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Bake Oven Requirements and Conceptual Design

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1 ABSTRACT

Requirements and conceptual design are presented for bake/qualification ovens for vacuum preparation areas at the observatories. Comments and corrections are invited.

2 KEYWORDS

bake oven, outgassing, contamination

3 OVERVIEW

LIGO interferometer components that are installed into vacuum chambers have vacuum preparation and certification procedures that will include a vacuum bakeout and RGA certification test, wherever possible ¹. Although a vacuum preparation program has been in effect for some time at the 40-meter lab, LIGO has not yet reviewed and adopted requirements for the LIGO interferometer components to be installed at Hanford and Livingston. This document is intended to address this need. Suggested requirements are discussed in section 4 and a conceptual design of a bake oven for LIGO interferometer components is given in section 5. Appendix 1 describes a conservative limit on allowable hydrocarbon partial pressures in LIGO vacuum chambers, which was used to derive the bake oven requirements and design.

4 REQUIREMENTS

4.1. Vacuum Oven Physical Characteristics

- 1. There must be sufficient diameter to accommodate the large optics suspension (LOS) support structure. This requires a minimum diameter of 22.38 in.
- 2. There must be sufficient height to accommodate the LOS support structure in a vertical orientation. Although it may be possible to bake the LOS in another orientation, the possibility that the LOS must be baked with a suspended optic cannot be ruled out at this time. This requires a minimum height of 30.5 inches.
- 3. Telescope tubes for COS must be baked and qualified. The site bake ovens are one possible way to accomplish this. This would require a minimum height of 60 inches.
- 4. There must be provision to accommodate bake loads that consist of free-standing structures (e.g., LOS support structure), long structures that require support (e.g., optical rails), or many small parts (e.g., fasteners, sensor/actuator assemblies, etc.). This requires provisions for mounting stands and trays inside the oven.
- 5. Oven and associated hardware must accommodate safe handling of heavy monolithic structures, weighing hundreds of pounds.

^{1.} In certain limited cases where vacuum bakeout is exceptionally costly or impractical, an air bake may be substituted, subject to LIGO internal review.

4.2. Vacuum Oven Thermal Capability

- 1. Oven shall accommodate temperatures of 300 C or greater to accommodate bakeout of SS assemblies and to aid with achieving low background gas spectra.
- 2. Oven shall be able to ramp up from room temperature to bake temperature within 12-24 hours.
- 3. Oven shall be able to ramp from bake temperature to room temperature within 12-24 hours.
- 4. Oven temperatures shall be uniform to within 5 C during soak periods.
- 5. Oven temperature controller shall accommodate an intermediate soak states on ramp down. These soak states will be used to accommodate possible outgassing and equilibration measurements during the ramp-down cycle.

4.3. Vacuum System Requirements

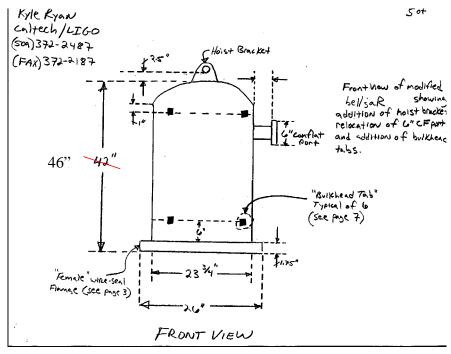
- 1. The hardware shall support a best effort to ensure that the vapor pressure of hydrocarbons in the vicinity of LIGO test masses contributed by any bake load is less than $2\times10^{10^{-15}}$ Torr. (See Appendix 1 for details.) This drives many of the subsequent requirements.
- 2. There shall be a residual gas analyzer (RGA) capable of measuring partial pressures of less than 10^{-13} Torr for mass fragments from 1 to 100 amu.
- 3. There shall be provisions for calibrating RGA measurements in-situ, using calibrated leak(s) as necessary.
- 4. To ensure the lowest possible background contamination of the RGA, the vacuum plumbing shall be configured so that the RGA and calibration manifold are isolated, separately pumped and separately bakeable from the main oven chamber during oven bake-out cycles.
- 5. There shall be no elastomers (except for possible specimens) exposed to the RGA during outgassing measurements. This requires all-metal valves and wire or conflat seals be used in the vacuum environment of the RGA during outgassing measurements. The foreline volumes of turbopumps shall be considered as a separate environment from the volumes on the UHV sides of the turbopumps.
- 6. Roughing and foreline pumping shall be accomplished by oil-free mechanical pumps.
- 7. A pump speed of approximately 10 liters/s shall be provided on the oven chamber during RGA measurements of the outgassing of a baked specimen. This pump speed shall be known to an accuracy of 30% and shall vary by less than 10% among measurements.
- 8. To provide for efficient removal of outgassing products and the lowest pressure at the end of the bake out, the main outlet flange shall allow pump speeds in excess of 100 liters/s.
- 9. The vacuum-system configuration shall allow for simple loading and unloading of bake loads, consistent with practices for clean and safe handling of UHV equipment.
- 10. There shall be provisions for computer logging of the RGA spectra for each bake load.

