

MSFC-SPEC-164 REVISION: E EFFECTIVE DATE: July 21, 2020

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

# **EM22**

# MSFC TECHNICAL STANDARD

# SPECIFICATION FOR CLEANLINESS OF COMPONENTS FOR USE IN OXYGEN, OXIDIZER, FUEL, AND PNEUMATIC SYSTEMS

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## **DOCUMENT HISTORY LOG**

Status (Baseline/ Revision/ Canceled)	Document Revision	Effective Date	Description
Baseline	-	04/16/1962	Initial Release
Revision	A	10/01/1970	
Revision	В	11/19/1994	
Revision	С	03/01/2010	Complete revision for Constellation and future projects. Will not be imposed on Space Shuttle or International Space Station.
Revision	D	01/30/2014	<ol> <li>Revision D release was authorized by the MSFC Technical Standards Document Control Board (DCB) through the Multiprogram Document Management System (MPDMS).</li> <li>Revise Table I and revise the "default" Table II cleanliness to reflect current practice and understanding of oxygen system particle impact ignition.</li> <li>Remove obsolete references to Space Shuttle and Constellation documents and requirements.</li> <li>Reduce the required levels of solvent for sampling and lower NVR background requirements to reflect current industry practice and minimize solvent use and processing time.</li> <li>Clarify sections on cleanliness testing, packaging, and qualification of alternative test methods.</li> </ol>
Revision	Е	07/xx/2020	Updated to identify EM22 as responsible organization. Grouped cleaning process verification sections. Grouped field cleaning sections. Removed obsolete reference to Class I particulates from Table 1. Changed final rinse solution NVR allowable to match limit for Level B NVR test fluid. Deleted callout for obsolete document MSFC-PROC-1832. Added Appendix C detailing NVR Swab Sample Testing procedure.

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#### 1. SCOPE

This specification establishes surface cleanliness requirements for oxygen, fuel, and pneumatic components used in space vehicle fluid systems and associated ground support equipment (GSE) and test facilities. This specification is not intended to specify cleanliness requirements for composite surfaces used in cryogenic service, but it may be used to specify internal cleanliness requirements for metal-lined composite over-wrapped pressure vessels (COPVs). General cleaning requirements, verification procedures, drying and packaging requirements are provided. Each user shall require sub-tier documents to address processing, system cleanliness, solvent selection, cleanliness verification, and maintenance. An engineering assessment shall be necessary to establish requirements for each system.

#### 1.1 Cleanliness Levels

Cleanliness levels for particulate and nonvolatile residue (NVR) are listed in Table I. Unless otherwise specified, the minimum cleanliness levels for new or re-cleaned systems, subsystems and components are specified in paragraph 3.2.3 and Table II.

#### 1.2 Alternate Procedures

Methods and procedures other than those enumerated in this document may be utilized with prior approval of the NASA procuring activity.

This document applies the following: all mandatory actions (i.e., requirements) are denoted by statements containing the term "shall." The following terms also apply: "may" or "can" denote discretionary privilege or permission, "should" denotes a good practice and is recommended, but not required; "will" denotes expected outcome, and "are/is" denotes descriptive material.

#### 1.3 Alternate Cleanliness Levels

For system performance or safety, some propulsion systems, such as hypergolic systems, may require cleanliness levels more stringent than those defined in Table I. When cleanliness levels more stringent than those shown in Table 1 are required they may be specified on the engineering drawing or specification in accordance with IEST-STD-CC1246, or particulate levels tailored from IEST-STD-CC1246, with cleaning, inspection, and packaging in accordance with this document. When a cleanliness level is required that cannot be defined by one of these specifications, the cleanliness requirement may be explicitly specified on the engineering drawing or specification as a custom cleanliness level. The custom cleanliness level shall state the maximum number of particles allowed per 0.1 m<sup>2</sup> in defined particle size ranges.

