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ORGANIZATIONAL WORK INSTRUCTION

EM50

Detection of Silicone in Processing Materials Using FT-IR Spectroscopy

Revision C

APPROVING AUTHORITY	NAME	TITLE	ORG	DATE
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**CHECK THE MASTER LIST—
VERIFY THAT THIS IS THE CORRECT VERSION BEFORE USE**

DOCUMENT HISTORY LOG

Status (Baseline/ Revision/ Canceled)	Document Revision	Effective Date	Description
Baseline		03-05-02	Baselined OWI
Revision	A	02-09-04	Deleted incorrect sentence.
Revision	B	08-25-04	Revised per HQ Rules Revision Action
Revision	C	04-27-05	Changes made due to reorganization

DETECTION OF SILICONE IN PROCESSING MATERIALS USING FT-IR SPCTROSCOPY

1. SCOPE

1.1 Scope

This document establishes the organizational work instruction for using FT-IR spectroscopy as a means of detecting silicone in processing materials.

1.2 Purpose

The purpose of this document is to outline the steps necessary for performing FT-IR analysis to determine whether processing materials contain solvent-extractable silicone or transferable silicone.

1.3 Applicability

This document applies to the analysis techniques used by EM50. This work instruction is not a substitute for formal training. Untrained personnel shall not attempt to use this document to analyze materials unless supervised by trained, experienced personnel.

2. APPLICABLE DOCUMENTS

MPD 1280.1	Marshall Management Manual
EM50-OWI-002	Document and Data Control
EM50-OWI-003	Control of Quality Records
EM50-OWI-004	Control of Customer Supplied Product
MPR 8730.5	Control of Inspecting, Measuring, and Test Equipment

3. DEFINITIONS

Warning - Warnings are used when failure to observe instructions or precautions could result in injury to personnel.

Caution - Cautions are used when failure to observe instructions could result in damage to equipment.

Note - Information to help clarify multi-person procedures or simultaneous multiple operations.

4. INSTRUCTIONS

Work performed and data generated within EM50 that affects the quality of products as specified in the scope of MPD 1280.1 shall be documented and controlled per EM50-OWI-002.

4.1 FT-IR Requirements & Analysis Accessories

The FT-IR requirements include adjustable gain setting, adjustable scan number, and spectral resolution capability to 4 cm^{-1}. The light source shall operate in the mid-infrared region of the spectrum. The beam splitter shall be made from KBr, and the detector shall be of the type MCT/A. An out-of-compartment horizontal attenuated total reflectance (HATR) sampler is required, along with 45-degree ZnSe HATR crystals with flat and trough configurations. Sealed-cell accessories made from NaCl or ZnSe are also required.

4.2 Analysis Parameters

Initial analysis parameters shall be a gain setting of “Autogain”, a resolution setting of 4 cm^{-1}, 32 scans for the reference spectrum and 32 scans for the sample spectrum.

4.3 Test Method Selection

Perform a transference test using the HATR crystal to evaluate materials for silicone residing on a surface. Contact the sample with the HATR crystal, remove the sample from the crystal, and then analyze the crystal for silicone that has been transferred from the sample. Use this test for soft, pliable materials such as rubbers, plastics, and woven and non-woven fibers.

The extraction technique is used for materials that will be exposed to solvent during processing operations. Immerse the material in solvent, collect the solvent, and then analyze the solvent/residue mixture or the non-volatile residue for silicone. The technique is equally useful for rigid and pliable materials.

One advantage of the extraction test relative to the transference test is that the extraction method also collects silicone from within the material matrix. An HATR crystal is used to analyze the neat residue or the solvent/residue mixture, and a sealed-cell is used for analysis of solvated residue.

4.4 Pre-Test Preparation for the HATR Crystal

Ensure that the HATR crystal is clean, and then obtain a reference spectrum.

4.4.1 Rinse both sides of the crystal with chloroform or acetone or hexane, and then allow the crystal to dry. Facilitate drying of the crystal by wiping with a clean cotton ball or Rymple cloth.