5 DESIGN DESCRIPTION

5.1. Physical Description

The physical configuration of the bake-oven system is comprised of a vertical bell jar with provision for heating, mounted on a base plate, that provides mounting surfaces for specimens and all

routine vacuum plumbing connections. The bell jar shape and physical dimensions are sketched in Figure 1 below. The vertical bell jar is sized to accommodate an LOS suspension to be mounted vertically on top of the largest height adapter or to accommodate the largest segmented baffle. The bell jar is provided with a single, auxiliary 6-in-OD conflat port. This port is not intended for use during production bake oven use, but allows for initial calibration of pump speeds and subsequent special measurements. A lifting hook is provided to allow placement and removal of the bell jar using a jib crane. A 24-in-height space below the bell is reserved for the oven-chamber base plate and the attached vacuum plumbing.



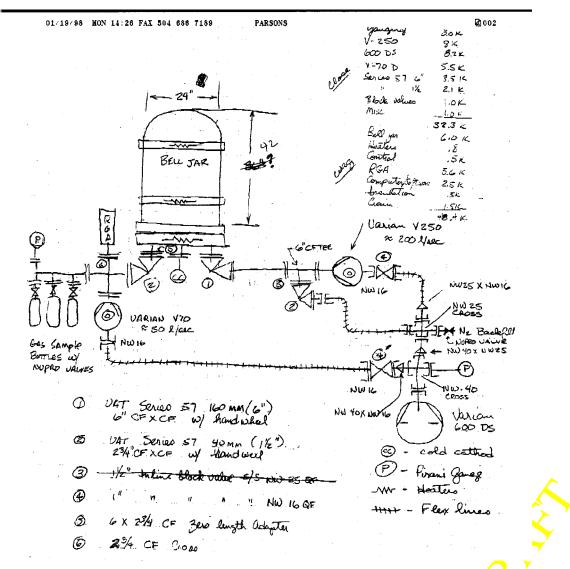
5.2. Vacuum Configuration

The conservatively recommended pass level for bake oven loads $P_{pass} \le 10^{-15}$, described in Appendix 1, is beyond the sensitivity that has been demonstrated within the LIGO project bake ovens and test chambers. The technique that has been used previously is to scan the residual gas with an RGA and to sum the partial pressures in peaks at 41, 43, 53, 55,and 57 amu¹ (hereafter referred to as the Σ mass). A typical RGA with electron multiplier can record partial pressures well below 10^{-13} Torr, but hydrocarbon backgrounds from the RGA itself often limit the useful sensitivity to higher partial pressures. For example, the best background achieved with a large bake oven at Caltech was $P_{\Sigma} = 2 \times 10^{-12}$ Torr. With great care much lower pressures can be

^{1.} This summing procedure, originally proposed by R. Weiss, picks up the main hydrocarbon fragments that are produced by cracking in the RGA ionizer and subsequently fall into the mass range sensed by a typically RGA. Some attempts to use this method are described in: R. Savage, S. Vass, B. Moore, and F. Raab, *Mark II Hydrocarbon Level Evaluation*, Apr 20, 1993; A. Abramovici, *Acceptance Process for Vacuum Systems Containing Optics*, Apr 22, 1993.

achieved as evidenced by the bake out qualification tests done at CBI in Springfield, where hydrocarbon backgrounds below 10^{-14} Torr were achieved after baking at 150 C for one month¹.