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**Table I. Classification of Cleanliness** 

	PARTICULATE LEVELS				
CLASS	PARTICLE SIZE IN MICRONS	MAX. NUMBER PER 0.1 m <sup>2</sup>			
II	>1000 700 <x≤1000 175<x≤700< td=""><td>0 40 150</td></x≤700<></x≤1000 	0 40 150			
III	NO SILTING >800 NO SILTING	0			
III X	>800 175 <x≤800 NO SILTING</x≤800 	0 5			
IV	>400 NO SILTING	0			
IV X	>400 175 <x≤400 NO SILTING</x≤400 	0 5			
V	VISUALLY CLEAN/NO SILTING	3			
	<u>NVR LEVELS</u>				
<u>LEVEL</u>	$\frac{\text{MAXIMUM}}{\text{mg/0.1 m}^2}$				
A	1				
В	5				

NOTE: For the purpose of this specification 0.1 square meter = 1 square foot.

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#### 2. APPLICABLE DOCUMENTS

Unless otherwise noted the latest revision of the following documents are applicable to the extent specified herein. In the event of a conflict between the documents referenced herein and the contents of this specification, the content of this specification shall take precedence. Equivalent grades of chemicals may be used upon approval from the NASA procuring activity.

#### **2.1** Government Documents

## 2.1.1 Specifications

MSFC-STD-3535

MIL-D-3464	Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-PRF-27401	Propellant Pressurizing Agent, Nitrogen
2.1.2 Standards	
MIL-STD-129	Military Marking for Shipment and Storage
MSFC-STD-246	Standard Design and Operational Criteria for Controlled Environmental Areas

Activities at SSC and MSFC

Standard for Propellants and Pressurants used for Test and Test Support

## 2.2 Non-Government Industry Standards

AMS 3647	Film and Sheet, Polyfluoroethylenepropylene (PFEP)
ASTM E 1216	Standard Practice for Sampling for Particulate Contamination by Tape Lift
ASTM F 303	Standard Practices for Sampling Aerospace Fluids from Components
ASTM F 311	Standard Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters
ASTM F 312	Standard Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters
ASTM F 331	Standard Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Flash Evaporator)

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ASTM Manual 36 Oxygen System Design, Materials Selection, Operations, Storage and

Transportation

IEST-STD-CC1246 Product Cleanliness Levels and Contamination Control Program

## 3. REQUIREMENTS

#### 3.1 General

All parts, components, assemblies, systems or related equipment for use in space vehicles and related GSE and test facility equipment shall be cleaned, inspected, and packaged in accordance with this specification. Assembled parts which may be damaged during the cleaning operation shall be disassembled to a level to permit cleaning. Designs for systems and system components should, where practical, include the capability to remove all valves and components from the system for precision cleaning.

**SAFETY PRECAUTION** – It is the responsibility of all users of this specification to review pertinent Safety Data Sheets (SDSs) and materials specifications to assure safety of personnel and protection of the environment and facilities in fulfilling the requirements of this document.

## 3.2 Cleaning

#### 3.2.1 Pre-Cleaning

All significant surfaces of system hardware shall be pre-cleaned to remove dirt, grit, scale, corrosion, grease, oil and other foreign matter prior to any final precision cleaning process. Metallic items shall be surface treated (cleaned, passivated, and/or coated), as applicable, to prevent latent corrosion and contamination. Assembled items that do not lend themselves to this type of treatment shall be treated prior to assembly. Surface treated areas degraded during subsequent fabrication and assembly shall be reprocessed, as required, to restore the original protective finish.

#### 3.2.2 Inspection Prior to Precision Cleaning

Significant surfaces of system hardware which have been pre-cleaned shall be visually free of dirt, grit, scale, corrosion, grease, oil, and foreign objects prior to proceeding to any precision cleaning operation. Scale-free discoloration due to welding or passivation is permitted.

## 3.2.3 Precision Cleaning

All significant hardware surfaces shall be precision cleaned to meet the cleanliness levels of Table II or as specified on the engineering drawing or component specification. Precision cleaning operations shall be performed in an environment compatible with the component

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cleanliness requirements to preclude adverse effect on the functional performance. Exemptions may be requested for hardware which, due to size or other considerations, cannot meet this requirement. As a minimum, cleaning shall be performed in an environment that does not adversely affect the function of the hardware. Cleaning and verification of components to alternate cleanliness levels specifying limits on particles smaller than 150 microns shall be performed under High Efficiency Particulate Air (HEPA) filtered conditions. Reference MSFC-STD-246 for information on HEPA filtration and clean work area facilities. Precision-cleaned articles shall be packaged per Section 5 immediately after verification and drying operations or suitably protected prior to leaving the controlled environment.

