**Water Vapor Outgassing and Readsorption Test Experiment**

**Overview**

The LIGO beamtube modules were originally vacuum baked at 160 C for 1 month to reduce water vapor outgassing from approximately J(H2O) ~ 1x10-11 Torr-liter/s/cm2 as fabricated,  to J(H2O) < 1e-17 Torr-liter/s/cm2 in operation. This final outgassing requirement was overestimated and is at least an order of magnitude better than required, even for Advanced LIGO.  However, gate valve and tube wall failures in the interim will require limited reexposure to atmospheric water vapor, compromising this performance. To be economically feasible, recovery from such interventions may demand a less aggressive and less time-consuming bakeout protocol.

For the original bakeout, water vapor coverage and desorption were successfully modeled using the semi-empirical formalism of Dubinin-Radushkevich (DR) isotherms [LIGO T890001 (Weiss 1996), etc.]. The first goal of this experiment is to test this formalism, and if necessary develop extensions or corrections, in untested intermediate regimes of partial vapor reexposure. Repair scenarios may invoke such limited repopulation, for example by use of short-duration backfills with artificially dry gas. Our second goal for this experiment is to develop and test recovery protocols (e.g., bakeout or other degassing methods) that economically restore the resulting compromised surfaces to an acceptable outgassing rate.

It is expected that these experiments will inform a subsequent scale test, using a sample section of beamtube currently stored at LLO. This will in turn enable repair and/or replacement of leaking LIGO gate valves without excessive expenditures or loss of observing time.

**DR Isotherm Experiment**

**Objective**

This experiment is performed in the molecular flow regime.

The goal is to experimentally determine if the steel used in the construction of LIGO follows the surface coverage equation predicted by the Dubinin-Radushkevich (DR) equation

where

= surface coverage (molecules/area)

= surface coverage (molecules/area) at the saturation vapor pressure P0 at temperature T

P is the pressure

P0 is the saturation vapor pressure at T

T is the temperature

T0 is the adsorption energy (temperature). T0 is also a Gaussian distribution function of adsorption site energies, with a tail skewed towards the high energy part of the distribution.

We will determine the energy distribution function

by monitoring the equilibrium pressure versus temperature behavior for steel samples degassed in a sealed vacuum chamber to which a known volume of water has been reintroduced.

In a (sealed) system the total number of molecules between the adsorption sites and the gas volume is conserved. With uniform energy distribution and surface sites depleted by thermal bakeout, the adsorption site residence time can be simply related to the equilibrium pressure (density). To keep the experiment time reasonable (residence time short) we want to perform the experiment in a regime where b<<1, with b defined as follows

 where

 = gas density (molecules/volume)

= the sticking probability, 0 <

 = the residence time at energy

 = the thermal speed

n is the number of monolayers

 is the surface coverage (molecules/area) at the saturation vapor pressure P0 at temperature T, ~ 3 x 1015 molecules/cm2 per monolayer.

**Procedure**

Sample preparation

The samples shall be made of 1 inch wide 304L stainless steel strip and will receive the following processing.

1. Cut the samples into 20” (inch) lengths. 282 samples minimum. This gives 11,300 inch2 of sample surface area or approximately 10x the chamber surface area of 1100 inch2.
2. Check that deionized (DI) water resistivity is a minimum of 2 MΩ-cm. Handle samples with nitrile or latex gloves.
3. Degrease and the samples using Mirachem 500 undiluted and at room temperature. Residence time is 30 minutes. Rinse with DI water and air dry. See C-CMBS1, "Coil Material Bake Specification", LIGO-[E950023](https://dcc.ligo.org/LIGO-E950023)-x0 modified by [C961636](https://dcc.ligo.org/LIGO-C961636), "CBI Request for Information (RFI) #21: Coil Material Bake Temperature Range" to 444C ± 12C (approved by LIGO Lab in document [C961612](https://dcc.ligo.org/LIGO-C961612)-x0) for additional information.
4. Stack the samples into bundles and tightly wire tie them. This step approximates coiled samples.
5. Air bake the samples at 444 C for 36 hours. Cool to room temperature (RT). Cut tie wires and destack the bundles.
6. Clean samples with Mirachem 500 and cold DI water 50% dilution. Rinse in cold DI water.
7. Rinse samples with 240 F (115 C) water/steam. An alternate process is to rinse in 65 C DI water (65 C was the final temperature of the LIGO beamtube during steam cleaning).
8. Air dry under a laminar flow bench.
9. Rinse several samples in reagent grade isopropanol, air dry.
10. Collect a FTIR sample using reagent grade isopropanol, a dilution of ~0.2 cc/cm2 sample area (50 cc/sample) matches LIGO beamtube FTIR dilution.
11. Compare surface finish, oxide blushing, and cleanliness of the processed strips to legacy LIGO material.
12. Store clean samples in nylon bags.
13. Collect several 1-gram samples for H2 hot extraction measurement. See <https://dcc.ligo.org/T880048> for historical data.

**Test chamber preparation**

The chamber shall be of all-metal construction. Prepare the chamber as follows.

