

During the research on the sticking VITON “O” rings in the large gate valves chemists at Dupont and 3m were contacted. Here is a relevant excerpt from memo “gatevalv.fm”

##### **Recommended pre - processing**

Ron Stevens of Dupont (330-929-6957) recommends more aggressive pre-processing to reduce the Carnuba wax and plasticizers in the VITON. The prescription he suggests is:

- Bake the completed rings in vacuum at 250C for 24 hours
- After cooling to room temperature wash in deionized water to remove surface Fluorides
- Soak rings in Toluene at 70C for 2 days (*This step included primarily to reduce the Carnuba wax used in facilitating the motion of the VITON cord through dies.*)
- Final bake in vacuum at 160C at 10 hours

The choice of bake temperature and duration is determined by the disintegration of the VITON. The first evidence of the disintegration is an increase in the Young’s modulus of the material. Table 6 gives the time to onset of initial disintegration at a range of bake temperatures.

NOTE: We processed the Flourel “O” rings for the gatevalves at 250C for 48 hours in an air flow at 1 torr with no change in Young’s modulus.

**Table 6: VITON disintegration time vs temperature**

<i>Temp C</i>	<i>onset of disintegration hrs</i>
232	3000
260	1000
287	240
315	48
400	flash point

**Walker Fluoride Cleaning Process**

The procedure used at Walker and Sons in New Castle, Oklahoma (405-392-4721) to remove surface fluorides on VITON pieces used in Seagate Disk Drives (George Whiting 405-324-3000 ext 3511)

- 1) Clean pieces with Valtron soap - a Phosphate free soap,
- 2) 4 individual cycles of 15 psi pressure cooking in deionized water,
- 3) rinse in cold deionized water,
- 4) dry in air at 130F for 4 hours.

I have tried to contact G. Whiting (will continue to try) about the procedure and, especially, to find out if there has been research done on step 4) concerning the drying temperature and time in the procedure. Expect that this is far less critical to Seagate than to LIGO since the pieces being cleaned have a thickness of only 3/4 mm and our seats have a thickness close to 1 cm - a factor of about 200 in diffusion time.

**Recommended procedure to determine a good compromise between removing water and fluoride generation on the surface.**

The concept is as we discussed in our telephone meeting on Friday August 20. Take Walker processed seats in pairs weigh them to a precision of 1 mg out of 38 grams before processing and then subject them to a "vacuum" bake in 1mm of flowing air. Follow this by remeasurement of the weight and a test for fluorides  $CF_3$  (amu 69) and  $CF_2$  (amu 50) in vacuum at room temperature with an RGA. Measure the water (amu 18) as well.

Since one would like to know the value of a low temperature bake to remove the water from Hanford LVEA components, these tests should include a low temperature run.

Start with a measurement of the empty RGA instrumented vacuum system and a measurement of a pair of virgin Walkerized seats at amu 18, 50 and 69. To improve the dynamic range of the measurement, reference the ion currents to amu 5 which is not usually populated directly but does give a measure of the RGA spillover between mass numbers.

Concurrently, vacuum bake pairs of Walkerized seats at the extreme temperatures of 50C for 48 hours and 250C for 48 hours . After establishing the weight loss, put the pairs of seats into the RGA instrumented vacuum system. While this is going on process pairs of seats at 80C and 125C in the vacuum oven for 48 hours and measure their weight loss. Based on the results of the RGA measurements of the first runs make a decision on whether it is necessary to also make RGA runs of the newly baked seats.

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