**Table II. Product Cleanliness Requirements** 

System	Cleanliness Level
LO2/GO2 Systems	
Metallic & Fluorocarbon Components	III A
Metallic Vessels	II B
Non-metallic (except Fluorocarbons)	V
Fuel Systems	
Metallic & Fluorocarbon Components	IV
Metallic Vessels	II
Non-metallic (except Fluorocarbons)	V
Pneumatic and Pressurant Systems (minimum)	
Metallic & Fluorocarbon Components	
Interfacing with Fuel Systems	IV
Interfacing with Oxygen Systems	III A
Metallic Vessels (with downstream interface filter)	
Interfacing with Fuel Systems	II
Interfacing with Oxygen Systems	II A
Non-metallic (except Fluorocarbons)	V

## 3.2.4 Final Rinsing Solution

The final rinsing solution shall meet or exceed the cleanliness requirements for which they are intended and meet the following minimum requirements at the point of use:

3.2.4.1 <u>Particle Allowable</u>. There shall be no particle greater than  $175\mu$  in any dimension and no more than 5 particles between 100 and 175  $\mu$  per 500 ml when tested per ASTM F 312.

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- 3.2.4.2 NVR Allowable. Nonvolatile residue (NVR) in fluids used to clean surfaces with an NVR requirement shall not be greater than 3 milligrams per 100 milliliters, as determined by ASTM F 331 or equivalent test method. When the final rinsing fluid is used for verification of surface NVR cleanliness, the NVR shall have a maximum NVR background level of 3 mg per 100 ml for sampling to Level B or 0.4 mg per 100 ml for sampling to Level A.
- 3.2.4.3 <u>Water as final rinse solution.</u> If water is used as the final rinse fluid it shall meet the particulate requirements of paragraph 3.2.4.1, the minimum specific resistance (maximum conductance) shall be 50,000 ohms/cm, and the pH shall be between 6.0 and 8.0.

**NOTE:** These are minimum final rinsing solution requirements established for cleaning of tanks and large components. Cleaning processes for small components or for components that require cleanliness levels cleaner than shown in Table I may require greater purity levels for the final rinsing solution and this final rinse solution may not be suitable as a verification flush solution.

## 3.2.5 Process Approval

Cleaning processes shall be left to the discretion of the user. However, the process shall not be detrimental to hardware being cleaned, per section 3.2.6 (compatibility), and process approval shall be obtained from the NASA procuring activity prior to cleaning and handling. Process verification details are provided in Section 4.

To obtain approval, the contractor shall submit to the procuring activity the following information:

- a. Cleanliness levels to be achieved, which shall meet or exceed those specified on the engineering drawing or specification. If the cleanliness level is not specified on the engineering drawing or documentation, the proposed cleanliness level shall be specified in the process document, including analysis and rationale for the selected cleanliness level.
- b. Description of items to be cleaned including identification of materials.
- c. Processing materials, to include as applicable, trade names, specifications, chemical and physical properties, and compatibility information as specified in 3.2.6.
- d. Processing equipment and cleaning procedures to be used.
- e. Quality assurance provisions to be utilized. This shall include in-process control procedures to prevent contamination, latent corrosion, or other degradation of surfaces and opened systems or vessels; and procedures to perform inspections and verify test results.

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- f. Controlled environment levels to be maintained for cleaning and handling. MSFC-STD-246 shall be used to evaluate, verify and monitor applicable areas.
- g. Packaging methods and materials.
- h. Verification methods and materials.

Cleaning processes to be performed on-site at MSFC or MAF shall be reviewed by MSFC Occupational Health Services.

**NOTE:** Documentation from all subcontractors shall be maintained and made available for review by the Government.

#### 3.2.6 Materials

Selection of materials used in processing and verification testing is left to the discretion of the user. However, the fluids shall be compatible with the item being cleaned and capable of removing the most probable contaminants.

The following compatibility issues, as applicable, shall be considered and evaluated in the selection of processing materials.

