

JPL ANALYTICAL CHEMISTRY LABORATORY

Analytical Chemistry and Materials Development Group 3531

Propulsion and Materials Section 3530

To: Jodi Fauver

12-08-11

From: Jerami Mennella

Subject: 1101134, HAM 11 Chamber Cleaning FTIR Samples (Box 1)

Purpose

The purpose is to determine the level and identity of molecular (oily) contamination.

Method

LVR Analysis

The low volatility residue (LVR) was analyzed using Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectroscopy (1). Blank samples were run along with the samples (2). FTIR provides chemical functional group information for quantitative analysis and qualitative identification of materials. The analysis followed the JPL ACL-120 procedure that complies with IEST-STD-CC1246D (3) and is sensitive to stringent levels (4).

Results

PARTS				SAMPLES				LVR Analysis Results	
#	Part No.	SN	Description	#	Type	Description	Sampled Area	Chemical Functional Group	Total Amount (µg / cm ²)
1	A		PRE-WORK SAMPLE TAKEN 11/15/11 ON C1	1	Surface	Chamber wall	1 sq ft	AHC	< 0.02
2	B		EMPTY BOTTLE CAP WAS NOT ON TIGHT ENOUGH – SAMPLE NOT USED	2	Surface	Chamber wall	1 sq ft	N/A	N/A
3	C		PRE-WORK SAMPLE TAKEN 11/15/11 ON SOUTH ENTRY FLOOR	3	Surface	Chamber wall	1 sq ft	Ester, AHC	0.06
4	D		PRE-WORK SAMPLE TAKEN 11/15/11 ON CENTER OF FLOOR	4	Surface	Chamber wall	1 sq ft	Ester, AHC	0.3
5	E		PRE-WORK SAMPLE TAKEN 11/15/11 ON NORTH ENTRY FLOOR	5	Surface	Chamber wall	1 sq ft	Ester, AHC	0.07
6	F		PRE-WORK SAMPLE TAKEN 11/15/11 ON UN 3 & 4	6	Surface	Chamber wall	1 sq ft	Ester, AHC	< 0.02
7	G		PRE-WORK SAMPLE TAKEN 11/15/11 ON M9	7	Surface	Chamber wall	1 sq ft	AHC	< 0.02
8	H		POST-WORK SAMPLE TAKEN 11/30/11 ON C1	8	Surface	Chamber wall	1 sq ft	AHC	< 0.02
9	I		POST-WORK SAMPLE TAKEN 11/30/11 ON SOUTH ENTRY FLOOR	9	Surface	Chamber wall	1 sq ft	AHC	< 0.02
10	J		POST-WORK SAMPLE TAKEN 11/30/11 ON CENTER OF FLOOR F1	10	Surface	Chamber wall	1 sq ft	Ester, AHC	< 0.02

Terminology:

AHC: Aliphatic hydrocarbon, base oil of common lubricants
 Esters: common sources are from plasticizers and fingerprints
 µg/cm²: micrograms per square centime

References and Notes

1. M. S. Anderson 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).

2. The wipe blanks are less than 10% the amount in the sample and this is subtracted from the reported sample amount. High blanks (greater than 10% of the sample) are noted in the report. A typical solvent wipe has a detection limit of $\sim 0.005 \mu\text{g}/\text{cm}^2$ of removed residue from a 100cm^2 sample. Note this limit is well below the adventitious carbon level (ref. 4). Lower limits are possible using modified methods.

3. The analysis conforms to the Institute of Environmental Science and Technology (IEST), Contamination Control Division Document IEST 1246D "Product Cleanliness Levels and Contamination Control Program". The contamination limits are generally set by Contamination Control Engineering. At typical limit is "Level A" (IEST-STD-CC1246D) and this is 1 microgram per square centimeter ($\mu\text{g}/\text{cm}^2$) and this corresponds to an average film thickness of 100 angstroms (assuming a density of 1.0). In many cases more stringent limits apply (4).

4. Very clean surfaces, $\leq 0.02 \mu\text{g}/\text{cm}^2$, with mono-molecular layers or less are more complex to describe when cleaning or analyzing. Carbon/hydrocarbon based substances are known to rapidly (within ~ 1 hour) accumulate on most, if not all, freshly exposed surfaces. This "adventitious" carbon is well documented in clean rooms and vacuum systems and compositionally varies by environment. Adventitious carbon is a discontinuous layer of approximately ~ 0.2 nanometers thick or $\sim 0.02 \mu\text{g}/\text{cm}^2$ up to $0.1 \mu\text{g}/\text{cm}^2$ (for $\rho = 1$). The last mono-layer fractions may in some cases be strongly adsorbed to the surface as a "corrosion" layer. Therefore solvent based sampling methods may not remove these fractions, particularly if the surface is porous. When specifying cleanliness level to less than A/10 IEST-STD-CC1246D ($0.1 \mu\text{g}/\text{cm}^2$) these monolayer effects become more significant. See also: H. Piao and N. S. McIntyre, "Adventitious carbon growth on aluminum and gold-aluminum alloy surfaces", *Surface and Interface Analysis*, 2002; 33: 591–594.