In this application, it is desirable to design the bake oven for lowest possible backgrounds compatible with a production schedule for oven use. To minimize hydrocarbon contamination of the RGA, we have chosen to configure the vacuum plumbing so that the RGA is isolated from contamination sources as much as possible. The vacuum configuration is sketched in Figure 2, below.



The RGA is placed in a separately-pumped vacuum volume that can be isolated from ambient air during oven-chamber vents² and is isolated from the specimen during the temperature ramp up and initial soak, when most of the hydrocarbons are emitted from the specimen. There could be

^{1.} R. Weiss, private communication.

^{2.} Experience with the 40-meter interferometer has shown that significant hydrocarbon outgassing results when discharge devices such as ion pumps are exposed to ambient air. (The ion pumps on the 40-m system are net hydrocarbon outgassing sources when operated in the 40-meter interferometer!) It is reasonable that similar results apply to other discharge devices.

concern that, once the RGA volume and bake chamber volumes are reconnected, the two systems will not establish equilibrium on a timely basis. To mitigate this effect, we intend to set the isolation valves to the measurement configuration at a temperature intermediate between the bake soak temperature and room temperature. This temperature will be set by experience, but an initial trial temperature of 60 C is suggested by the data in Appendix 1, Table 2. We intend to monitor the evolution of partial pressures as a function of temperature and time, to establish the sticking rates for the remaining contaminants. In this way it may be possible to (1) achieve lower backgrounds, consistent with the ultimate pressure sensitivity of the RGA and (2) obtain information on the sticking coefficients that could help in defining the ultimate surface density of hydrocarbons on surfaces.

A reasonably large aperture has been used on the main pumping port to provide for lowest possible pressures at the end of the bake soak. After the pressure falls with cooling of the bake chamber and the valves are set to their measurement configuration, the bake chamber and RGA volumes will be pumped by a single pump with pump speed set by conductance limitation to approximately 5-10 liters/s. This range of pump speeds is chosen to dominate the smaller, unreliable pumping speed of the RGA discharge and surrounding manifold, while avoiding unnecessary reduction of the pressures to be measured.

A calibration manifold is provided to enable RGA calibrations in-situ. We envision using a single leak with a mixture of N_2 , Ar and Kr.

A Stanford Model RGA100 Residual Gas Analyzer, with a specified minimum partial pressure of 5×10^{-14} Torr, has been selected for use on the bake apparatus. The planned operational mode, supported by the design of this vacuum system, is to initially energize the RGA at a pressure below 3×10^{-6} and to prevent subsequent exposure of the RGA to higher gas pressures. By preventing the RGA from coming into contact with potentially contaminating gas loads, we hope to achieve consistent instrumental backgrounds close to the specified sensitivity of this RGA model.

APPENDIX 1 ALLOWABLE HYDROCARBON PAR-TIAL PRESSURES IN LIGO VACUUM CHAMBERS

The allowable hydrocarbon partial pressure in LIGO vacuum chambers is estimated here based on estimates of film deposition on optical surfaces and estimates of absorption by these films. The number of monolayers x grown per year, assuming no desorption, is given by

$$x = P/P_{mono} \tag{A1.1}$$

where

$$P_{mono} = \frac{\sigma}{n_0 \langle v_x \rangle t_{yr}} \tag{A1.2}$$

The growth of monolayers of nitrogen provides a well-understood baseline for estimating surface deposition of other materials. The parameters for this case are given in Table 1.

Symbol	Parameter	Value	Units
d_0^2	Mean Molecular Area ^a	3.7	Angstrom ²
σ_{N_2}	Surface Density of Monolayer	7×10 ¹⁸	m^{-2}
n_0	Vapor Density @ 1 Torr	3.5×10 ²²	$m^{-3} \bullet Torr^{-1}$
$\langle v_x \rangle$	Average Normal Speed	120	m/s
P_{mono, N_2}	Pressure for One Monolayer/Yr	5×10 ⁻¹⁴	Torr
t_{yr}	Time in One Year	$\pi \times 10^7$	S

a. John F. O'Hanlon, <u>A User's Guide to Vacuum Technology</u>, <u>2nd edition</u>, Wiley, New York, (1989).

