FTIR Testing to Qualify Parts for LIGO UHV Service

AUTHOR(S)	DATE	Document Change Notice, Release or Approval
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1 Introduction

All components intended for service within the LIGO Ultra-High Vacuum (UHV) system shall be tested to confirm that the surface cleanliness meets requirements set forth in Reference 1. In particular the Non-Volatile Residue (NVR) for organics shall be measured to meet requirements by either Mass Spectrometry (also known as Residual Gas Assay (RGA)) or by Fourier Transform InfraRed (FTIR) analysis. RGA testing is the preferred method, since it directly measures the outgassing from the component. However, components which are to large to fit into an UHV bake oven must be air baked and tested with FTIR. FTIR provides chemical functional group information for quantitative analysis and qualitative identification of contaminants. This specification defines the requirements for FTIR testing to meet LIGO UHV requirements.

2 Scope

This specification covers FTIR testing for NVR contamination for all parts intended for the LIGO UHV system.

Components must be comprised of approved materials (as defined in Reference 3) before undergoing FTIR qualification.

If the part is not in the vacuum system, then this specification does not apply. If NVR cleanliness is to be determined by RGA, then this specification is not applicable (see Reference 5)

3 Abbreviations and Acronyms

AHC Aliphatic hydrocarbon

FTIR Fourier Transform Infrared Transmission

HPCL high-performance liquid chromatography

OAS Organic Acid Salt

RGA Residual Gas Analyzer

UHV Ultra-High Vacuum

4 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.

5 Surface NVR Cleanliness Requirement

Air baking is mentioned in document E960022-B (section 6.1.2.1.2) as an alternative to vacuum baking for large parts. NVR surface cleanliness must be verified by an FTIR test. It is preferable to perform the FTIR test prior to an air bake (so that the FTIR results are obtained while, or shortly after, the air bake has been completed). The acceptance criteria for pre-bake FTIR results are NVR Level A/20 (or ≤ 0.05 micrograms/cm^2) per MIL-STD-1246C or IEST-STD-CC1246D. For threaded holes the maximum acceptable NVR level is ≤ 1.2 microgram/hole for through holes and ≤ 2.0 microgram/hole for blind holes, both prior to the air bake.

If an FTIR is conducted after an air bake, the surface should be at Level A/50 or better (per section 6.1 of Reference 1, version v1) or ≤ 0.02 micrograms/cm^2 and ≤ 0.4 microgram/hole for through holes and ≤ 0.7 microgram/hole for blind holes. It is not necessary to perform the FTIR sampling both before and after an air bake.

5.1 When to FTIR?

As above, it is preferable to perform the FTIR test prior to an air bake (so that the FTIR results are obtained while, or shortly after, the air bake has been completed). Parts should be held within Vacuum team until FTIR is completed, approved and sign off on the DCC. It is not necessary to perform the FTIR sampling both before and after an air bake

If FTIR is taken off-site from LIGO then the parts should be held at the off-site location (e.g. vendor) until the FTIR is completed, approved and sign off on the DCC. As long as it is confirmed on arrival at a LIGO site (during incoming inspection) that the packaging is intact (then again) it is not necessary to perform the FTIR sampling both before and after an air bake. If there are any concerns about the packaging on arrival (or other issues) the local vacuum lead can request a further sample set to be collected in line with the sampling steps below.

5.2 Surface Sampling

The cleanliness of the part surfaces is established by sampling the surface with a high purity solvent and collecting the solvent with clean tools and sampling containers. The solvent should be effective at removing aliphatic hydrocarbons (oils, greases), plasticizers and esters (fingerprints, adhesives, etc.), silicones (lubricants, sealants, adhesives) and organic acid salts (soap/cleaner residue). Potential solvents include HPCL 2-isopropanol, Freon-TF, Hexanes or dichloromethane.