- a. Corrosion
- b. Stress corrosion cracking
- c. Embrittlement
- d. Leaching
- e. Masking of crack-like indications
- f. Residue
- g. Crazing (non-metallics)
- h. Reversion (non-metallics)
- i. Hydrolysis (non-metallics)

**NOTE:** ASTM Manual 36 provides additional guidance for the safe use of oxygen and oxygen systems.

## 3.3 Detailed Product Cleanliness Requirements

Precision cleaned components and vessels shall be verified to meet applicable cleanliness requirements specified in Table II or as specified on the engineering drawing or component specification. Verification sampling shall be performed in an environment that is compatible with the environment in which cleaning was performed.

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## 3.3.1 Use of Alternate Acceptance Criteria

Alternate verification procedures and/or corresponding cleanliness levels may be used only upon demonstration of equivalence in accordance with 6.1 and upon written approval by the NASA procuring activity. Supplemental information concerning chemical species of contaminants and their potential reactivity may be useful in determining alternate acceptance criteria.

*CAUTION:* It has been observed that both NVR and particulate levels increase after a system has been exposed to cryogenic media. This increase in contamination levels should be taken into consideration in establishing methods for system verification.

#### 3.3.2 Visual Cleanliness

Surfaces of all components that will contact the respective service medium shall be visually free of contaminants such as moisture, corrosion, scale, dirt, oil, grease, wax, gum, accumulations of fiber or silt, and other foreign material when inspected in accordance with 4.5.1. Scale-free discoloration due to welding or passivation is permitted. Discrete particles and fibers that are within the size and numerical limits of the Class are acceptable.

- 3.3.2.1 <u>Surfaces Inaccessible to Visual Inspection</u>. Surfaces inaccessible to visual inspection shall be accepted or rejected based upon 3.3.4 (NVR) and 3.3.5 (particulates), provided that adequate measures have been implemented to assure that no foreign object debris is entrapped within the inaccessible area. Borescope/videoscope inspection may be required.
- 3.3.2.2 <u>Use of Borescope/Videoscope</u>. Care shall be taken when using borescope/videoscope equipment not to introduce contamination into the part or component. Borescope/videoscope equipment contains sensitive optics and care shall be taken in cleaning the equipment such that it is acceptable to use in a clean system. Consult with the manufacturer regarding acceptable solvents and evaluate whether they are compatible with clean systems.

## 3.3.3 Acidity or Alkalinity

Surfaces that have been cleaned or that have come in contact with aqueous or semi-aqueous media or chemical solutions (e.g., caustics, acids, etc.) shall register a pH between 6.0 and 8.0 on completion of the final rinse when evaluated in accordance with 4.5.2 (acidity/alkalinity).

#### 3.3.4 Nonvolatile Residue

Nonvolatile residue, as defined in Appendix B, shall not exceed the limits specified in the engineering documentation when tested in accordance with 4.5.3.

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#### 3.3.5 Particulate Contamination

Particulate contamination shall not exceed the limits specified in the engineering documentation when tested in accordance with 4.5.3.

#### **3.3.6 Silting**

Silting, as defined in Appendix B, shall not be permitted.

## 3.4 Dryness

Components shall be thoroughly dried to remove residual cleaning, rinsing, and/or verification media. Effluent gas shall not exhibit an increase in moisture content greater than 5 PPM or measurable increase in concentration of residual organic solvents when tested in accordance with 4.4.1. Items which do not lend themselves to this type of drying or testing procedure shall be dried and tested in accordance with procedures approved by the NASA procuring activity.

*CAUTION:* Most solvents are not oxygen compatible. It is therefore critical to assure removal of these substances prior to packaging or placing hardware into service.

## 3.4.1 Drying and Purging Gases

Gases used in drying processes shall conform to the following and to cleanliness and quality assurance requirements for gases as specified in MSFC-STD-3535. Condensable hydrocarbon sampling per MSFC-STD-3535 is not required on gas delivery systems that do not contain hydraulic pumps or other components that could be a source of condensable hydrocarbon contamination to the system.

- 3.4.1.1 <u>Nitrogen</u>. Nitrogen used for drying or purging shall conform to MIL-PRF-27401, type 1, grade B.
- 3.4.1.2 <u>Air</u>. For drying/purging of tanks/vessels where the use of dry gases is impractical, air shall meet the following minimum requirements:
- a. No particulate matter > 100 microns
- b. Relative humidity 60% maximum
- c. Hydrocarbon content 15 PPM maximum except 20 PPM when the carbon chain of 5 or above does not exceed 5 PPM.