We are primarily concerned here with films of hydrocarbons whose molecular weight is much larger than for nitrogen. The larger molecular weight affects both the speed and the surface density of a monolayer. However the dependence of P_{mono} has a relatively slow dependence on molecular weight M. For a wide variety of models, the relation

$$\left(\frac{28}{M}\right)^{1/2} P_{mono, N_2} < P_{mono, M} < \left(\frac{M}{28}\right)^{1/2} P_{mono, N_2}$$
(A1.3)

holds. The condition to the left of equation (A1.3) can be expected to apply for linear-chain or planar molecules that lay with their long dimension(s) in the plane of the surface, whereas the condition on the left applies for linear-chain molecules that align their long dimension normal to the surface. Thus we have the result that

$$10^{-14} \text{Torr} \le P_{mono, M \le 500} \le 2 \times 10^{-13} \text{Torr}$$
 (A1.4)

for most hydrocarbons of interest.

In the presence of desorption, molecules typically leave the surface after a finite dwell time, τ , given by

$$\tau = \tau_0 \exp\left(\frac{Q}{RT}\right) \tag{A1.5}$$

where Q is the heat of adsorption, R is the gas constant and T is the temperature. Typical values of Q are approximately 10 kcal/mol for water, up to 25 kcal/mol for oils with molecular weights

^{1.} M. H. Hablanian, <u>High-Vacuum Technology</u>, <u>A Practical Guide</u>, Marcel Dekker, Inc., New York, (1990); pg 27.

of order 500 amu. The quantity τ_0 is related to the typical vibrational frequency for molecules absorbed on surfaces, typically ranging from 0.1 to 1 picosecond. Desorption affects surface films by setting the characteristic time for equilibration of surfaces equal to the dwell time. This affects both the ultimate surface density and the rate at which contamination moves inside of a vacuum chamber. Table 2 gives some characteristic dwell time estimates for adsorbed films.

Heat of Adsorption **Temperature** Dwell Time Time Units (kcal/mol) (Centigrade) 25 22 5 weeks 7 25 60 hours 20 22 11 minutes 22 10 25 μs

Table 2. Typical Surface Dwell Times

The fractional number x_M of monolayers covering the surface at equilibrium can be estimated as

$$x_M = \frac{P_M \cdot \tau}{P_{mono, M} \cdot t_{yr}} \tag{A1.6}$$

where P_M is the hydrocarbon vapor pressure. For $P_M = P_{mono, M}$ at room temperature, fractional coverages of $x_M < 0.1$ would be expected using the data of Table 2. The system should also come to equilibrium at this surface coverage in a period of a few months instead of years.

A major uncertainty is the absorptivity of molecules adsorbed in known thicknesses onto mirror surfaces. For the 514.5 nm wavelength, we have a single data point from Rutherford Backscattering Spectroscopy (RBS) of a mirror that was contaminated by a residue of Alconox detergent 1. Here a reflection loss of 3000 ppm was correlated with an RBS imaged carbon residue in the form of a spot that was 30 Angstroms thick. Assuming a linear scaling, this gives a loss of approximately 300-400 ppm/ monolayer. Under optical illumination, strong localized fluorescence was observed from a spot comparable in size to the accumulated carbon spot seen in the RBS scan, indicating there was a strong absorption component to the observed loss. This gives reason to suspect that small fractional surface coverages can cause absorptions in excess of 1 ppm per year. The LIGO requirement on the change in absorption due to contamination is less than 1 ppm/yr. Using a *conservative* absorptivity of 400 ppm/monolayer, estimated from the data above, a reasonable goal would be to aim for $x_M < 0.002$, or a requirement on chamber hydrocarbon pressure

of approximately 2×10^{-16} Torr. It should be noted that the detergent residue which gave rise to this estimate could have had an anomalously high absorptivity² in the green compared to the absorptivity at 1060 nm of typical hydrocarbon traces that might enter LIGO.

^{1.} J. Camp,

^{2.} Detergents such as alconox contain ionic functional groups containing Al, Si, Ca, etc.

If we assume 100 bake-oven loads of equipment are placed inside the LIGO vacuum chambers and the allowable hydrocarbon pressure receives equal contributions from each load, then each load could contribute as much as 2×10^{-18} Torr partial pressure. The pumping speed for condensable molecules for the LIGO test mass chambers typically exceeds 10,000 liters/s whereas the pumping speed at the RGA during bake-oven outgassing measurements can be restricted to 10 liters/s or less. At this pumping speed a conservative pass level for bake-oven loads would be $P_{pass} \le 2 \times 10^{-15}$ Torr.

