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George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

PROCEDURE

THE SAMPLING AND ANALYSIS OF NONVOLATILE RESIDUE CONTENT ON CRITICAL SURFACES

GEORGE C. MARSHALL SPACE FLIGHT CENTER
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GEORGE C. MARSHALL SPACE FLIGHT CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

PROCEDURE

THE SAMPLING AND ANALYSIS OF NONVOLATILE RESIDUE CONTENT OF CRITICAL SURFACES

1. PURPOSE

The purpose of this procedure is to establish a standard method for sampling and analysis of nonvolatile residue content on critical surfaces.

2. SCOPE

This procedure specifies the method by which critical surfaces shall be sampled and analyzed to quantitatively determine nonvolatile residue content. This procedure contains two methods, *"swab"* and *"flush"*.

3. DEFINITION

Nonvolatile residue (NVR) is the material remaining after filterization and temperature controlled evaporation of a volatile liquid (usually measured in milligrams per unit volume).

4. APPLICABLE DOCUMENTS

MSFC-STD-246 -- Design and operation Criteria of Controlled Environment Areas.
MSFC-PROC-1831 -- The Analysis of Nonvolatile Residue.
MIL-STD-81302B -- Cleaning Compound, solvent, Trichlorotrifluoroethane.

5. ABBREVIATIONS

ml	milliliter
mg	milligram
ft	feet
ft2	foot/square
C	centigrade

NVR	nonvolatile residue
µm	micrometer

6. SUPPLY REQUIREMENTS

A. *Equipment Required*

Item	Description
Oven	Stainless steel lined, with >50° C capability
Analytical Balance	Capable of weighing in grams to four decimal places
Tongs	Laboratory
Flash Evaporator	Buchler, Model FE-2C (or equal)
Vacuum Source	Capable of pulling 25 inches of mercury
Vacuum Funnel	Millipore
SOXHLET Extractor Apparatus	minimum of 200 ml.
Teflon Wash Bottle	500 ml.

B. *Materials Required*

Item	Description
Weighing Dish	Disposable Aluminum weighing pan with tabs
Wipes	Kim Wipes (or equal)
Desiccator (2)	Glass
Filter Membrane	0.45 µm
Beaker	1,000 ml glass
Aluminum Foil	Reynolds (or equal)
Joy (liquid)	Washing detergent
LOC	Liquid Organic Cleaner (AMWAY)

Gloves	Teflon or polyethylene
Aluminum foil	Cleaned
Glass beaker	1000 ml.

C. *Chemical Solvents Required*

Item	Description
Ethyl Alcohol	Dehydrated 200 proof
Acetone	Reagent Grade (or equal), Nonvolatile residue content shall not exceed 02.00 mgs. per 500 mls.
Freon	Precision Trichlorotrifluoroethane per MIL-C-STD 81302 B

7. GENERAL REQUIREMENTS

A. Perform nonvolatile residue analysis in a clean room meeting requirements of a class 100K or better area as defined in MSFC-STD-246. The use of adequate ventilation is necessary during swab sampling in confined areas.

B. The evaporating process shall be performed in fume hood unit equipped with exhaust blower capable of a minimum face velocity of 100 feet per minute. The exhaust blower must be in operation continually during the evaporation process.

C. Rinse laboratory tongs with solvent prior to each use.

D. Samples to be analyzed for NVR shall be filtered via 0.45µm millipore filter.

E. Vacuum source shall be adequately trapped to prevent back streaming.

F. Swabs shall be cleaned and blanked prior to each swab test per para.8.3.

G. The Teflon wash bottles used in the flush sampling shall be cleaned and baselined before use.

8. EQUIPMENT PREPARATION

A. *SWAB METHOD*

1. Initial cleaning as follows:

   a. All trichlorotrifluoroethane (Freon 113) used in this procedure shall be analyzed for NVR by evaporating 500 ml and analyzing in accordance with

MSFC-PROC-1831. The NVR shall not be higher than the MIL-STD 81302B limit.

b. Wash all swabs with hot soapy (Joy or LOC) water until clean. Rinse with distilled water.

c. Wash all equipment surfaces, which will contact samples, with hot soapy (Joy or LOC) water. Rinse well with distilled water.

d. Rinse with clean acetone - dry with Nitrogen (GN_2)

e. Rinse with trichlorotrifluoroethane (Freon 113) and dry with Nitrogen (GN_2).

