Pulse-Purge Vacuum Baking

AUTHOR(S)	DATE	Document Change Notice, Release or Approval
Dennis Coyne	29 Jun 2011	see LIGO DCC record Status

1 Introduction

Each component intended for service within the LIGO Ultra-High Vacuum (UHV) system must comply with the requirements of LIGO-E1000088-v1, "Qualifying Parts for LIGO UHV Service". In particular, a necessary processing step for preparing components is to bake off hydrocarbon contaminants and semi-volatile organics (such as alkanes, siloxanes and organometallics). This step is particularly important for polymers due to their inherent outgassing. This specification defines a vacuum bake process using a pulse-purge technique[1] for accelerating the outgassing and cure of polymer components for use in LIGO's ultra-high vacuum (UHV) systems.

The LIGO specification LIGO-E960022 defines one high vacuum bake process for cleaning components, including polymers, which are intended for in-vacuum service. This specification provides an alternative approach which may be faster and more efficient for baking polymer components.

An appropriate pulse-purge vacuum bake "treatment type" (time and temperature) must be specified for each LIGO part and vacuum bake load. If no treatment type has been specified, contact the cognizant LIGO Laboratory engineer to obtain the appropriate treatment type in writing.

2 Scope

This specification does not cover cleaning of vacuum components prior to vacuum baking. All parts are to be cleaned before vacuum baking in accordance with the appropriate sections of document LIGO-E960022.

Verification that the cleanliness (i.e. the hydrocarbon outgassing rate) meets requirements is required for each vacuum bake load -- not just the process(es) used to clean these parts for UHV service. This specification does not cover verification of the cleanliness of the parts. The method and criteria for qualifying cleanliness via Residual Gas Assay (RGA) testing is defined in LIGO-E080177. The RGA qualification of components which have been pulse-purge, vacuum baked (per this specification) may be performed either as (1) a final step in the pulse-purge process, once the parts have cooled to approximately room temperature (~22C), or (2) as a secondary process in another vacuum oven.

3 Abbreviations and Acronyms

LIGO Laser Interferometer Gravitational-wave Observatory

RGA Residual Gas Analyzer or Assay

UHV Ultra-High Vacuum

[1] The pulse-purge vacuum bake technique was developed by Lawrence Livermore National Labs (LLNL) for use on the National Ignition Facility (NIF) project. The original LLNL specification for this technique is MEL98-006-OK

4 Definitions

4.1 ***Bake-Out Oven***: A clean (free of dust, hydrocarbons and other foreign materials), dry-vacuum oven with the following specifications:

Temperature Range	50C [122°F] to ≥ 200C [392° F]
Uniformity	± 5C [± 9°F]
Control Accuracy	± 5C [± 9°F]
Pressures	A base (empty oven) pressure of ≤ 20 millitorr at 200C
Vacuum Pump	Oil-free ("dry")
Purge Capability	Equipment necessary to provide a ***Pulse Purge***, ***Dry-Gas Venting*** and ***Gas Cooling***.

4.2 ***Clean Area***:
The ***Bake-Out Oven*** shall be located in a clean, distinct area of the facility. Every reasonable effort shall be made to maintain that area to a good level of cleanliness in this area. Traffic in and out of that area shall be held to a minimum. This area must not open directly to the exterior. Air in this ***Clean Area*** should be conditioned for temperature and humidity levels typical of a laboratory or office environment. This ***Clean Area*** (in the immediate vicinity of the bake-out oven) shall be cleaned routinely, in particular before loading and unloading parts from the oven, in order to keep particulate density low. Exposure of LIGO parts within this ***Clean Area*** should be minimized.

4.3 ***Cleaning Materials/Supplies***:
Cleanroom Bag: a low outgassing, cleanroom ISO Class 5 (Fed Std Class 100) compatible bag, approved by LIGO, as listed in document LIGO-E0900047

Cleanroom Aluminum Foil UHV certified clean, annealed aluminum foil, approved by LIGO, as listed in document LIGO-E0900047.

Other LIGO approved supplies for cleaning, including ***Cleanroom Wipers***, ***Isopropyl Alcohol***, ***Acetone***, ***Methanol***, and ***Cleanroom Tape*** are listed in document LIGO-E0900047.

4.4 ***Cleanroom***:
An ISO Class 6 (Fed Std Class 1000), or better, cleanroom with positive pressure, HEPA filtered air ventilation system. Personnel responsible for the handling and packaging of parts in, and maintenance of, the ***Cleanroom*** shall be familiar with guidelines, and appropriate protocols, for clean rooms. The ***Cleanroom*** must have regular (at least weekly) cleaning scheduled while in operation.