#### 3.5 Post-Verification Operations

Fluids or gases contacting cleaned surfaces after final cleaning or testing for cleanliness, as a minimum, shall meet the requirements for final rinsing fluids per 3.2.4 and drying gases per 3.4.1.

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## 3.6 "Excepted" Components

Excepted components shall consist of items and systems that cannot be processed per the requirements of this document. Components that cannot be cleaned and certified using normal procedures or facilities because of size, construction, materials of construction, etc. may be processed as excepted components. Excepted components require written approval of the NASA procuring activity. These items shall be cleaned to the intent of this specification as practical, and identified as "EXCEPTED" on all tags and documentation.

## 3.7 Ground Test Systems

Ground or facility test equipment for LO2, GO2, and fuel systems shall be evaluated for cleanliness on a predetermined schedule or upon assessment of need. An engineering evaluation shall be made to identify the component or area to be tested based upon the systems configuration and pressure. As a minimum, the evaluation shall identify the component most likely to accumulate contaminants due to geometry, location, internal surface finish, and other technical considerations.

## 3.7.1 Oxygen Ground Test Systems Operating at 5000 psig or Greater

For oxygen systems operating at 5000 psig or greater, the selected component(s) shall be evaluated for particulate and NVR per 4.3 except that an NVR analysis shall not exceed Table 1 Level B (5 mg/0.1 m<sup>2</sup>).

## 3.7.2 Oxygen Ground Test Systems Operating at Less than 5000 psig

For oxygen systems operating at less than 5000 psig the following NVR criteria shall apply:

- a. An NVR analysis of Table 1 Level B (5 mg/0.1 m²) or greater shall require approval of the organization's Test Director for continued operation.
- b. An NVR analysis of 10 mg/0.1 m<sup>2</sup> or greater shall require the approval of the organization's Director of Safety /Quality Assurance or designated authority with concurrence of applicable Materials and Processes and Test organizations for continued operation.
- c. An NVR analysis of 20 mg/0.1 m<sup>2</sup> or greater shall be cause for discontinued use until the entire system is re-cleaned and verified clean to a minimum of Table I Level B (5 mg/0.1 m<sup>2</sup>).

#### 3.7.3 Field Cleaning of Ground Test Systems

For ground test systems only, field cleaning shall only be performed when one of the following criteria is met:

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- a. The item is a part of a fixed installation and cannot be removed to a remote cleaning site.
- b. Cleaned spare parts are not available and removal to a remote site would unduly impact operations.
- c. The item is precision cleaned and field cleaning is done during the assembly process on-site as an assurance function.
- 3.7.3.1 <u>Field Cleaning Process Requirements.</u> Field cleaning shall be performed using fluid flow velocities greater than 3 m/s, pressurized spraying, and rapid draining to achieve visual cleanliness. Alternative cleaning methods shall be approved by the NASA procuring activity.

#### 4. CLEANING PROCESS AND DRYING VERIFICATION

The cleaning process shall be verified as follows.

The cleaning activity is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the cleaning activity may utilize their own or any other inspection facilities and services acceptable to the NASA procuring activity. Inspection records of examinations and tests shall be kept complete and available to the government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure supplies and services conform to specified requirements.

#### **4.1 Visual Inspection**

The sample for visual inspection shall consist of all cleaned components except as noted. Components, such as small diameter tubing (1/2 inch diameter or less), having limited accessibility to visual examination shall be accepted or rejected on the basis of the inspections of 4.5.2 (acidity/alkalinity) and 4.5.3 (particulate and NVR).

**NOTE:** Tubing and pipe can pass fluid sample analysis and still fail visual inspection. Depending on the geometry of the part and nature of the contamination, borescope/videoscope inspection or black light may be required.

## 4.2 Acidity/Alkalinity

Surfaces which have been cleaned and rinsed with aqueous media shall be evaluated in accordance with 4.5.2.