2. Preparation of Swab

NOTE

The swab material may vary with each job. Care shall be taken in selecting swabs to assure compatibility with solvent to be used. These swabs shall be compatible with Freon 113.

a. Add 600 ml of Freon 113 to a 800 ml beaker. Place a swab in the Freon 113.

b. Bring the solvent to a boil and continue to boil for 10 minutes, agitating the swab by means of a clean pair of laboratory tongs.

c. Remove the swab from the beaker and place in a second beaker containing 600 ml Freon 113.

d. Repeat the boiling process.

e. Transfer the swab into a SOXHLET Extractor Vessel. Add 200 ml of blanked Freon 113 to the Soxhlet flask and reflux for 2 hours.

f. Transfer the 200 ml solvent into a beaker and analyze for NVR content according to MSFC-PROC-1831.

g. Repeat the extraction process until 2 identical results are obtained.

h. Remove the swab from the SOXHLET extractor by means of a clean pair of tongs. Wrap the swab in clean aluminum foil.

B. FLUSH METHOD

1. Initial cleaning as follows:

a. Wash all containers with hot soapy (Joy or LOC) water until clean. Rinse well with distilled water.

b. Wash all equipment surfaces, which will contact samples, with hot soapy (Joy or LOC) water. Rinse well with distilled water.

c. Rinse with reagent grade Ethyl alcohol – dry with Nitrogen (GN_2).

d. Rinse with clean acetone – dry with Nitrogen (GN_2).

e. All trichlorotrifluoroethane (Freon 113) used in this procedure shall be analyzed for NVR by evaporating 500 ml and analyzing in accordance with MSFC-PROC-1831. The NVR shall not be higher than the MIL-STD 81302B limit.

f. Rinse with precision trichlorotrifluoroethane (Freon 113) and dry with Nitrogen (GN_2).

CAUTION!!

2. Prepare weighing dish as follows:

3. Identify each dish to be used with unique number (scribe with instrument that has been cleaned with Freon).

4. Soak new aluminum weighing dish in clean acetone for a minimum of one hour.

5. Remove dish from acetone. Rinse inside of dish with a stream of Freon (use appropriate wash bottle or pressurized source).

6. Place weighing dish in oven (50°C) and allow to dry for a minimum of 1.5 hours.

7. Remove from oven and place in desiccator and allow to cool for a minimum of 30 minutes.

CAUTION!!

Cover shall be kept securely on desiccator except to insert or remove dishes or replace desiccant.

8. Check desiccant in desiccators daily, replace or reclaim when needed.

9. SAMPLING SWAB METHOD

A. Using a clean pair of tongs which have been rinsed with Freon 113, remove the clean, blanked swab from the aluminum foil wrapper, taking care during the whole sampling process not to touch the swab with the hands.

NOTE

Sampling can be done by hand if the person is wearing clean Teflon gloves. If tubing is being sampled one can place the swab into a stainless steel wire loop held by a stainless steel quarter inch tube.

B. Saturate the swab with blanked Freon 113.

C. Swab the critical surface by wiping up the area, turning the swab often to expose a clean area to the critical surface being swabbed. If possible a minimum of three square feet shall be sampled and the results reported in square feet.

D. Return the swab to the wrapper (aluminum foil) without drying and enclose securely.

E. Note the area sampled in square feet and label the swab package (a minimum of three square feet per swab shall be sampled).

10. SAMPLING FLUSH METHOD

A. Transfer a 500 ml. volume of the solvent to the Teflon bottle. There shall be a container of solvent for every sample to be taken.

B. After the surface to be sampled has been selected, flush that area from top to bottom into a clean baseline 1000 ml. beaker. If possible at least three square feet of surface will be sampled.

C. Cover the above beaker with Aluminum foil and identify the sample.

11. ANALYSIS

A. Swab method

1. Using a pair of tongs which have been rinsed with Freon 113, place the swab in a SOXHLET extracting vessel using care not to let the swab extend beyond or obstruct the siphon.