4.5 ***Cleanroom Garb***:
Cleanroom Garb is outer wear which is suitable for an ISO Class 6 (Fed Std Class 1000) clean room, including hooded suits (or separate suits and bouffant caps), boots, beard/face masks and ***Cleanroom Gloves***. ***Cleanroom Garb*** must be worn at all times in the ***Cleanroom*** and when using the ***Laminar Flow Bench***. ***Cleanroom Garb*** must also be worn when loading and unloading parts from the ***Bake-Out Oven***. The approved sources for these cleanroom garments are given in document LIGO-E0900047.

4.6 ***Cleanroom Packaging Material***:
Prior to the Pulse-Purge vacuum bake, all cleaned LIGO parts are to be at least double-wrapped using ***Cleanroom Bags***. After the ***Pulse-Purge*** vacuum bake, all LIGO parts are to be wrapped in ***UHV Aluminum Foil*** and then double bagged using ***Cleanroom Bags***. Approved part numbers and sources for the ***Cleanroom Packaging Materials*** are define in document LIGO-E0900047.

4.7 ***Dry-Gas Venting***:
Controlled back-filling of vacuum ***Bake-Out Oven*** with ***High-Purity Inert Gas*** to atmospheric pressure.

4.8 ***Gas Cooling***:
Continuous flow of ***High-Purity Inert Gas*** in the ***Bake-Out Oven*** at the minimum rate of 1 volume change every 5 minutes or a continuation of the ***Pulse Purge*** cycle until the parts cool down to ≤50C [≤122°F].

4.9 ***High-Purity Inert Gas***:
Inert gas such as argon or nitrogen of purity ≥ 99.995% and filtered with a 0.5 micrometer or better filter.

4.10 ***Laminar Flow Bench***:
An ISO Class 6 (Fed Std Class 1000), or better, laminar flow bench, which is located within a ***Clean Area***. Suitable clean room quality protective clothing, including such as a smock, bouffant cap, boots, beard/face mask and gloves must be worn at all times when using the ***Laminar Flow Bench***.

4.11 ***Pulse Purge***:
Controlled flow of ***High-Purity Inert Gas*** into the vacuum ***Bake-Out Oven*** so as to alternately cycle the pressure in the oven between one to five (1 - 5) Torr and ≤20 millitorr. The ***Pulse-Purge*** cycle frequency is ≥ 4 cycles per hour.

5 Exceptions, Deviations, Clarifications

Exceptions, additions or clarifications should be obtained, by the LIGO subsystem Designer or Cognizant Engineer, from Systems Engineering by contacting Dennis Coyne coyne@ligo.caltech.edu or Calum Torrie ctorrie@ligo.caltech.edu.

6 Part Preparation & Handling

6.1 Part Cleaning:
All parts must be cleaned in accordance with document LIGO-E960022 prior to placing into the ***Bake-Out Oven***.

6.2 Part Handling:

Pulse-Purge Vacuum Baking

At all stages, all LIGO parts must be handled with cleanroom gloves, while dressed in the following ***Cleanroom Garb***. If the gloves should touch unclean surfaces, such as the face, clothing, tools, bench, chairs, etc. they must be replaced immediately.

7 Bake-Out Procedure

7.1 Environment: The ***Bake-Out Oven*** and performance of steps ? through ? shall be located in a ***Clean Area*** in accordance with section 4.2.

7.2 Oven Pre-Cleaning: Prior to the oven's use for baking LIGO components, the oven shall be cleaned by wiping oven walls and internal racks which support and/or come into contact with part surfaces (hooks, screens, shelves, etc.) with Acetone saturated Cleanroom Wipers while wearing polyethylene Cleanroom Gloves. This pre-cleaning shall be done initially and after each bake-load/batch.

7.3 Empty Bake: After pre-cleaning, the empty oven shall be baked out at a temperature between 250 and 350C [482-662°F] for ≥8 hours using a ***Pulse Purge*** as defined in section 4.11. This oven cleaning operation is required prior to each use for LIGO parts, but not necessarily before each batch consisting of similar formulations of a given material family, e.g. two similar types of cables.
On a regular basis, determined by the type and number of parts baked or evidence of contamination, the interior surfaces of the piping between the oven and the inlet to the vacuum pump shall be thoroughly cleaned by wiping with Acetone saturated Cleanroom Wipers while wearing polyethylene Cleanroom Gloves. Other cleaning methods, such as ultrasonic cleaning in solvents or vapor degreasing, may be used for cleaning vacuum line plumbing or mechanical oven parts if approved by LIGO prior to implementation, or if in accordance with document LIGO-E960022.

7.4 Loading Parts in the Oven: The parts shall be arranged in the oven to maximize throughput while maintaining free movement of air around the parts. Gaskets, cables and similar flexible parts can be folded and can touch each other but cannot touch the walls of the chamber, or any radiative/heating liners (panels) within the chamber. The maximum number of any given part which can be placed within the chamber shall be determined by the oven operator, consistent with the requirement to achieve the pressures required for the ***Pulse-Purge*** and keeping the ***Bake-Out Oven*** system relatively clean.