4.4.2 Collect a reference spectrum of the crystal using the selected analysis parameters (Section 4.2).

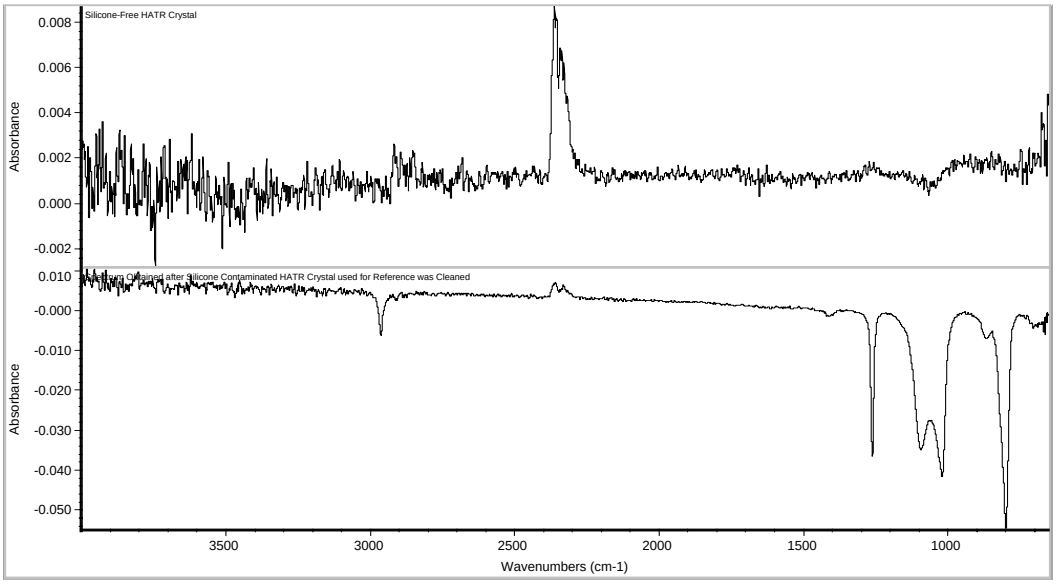
4.4.3 Repeat cleaning of the crystal per Section 4.4.1.

4.4.4 Collect an absorbance spectrum of the crystal using the selected analysis parameters (Section 4.2).

4.4.5 Analyze the spectrum for negative absorbencies, which indicate that contamination was removed from the crystal by the cleaning operation performed in step 4.4.3.

4.4.5.1 If negative absorbencies are observed, obtain a new reference spectrum of the crystal, perform the cleaning operation described in step 4.4.1, and then obtain a new spectrum of the crystal. Repeat this process until negative absorbencies are no longer observed. When no negative absorbencies are observed, proceed to step 4.4.6.

Figure I: Lower Spectrum Illustrates Negative Absorbencies Observed when Silicone Contamination is Removed from HATR Crystal by Cleaning Operation. Upper Spectrum Illustrates Clean HATR Crystal (Absorbance at 2400 cm^{-1} is CO_2).



4.4.6 Once it has been verified as clean, obtain a final reference spectrum of the crystal. This reference spectrum will be used against the sample being analyzed for the presence of silicone.

4.5 Pre-Test Preparation for the Sealed-Cell

Ensure that the sealed-cell is clean, and then obtain a reference spectrum.

4.5.1 Rinse the interior of the sealed-cell using acetone or chloroform or hexane, and then rinse the interior a minimum of three times using the solvent selected for FT-IR analysis.

Note – The solvent used for extracting the test sample need not be the solvent used for IR analysis. One solvent can be better for extracting silicone, while another can offer less potential for interfering with silicone detection.

4.5.2 Collect a reference spectrum of the FT-IR solvent using the selected analysis parameters (Section 4.2).

4.6 Sample Analysis by Transference Method to HATR Crystal

4.6.1 Identify the sample to be analyzed. If it is a customer-supplied sample, it shall be logged per document EM50-OWI-004.

4.6.2 Handle the sample carefully, taking care not to touch the surface that will be analyzed.

4.6.3 Clean the HATR crystal and obtain a reference spectrum, per Section 4.4.

4.6.4 Contact the sample surface to the HATR crystal (flat crystal recommended).

4.6.5 Maintain contact with the HATR crystal for 5-10 seconds, applying moderate pressure.

4.6.6 Remove the sample from the crystal.

4.6.7 Collect the spectrum of the residue using the analysis parameters employed for the reference spectrum.

4.6.8 Examine the sample spectrum for silicone absorbencies per Section 4.8.

4.7 Sample Analysis by Extraction Method

Solvent(s) used with the material during processing operations shall be used to conduct the extraction. If the processing material is not exposed to solvent during use, perform the extraction using hexane or chloroform.

Prior to beginning analysis of the test sample, a control test shall be performed. The control test shall involve mimicking the extraction and analysis procedure using all glassware, solvents, and other handling items but excluding the test sample. There are two principle reasons for performing this test. First, solvents often contain residue that interfere with the detection of silicone. Second, this will serve as a control test to ensure that the solvents and test process are not providing a positive silicone signature.

4.7.1 Identify the sample to be analyzed. If it is a customer-supplied sample, it shall be logged per document EM50-OWI-004.

4.7.2 Handle the sample carefully, taking care not to inadvertently contaminate it due to use of unclean utensils.

4.7.3 Immerse the sample in the extraction solvent for 15-30 minutes. Use a 1:15 ratio of sample to solvent, by

weight. Samples that are large shall be cut into smaller pieces that are more easily handled and require less solvent.

Note – The 15-30 minute immersion time is a guideline for use when actual exposure time to solvent is unknown. Increase or decrease the immersion time to simulate processing conditions when the exposure time is known.