2. Add 200 ml of blanked Freon 113 in the bottom flask of the SOXHLET extractor apparatus.

3. Allow the solvent to siphon a minimum of 10 times.

4. Quantitatively transfer the extraction solvent from the boiling flask to a clean beaker.

5. The analysis shall be done per paragraph B. below.

B. Flush method

NOTE

Consideration should be given to sample types, (i.e., different solvent) which would dictate temperature, etc. This procedure assumes that the solvent used is trichlorotrifluoroethane.

The following steps shall be performed within the confines of the fume hood unit.

1. The sample shall be filtered with 0.45 µm millipore filter into a clean vacuum flask. Note: Use appropriate vacuum hose – Urethane, etc.

2. Transfer the sample (normally 500 ml) to the clean flash evaporator flask.

3. Fill evaporation pan with water.

4. Turn on the immersion heater and adjust temperature to 50 degrees Centigrade (50°).

5. Turn cold water supply (approx. 25°C) and adjust water feed tube so that running water covers the entire outer surface of the condensing flask.

6. Evacuate the system with vacuum source (a liquid trap should be used in-line of vacuum to prevent solvent from reaching pump oil) and start flash evaporator motor.

7. Evaporate the sample to a 10–20 milliliter volume.

8. Release vacuum.

9. Turn of motor.

10. Turn off cooling system.

11. Using clean laboratory tongs, remove a clean dish from desiccator. Weigh the dish on the analytical balance. Record the weight (tare), dish number, sample number and sample identification in the laboratory log book.

12. Remove the flask from evaporator and wipe the residue from the outside of flask with Kim wipe or equal. Care shall be taken not to introduce water into dish.

13. Transfer sample from evaporator flask to the clean, tared aluminum dish.

14. Rinse inner wall of evaporation flask with 5–10 milliliters of Freon and pour contents into the tared aluminum dish.

15. Using clean laboratory tongs, place into second oven for a minimum of 1.5 hours at 50°C.

16. Remove with tongs and place into second desiccator to cool for a minimum of 30 minutes.

17. Weigh dish again.

18. Record second weight.

19. Subtract tare weight from second weight then report results as * NVR.

20. If identification of contamination is desired infrared spectroscopy will be used.

* The NVR should be reported in milligrams per foot square (mg/ft2).

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DOCUMENTATION RELEASE LIST
GEORGE C. MARSHALL SPACE FLIGHT CENTER

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CH	DOCUMENT NUMBER	DRL DSH	DRL REV	TITLE	CCBD NO.	PCN	PC	EFFECTIVITY
*	MSFC-PROC-1832	202	-	SAMPLING/ANALYSIS OF NONVOLATILE CRITICAL SURFACE PROCEDURE	000-00-0000	0000000	ZA	NONE

CHG NO.	CHG REV	CHG NOTICE	RESPONSIBLE ENGINEER	RESPONSIBLE ORGANIZATION	ACTION DATE	DESCRIPTION
			B. H. NERREN	EH32	02/23/94	BASELINE RELEASE
*	1	DCN000	EUGENA GOGGANS	EO03	02/22/07	DOCUMENT RELEASED THRU PDS. NO LONGER TRACKED IN ICMS.

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DWG SIZE	DRAWING NUMBER	DWG REV	EPL/DRL/DDS NUMBER	DWG REV	EPL DSH	EPL REV	EO DASH NUMBER	EO REV	PART NUMBER
			MSFC-HDBK-1453		202	-			
			MSFC-HDBK-1674		202	-			
			MSFC-HDBK-2221		203	-			
			MSFC-HDBK-505		202	-			
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			MSFC-SPEC-521		202	-			
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			MSFC-STD-1249		202	-			
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			MSFC-STD-246		202	-			
			MSFC-STD-2594		203	-			

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DWG SIZE	DRAWING NUMBER	DWG REV	EPL/DRL/DDS NUMBER	DWG REV	EPL DSH	EPL REV	EO DASH NUMBER	EO REV	PART NUMBER
			MSFC-STD-2903		202	-			
			MSFC-STD-2904		202	-			
			MSFC-STD-2905		202	-			
			MSFC-STD-2906		202	-			
			MSFC-STD-2907		202	-			
			MSFC-STD-366		202	-			
			MSFC-STD-383		202	-			
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			MSFC-STD-506		203	-			
			MSFC-STD-531		202	-			
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			MSFC-STD-561		203	-			
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EUGENA GOGGANS
12/19/06

SUBMITTED BY:

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