7.5 Pulse Purge Bake: The parts shall be pulse-purge by selecting the applicable temperature and time requirements from Table 1. The treatments in Table 1 are not interchangeable; The effectiveness of the conditions depends upon the material being baked. Alternative bake-out procedures (e.g., different bake-out temperatures and/or bake-out durations) may be proposed; however these must be approved or qualified by LIGO.

7.6 Vacuum bake-out Cycle Times shall begin when the oven is at the required temperature and the Pulse Purge cycle frequency is ≥4 cycles per hour. Minimum pressure of Pulse Purge cycles must be ≤50 millitorr at the start of a bake-out Cycle Time and ≤20 millitorr by the completion of the Cycle Time as listed in Table 1.

7.7 The vacuum oven shall be vented to atmospheric pressure by ***Dry-Gas Venting***, as defined in Section 4.7.

7.8 The parts are then to be gas cooled in the Bake-Out Oven to ≤ 50C [≤122°F] as defined in Section 4.8.

8 Post Bake-Out Handling

8.1 Baked parts shall be handled only with Cleanroom Gloves. Gloved hands that touch cleaned parts shall not touch anything else. Soiled or torn gloves shall be replaced with a fresh pair.

8.2 Baked parts shall be allowed to come in contact only with gloved hands, Cleanroom Packaging Material, or Cleanroom Aluminum Foil. ***Cleanroom Packaging Material*** or Cleanroom Aluminum Foil may be used to cover work-surfaces.

8.3 Baked parts may remain in the ***Cleanroom*** or on the ***Laminar Flow Bench*** provided they are covered by ***Cleanroom Packaging Material*** or ***Cleanroom Aluminum Foil***. ***Cleanroom bags*** are to be closed by zippers or ties.

8.4 Final packaging and labeling shall be done in accordance with document LIGO-E960022, in the ***Cleanroom*** or on the ***Laminar Flow Bench***. Do not re-use the packaging used for the parts before vacuum baking. Specifically (from section 8 of E960022-v16):
(a) Wrap the part(s) with UHV quality ***Cleanroom Aluminum Foil***.

(b) Place each part(s) in an approved ***Cleanroom Bag***.

(c) Compress the bag tightly around the part(s) to purge excess air. Tie wrap the bag for closure, or use a bag with a zipper. Do not heat seal or tape this inner bag.

(d) Two labels must be used on the outer layer of all bagged components: (i) a warning label stating: "UHV CLEAN PART -- HANDLE ONLY WITH PROPERLY GLOVED HANDS" and (ii) an identification label. If the labels are not self-adhesive, then they shall be affixed with tape. All empty fields on the ID label shall be filled in with the relevant information; use “N/A” rather than leaving a field blank.

(e) Place the part(s) in a second approved ***Cleanroom Bag***, remove excess air, and seal with a zipper, heat seal or tape.

(f) Place the double bagged part(s) in an appropriate shipping container, using care to not puncture or cut the bags. Take care in the packaging to ensure that the part(s) are not damaged in shipping.

Pulse-Purge Vacuum Baking

Table 1: Pulse-Purge Treatment

Treatment Type	Temperature	Cycle Time (hrs at temperature)	Example Applications
LA	100C ± 5C [212 ± 9°F]	48	• ADE capacitive position sensor probe assembly with cable
LB	120C ± 5C [248 ± 9°F]	48	• BOSEM assemblies
LC	145C ± 5C [293 ± 9°F]	96	• AOSEM assemblies
LD	182 ± 5C [360 ± 9°F]	48	• Fluoroelastomers (Viton™, Fluorel™, etc.)
LE	200 ± 5C [392 ± 9°F]	48	• Cable assemblies (PEEK connectors, Kapton or PFA insulation, braided copper shield, overall PEEK fiber braid) • PEEK components • PFA grade Teflon components

9 References

1. LIGO-E960022 LIGO Vacuum Compatibility, Cleaning Methods and Qualification Procedures
2. FED-STD-209E Airborne Particulate Cleanliness Classes in Cleanrooms and Cleanzones
3. ISO 14644-1 Classification of air cleanliness *(the ISO standard which replaces FED-STD-209E)*
4. LIGO-E0900047 LIGO Contamination Control Plan
5. IEST-CC1246D Product Cleanliness Levels and Contamination Control Program (replaces MIL-STD-1246C)
6. LIGO-E080177 Specification: RGA Test Qualification
7. MEL98-006-OK LLNL Specification: High Temperature Bake-Out to Remove Non-Volatile Residue for NIF *(used as a basis/guide for this specification)*