Surface sampling can be accomplished by either (a) wiping the surface with a solvent soaked fiber-free lens tissue (which has been cleaned by chemical extraction; see Reference 8) or (b) by pouring the solvent over the surface and collecting the fluid as it runs or drips off the part.

The surfaces of each part must be sampled such that > 5% of the area and > 5% of the holes are sampled. Multiple holes can be sampled in a single swipe or fluid volume. Likewise multiple surface patches can be sampled in a single swipe or fluid volume. However hole samples and area samples should ideally not be combined in a single sample; The effective area of a threaded hole is more

difficult to estimate and holes (threaded in particular) tend to be the most problematic to clean. The amount of surface wetted by the solvent in a single sample is limited by the amount of solvent and the need to insure that the solvent is collected before evaporating.

The sample, or a measured volume of the collected solvent sample, is then evaporated onto an infrared transparent slide, or window, for insertion into the FTIR (Michelson interferometer) instrument. The slide is generally a Sodium Chloride (NaCl) or Potassium Bromide (KBr) crystal slide, but other materials may also be appropriate.

5.3 FTIR Testing Data Requirements

We need a quantitative FTIR measurement of the NVR in order to verify that the amount of residue meets the requirement. The results ideally should be expressed in micrograms/cm^2 and micrograms/hole. One approach is to use Diffuse Reflectance/ Fourier Transform Infrared (DRIFT/FTIR) spectroscopy.

An absorption spectra for the sample should be provided that ranges from ~400 to ~4000 1/cm wavenumber. In addition to the absorption spectra, a calculation of the "specific absorption", z, should be made at specific wavenumbers of interest (see table below), which takes into account the absorption from a reference sample of the solvent and is normalized by the sample area and volume:

$$z = \frac{\left(\ln(I/I_0)_{sample} - \ln(I/I_0)_{reference}\right)A_w V_s}{A_s V_e}$$

Where

I = FTIR output at wavenumber n with the sample or reference

I_0 = FTIR output at wavenumber n without a sample or reference (background)

A_w = area of the FTIR window (KBr or other)

A_s = sampled surface area of the part (or number of holes sampled)

V_e = volume of the solvent evaporated onto the FTIR window (KBr or other)

V_s = total volume of the solvent sample

The wavenumbers of interest to LIGO are indicated in the following table. The dominant peak in our experience is CH3 at 2950 1/cm.

Wavenumber (1/cm)	Mechanism	designation
3420	O-H stretch	OH3
2950	C-H stretch (hydrocarbons)	CH3
2850	C-H stretch	CH2
1730	C-O, H-C-H stretch (esters)	CO1

1480	C-H stretch	CH1
1390	CH2 and CH3 bend	CHB
1280	C-O twist	COT
1146	silcones	SIL
1090	C-O stretch	COS

Identification of candidate contaminants by matching the spectra to a library of FTIR spectra is of interest in order to help in re-cleaning the part (if needed) or to potentially reduce the contamination level of future parts.

6 References

1. LIGO-E010613 Generic Requirements & Standards for Detector Subsystems
2. LIGO-E960022 LIGO Vacuum Compatibility, Cleaning Methods and Qualification Procedures
3. LIGO-E960050 LIGO Vacuum Compatible Materials List
4. IEST-CC1246D Product Cleanliness Levels and Contamination Control Program
5. LIGO-E080177 Specification: RGA Test Qualification
6. LIGO-E0900479 Instructions for taking Low Volatility Residue (LVR) Wipe Samples
7. LIGO-T0900523 Beam Tube Cleaning in Pasco (describes FTIR testing of the LIGO Beam Tubes)
8. J.J. Herrick, et. al., "Analysis of Semi-Volatile Residues Using Diffuse Reflectance Infrared Fourier Transform Spectroscopy" in Optical System Contamination: Effects, Measurements, and Control VII; July 2002, edited by Phillip T. C. Chen and O. Manuel Lee; Proceedings of the SPIE, Vol. 4774, pp. 251-261, (2002).