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Marshall Space Flight Center, Alabama 35812

PROCEDURE

THE ANALYSIS OF NONVOLATILE RESIDUE CONTENT

BASED ON ASTM F 331-72

GEORGE C. MARSHALL SPACE FLIGHT CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

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Prepared by:

Billy H. Nerren, EH32 — Date: 4-4-90

Approved by:

S. V. Caruso, EH32
Chief, Analytical and Physical
Chemistry Branch — Date: 4-11-90

C. R. McIntosh, EH31
Chief, Non-Metallic Materials
Division — Date: 4/11/90

C. K. Key, EH02
Chief, Materials Selection
And Control Office — Date: 4-18-90

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

PROCEDURE

THE ANALYSIS OF NONVOLATILE RESIDUE

BASED ON ASTM F 331-72

1. PURPOSE

The purpose of this procedure is to establish a standard method for analysis of nonvolatile residue in liquids.

2. SCOPE

This procedure specifies the method by which liquids shall be analyzed to quantitatively determine nonvolatile residue. Nonvolatile residue (NVR) by this procedure are those materials measured in milligrams per square foot, after filtration of the suspended solids in the solvent.

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. ABBREVIATIONS

µm	micrometer
ml	milliliter
mg	milligram
ft	feet
ft^2	foot/square
C	centigrade
NVR	Nonvolatile Residue

5. 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

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	Trichlorotrifluoroethane (or equal) per MIL-C-STD 81302 B

6. 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.

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 (Freon 113) prior to each use.

D. Samples to be analyzed for NVR shall be filtered through a 0.45 µm filter.

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

7. EQUIPMENT PREPARATION

A. Initial cleaning as follows:

1. Wash all surfaces that will contact samples,with hot soapy (Joy or LOC) water until clean. Rinse well with distilled water.
2. Rinse with 200 proof Ethyl Alcohol and dry with Nitrogen (GN_2).
3. Rinse with clean acetone - dry with Nitrogen(GN_2).
4. Rinse with trichlorotrifluoroethane (Freon 113) and dry with Nitrogen (GN_2).

B. Prepare weighing dish as follows:

CAUTION!!

Never touch weighing dish with fingers. Always use laboratory tongs when handling.

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

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

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

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

5. 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.

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

8. ANALYSIS

This procedure assumes that the solvent used is trichlorotrifluoroethane . If other solvents are selected then consideration should be given to sample types which would dictate temperature and millipore pad selection.

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

A. The sample shall be filtered with 0 .45 µm filter into a clean vacuum flask.

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

C. Fill evaporation pan with water.

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

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

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

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

H. Release vacuum.

I. Turn off motor.

J. Turn off cooling system.

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

L. 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.

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

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

O. Using clean laboratory tongs, place in oven for a minimum of 1.5 hours at 50°C.

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

Q. Weigh dish again.

R. Record second weight.

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

T. Infrared Spectroscopy should be used If identification of the contamination is desired

*The NVR can be from a surface area in square feet or volume from some system.

PACKAGE NO. 10443R

DOCUMENTATION RELEASE LIST
GEORGE C. MARSHALL SPACE FLIGHT CENTER

MSFC CODE IDENT 14981/339B2
ISSUE DATE FEB 22 2007

C H	DOCUMENT NUMBER	DRL DSH	DRL REV	TITLE	CCBD NO.	PCN	PC	EFFECTIVITY
*	MSFC-PROC-1831	202	-	ANALYSIS OF NONVOLATILE RESIDUE 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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02/15/07

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PROGRAM/PROJECT: MULTI LAST UPDATED: 02/22/07

NOMENCLATURE: MSFC-STD- GOING TO NONE EFFECTIVITY

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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	-			
			MSFC-HDBK-670		202	-			
			MSFC-MNL-1951		209	-			
			MSFC-PROC-1301		202	-			
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			MSFC-STD-1249		202	-			
			MSFC-STD-1800		202	-			
			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	-			
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			MSFC-STD-486		202	-			
			MSFC-STD-506		203	-			
			MSFC-STD-531		202	-			
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			MSFC-STD-561		203	-			
			MSFC-STD-781		202	-			

SUBMITTED BY ENGINEERING AREA:	BASIC CHANGE	PARTIAL	COMPLETE	CLOSES ACTION
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PREPARED BY:
EUGENA GOGGANS
12/19/06

SUBMITTED BY:

CONCURRENCE:

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TO RELEASE DESK 02/22/07 10:00
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REMARKS:

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