4.7.4 Remove the test sample from the solvent.

4.7.5 For HATR analysis, prepare the trough crystal per Section 4.4 and then transfer the solvent/residue mixture to the crystal. Analyze the solvent/residue mixture or allow the solvent to evaporate and then analyze the non-volatile residue.

4.7.6 For sealed-cell analysis, prepare the cell per Section 4.5 and then transfer the solvent/residue mixture to the sample holder using a pipette. Or, if the solvent used for extraction is not the one that will be used for FT-IR analysis, allow the extraction solvent to evaporate, then dissolve the residue in the FT-IR solvent and transfer this mixture to the sealed-cell.

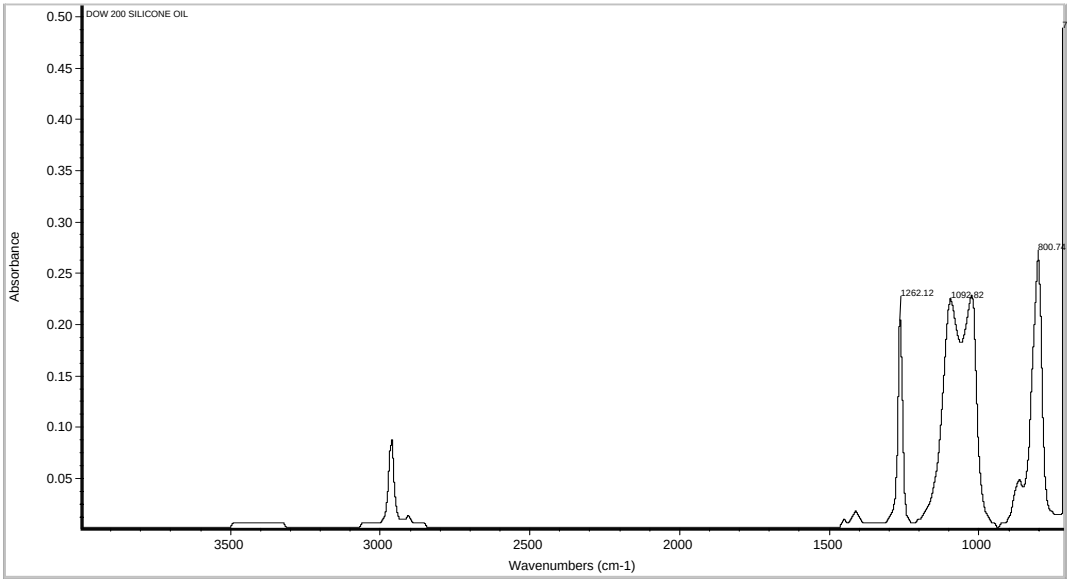
4.7.7 Collect the spectrum of the residue or solvent/residue mixture using the parameters employed for the reference spectrum.

4.7.8 Examine the sample spectrum for silicone absorbencies per Section 4.8.

4.8 Spectrum Analysis

Perform a visual analysis of the sample spectrum, looking for absorbencies located approximately at 1260 cm^{-1}, 1090 cm^{-1}, 1020 cm^{-1} and 800 cm^{-1}. If these collective absorbencies are not present, report the results as silicone not detected relative to the baseline instrument noise. Absorbency heights are considered to be above instrument noise if they measure a minimum of 2 times the baseline noise in the regions to the immediate left and right of the absorbance.

Figure II: FT-IR Spectrum of Typical Silicone Oil



5. NOTES

None.

6. SAFETY PRECAUTIONS AND WARNING NOTES

This work instruction is not a substitute for formal training.

Appropriate personnel protective equipment (PPE) shall be used when performing the procedures described in this document. Minimal PPE includes safety glasses, lab smock, and solvent resistant gloves. Solvents shall be handled in areas that are adequately ventilated. Use vapor masks to minimize exposure to solvent vapors in poorly ventilated areas.

7. APPENDICES, DATA, REPORTS AND FORMS

When evaluating materials used in the processing of flight hardware or in other critical operations, a memorandum containing the information listed below shall be provided to the requestor. This data shall be presented to Quality personnel for stamping. MSFC Quality may choose whether or not to be present for the sample analysis. The following information shall be provided in the memorandum:

1) Material identity

2) Material source
3) Material lot number, part number, and as-received description (packaging, condition, etc)
4) Analysis procedure used (extraction versus transference method)
   a. For extraction testing, list solvent(s) used and extraction time
5) Instrument analysis parameters including scan number, gain setting and resolution setting
6) Analysis results (silicone detected/not detected)
7) A statement of any other relevant information which might be deemed important

8. RECORDS

Records shall be retained in accordance with Document EM50-OWI-003. Copies shall be maintained in EM50 for a period of not less than 2 years.

9. TOOLS, EQUIPMENT, MATERIALS

As required.

10. PERSONNEL TRAINING AND CERTIFICATION

The leader of the Environmental Effects Group shall be responsible for ensuring that all personnel performing these techniques are trained. Training is accomplished as on-the-job training by certified personnel. Records of personnel training shall be kept in the group office.