1. Assemble components using UHV techniques; pre-clean flanges and chamber using acetone followed by an isopropanol rinse. Handle all components using gloves. Do not cross contaminate clean and dirty parts. Bottom all UHV (CFF Conflat) flanges until line to line contact is achieved.
2. Evacuate system and leak test using in-situ mass spectrometer. Vent system.
3. Clean, assemble (stack) and tie wire together approximately 20 to 50 strips as a thermal load and place in chamber. This stack will be used to measure the thermal time constant between the load and the external heaters, and to tune the PID loop.
4. Assemble heating mantle and tapes, heat system to 150 C and bake out. Target pressure < 5x10-9 Torr (Bayard-Alpert gauge). Evacuate and heat gas fill system simultaneously. Monitor all thermocouples continuously.
5. Record residual gas spectra at pre, hot and post bake. Water peak should be less than 1x10-9 Torr (system at room temp), peaks at 2 amu (H2) and 28 amu (CO) should dominate the spectrum. Air leaks, hydrocarbons, etc. should not be evident. Monitor pump down time and note valve/pump configuration. Compute H2 and CO outgassing rates using pump speed/conductance values.
6. Test programmed heating and cooling, tune PID loop if required. Profile should be as close to a linear ramp as possible (linear cooldown preferred). Temp range is 500K to RT. Perform post-bake helium leak test using QMS tuned to He peak.
7. Calibrate/crosscheck pumping speed measurement arm with calibrated leak, known conductance.
8. If available, test ultra-pure gas backfill (future experiment) system for water carryover by using QMS. Record dew point if available.
9. If doing water injection experiment: Set up the gas inlet system for water backfill. Evacuate any air in the headspace above the water column using a LN2 freeze-thaw (Schlenk-line) technique, minimum 3 cycles.
10. If doing water injection experiment: Perform water backfill. Monitor mass 18 partial pressure with QMS and calibrate. The backfill is done without the pumps vavled-in. Target pressure is 3x10-7 Torr partial pressure. Valve off QMS and hold with 8 l/s ion pump when completed.
11. Vent system using GN2 backfill.
12. Install previously prepared and cleaned samples in chamber. Place thermocouples at edge and center of steel strips.

 **Experiment procedure**

1. Set up the gas inlet setup for water backfill.
2. Evacuate vacuum chamber and cross over to the 300 liter/sec turbopump at P~100mTorr. Also evacuate the back to air system to the (closed) GN2 regulator valve. The 8 and the 60 liter/sec ion pumps are to be valved-off.
3. At P ~ 1x10-5 Torr open valve to QMS.
4. Monitor pressure reduction of mass 18, to convert to monolayers. Pump speed for water vapor is approximately 250 liter/second. Also monitor Bayard-Alpert gauge (BA gauge sensitivity should be set for N2 ionization, and correct gauge constant).
5. Perform thermal bakeout to 200 C, (500K) continuously monitor mass 18. Want to achieve 0.01 < b < 0.1 for T ~ 1.5x104 K activation energy.
6. Stop pumping (valve off) the system when P < 3.8x10-7 Torr. Sample calculation (Table 1) is for b~.05 and Tact=1.5x104 K. Additional values can be calculated by using spreadsheet “gas law calculates b.xlss”.

**Table 1.** Sample calculation for water vapor valve-off pressure versus activation energy. Accessible value (highlighted) has a residence time of 1 second, b<<1, T=500K. Pressure must be well below 3.8x10-7 Torr before pumping system is valved-off.



1. Introduce “n” moles of water by raising partial pressure (see tabulation from spreadsheet for partial pressure required) of mass 18 as indicated by mass spectrometer.
2. Start programmed temperature ramp to room temp. Continuously monitor equilibrium pressure of mass 18 vs temperature.
3. Calculate Tact from pressure vs temperature data. Assume constant energy with T=Tm. This procedure is outlined in LIGO MEMO 61558.TEX or LIGO T890001. Tabulate the initial pressure (density), and equilibrium pressure (density) after water injection and temperature ramp.

**Equipment List: Consumables**

1. Steel strip per UNS S3043 Finish 2D (anneal and acid pickle), 0.030 (22 gauge) thick x 1” width x 20” length, 350 pcs.
2. Isopropyl alcohol reagent grade (2-propanol) CAS 67-630 >99.5%.
3. Acetone (2-propanone) reagent grade CAS 67-64-1 >99.5%.
4. Wire UNS3043 Finish 2D or BA, 20 gauge.
5. Backfill gas: GN2 UHP 99.99 (backfill only).
6. Test Gas: GN2 Research Grade <20 PPB H20, Air Products.
7. Water, deionized, 2 MΩ-cm.
8. Mirachem 500, Mirachem Chemical, Phoenix AZ.
9. Thermocouple, Type K.
10. Air, compressed, desiccant dry and hydrocarbon-free for furnace atmosphere.
11. Argon calibrated leak, LACO Technologies, Salt Lake City, UT.

**Equipment List: Vacuum System**