#### 4.3 Particulate/NVR Sample

Except as noted in 4.3.1, the sample for the tests of 4.5.3 (particulate and NVR) shall consist of a minimum of 5% of the items cleaned, but not less than one sample for each group of 20 or less of the items cleaned. The sample shall be selected at random from production items that have

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been cleaned, visually inspected in accordance with 4.5.1, and found acceptable. The sample shall be selected in a manner that will provide maximum representation of the affected lot. In this context a lot does not necessarily require identical parts but does include all hardware processed in one operation. The test sample and the segment of production that it represents shall be clearly identified as specified by the NASA procuring activity. All items shall be verified visually clean, and filter pads for particulate counts inspected for silting.

## 4.3.1 Procedure for Obtaining NVR/Particulate Sample

Samples for quantitative analyses shall be obtained as follows. The procedure for particle and NVR sampling shall be flushing of the product with an approved solvent in accordance with ASTM F 303. The collected flush sample shall be processed in accordance with ASTM F 311. Hardware shall be thoroughly dried after the flushing operation.

**NOTE:** Where component flushing is impractical, particle count sampling may be performed by the tape lift method in accordance with ASTM E 1216 or an approved equivalent, and NVR sampling may be performed by the swab method in accordance with Appendix C or by sampling with extracted wipers in accordance with a procedure approved by the NASA procuring activity.

- 4.3.1.1 Components with Surface Area Less than 0.1 m<sup>2</sup>. Samples for small components (components having a significant surface area less than 0.1 m<sup>2</sup>) shall consist of a sufficient number of components to make up 0.1 m<sup>2</sup> of surface area. When the total quantity of items procured have a combined surface area less than 0.1 m<sup>2</sup>, a quantity of cleaned items sufficient to make up 0.1 m<sup>2</sup> of surface area shall be used in preparation of the sample or a special test procedure may be used upon written request to and approval by the NASA procuring activity. For particularly small items with a total area significantly less than 0.1m<sup>2</sup>, the criterion is 1 milligram NVR maximum, or equivalent, per 500 ml of verification solvent.
- 4.3.1.2 Components with Surface Area Between 0.1 and 0.5 m<sup>2</sup>. A 200 ml, nominal, test solution shall be flushed over the significant surfaces of components with surface area between 0.1 and 0.5 m<sup>2</sup> and collected for quantitative analysis per 4.3.2.
- 4.3.1.3 Components with Surface Area Greater than 0.5 m<sup>2</sup>. The significant surfaces of components with surface area greater than 0.5 m<sup>2</sup> shall be flushed with 100 ml, nominal, of test solvent per 0.1 m<sup>2</sup> of surface area. The test solution shall be collected and thoroughly agitated. A 200 ml sample shall be obtained from the agitated solution for quantitative analysis per 4.3.2.
- <u>4.3.1.4 Large Containers</u>. Large containers/vessels shall be verified by a procedure submitted by the user and approved by the NASA procuring activity.
- 4.3.1.5 Small Containers. All small containers shall be submitted for testing.

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<u>4.3.1.6 Rigid Tubing.</u> Verification of the cleaning procedure for rigid tubing may be determined and the cleaning process qualified as specified herein. Samples for qualification of the cleaning process shall be selected as follows:

- a. Select a minimum of 5 cleaned tubes with a minimum of 3 feet cumulative length and having a minimum combined significant surface area of 0.1m<sup>2</sup> from each size (diameter) and type tube (material) to be cleaned. Tubes shall have been cleaned in accordance with the exact procedures and utilizing equipment approved in accordance with 3.2.
- b. Evaluate sample, as applicable, to meet requirements of 3.3.3 and 3.3.4.
- c. Upon satisfactory qualification of the cleaning procedure and equipment to meet applicable requirements of 3.3.4(NVR) and 3.3.5 (particulate contamination), periodic spot checks in addition to the inspections of 4.5.1 (visual inspections) and 4.5.2 (acidity/alkalinity) shall be made to insure cleaning procedures continue to be effective.

4.3.1.7 Convoluted Flex Hoses. Special attention to cleaning is required for convoluted flex hoses. All convoluted flex hose components shall be verified as precision-clean in a vertical orientation. For flex hose tube diameters equal to or greater than one inch, verification of precision cleanliness shall be performed by sampling a rinse fluid applied internally through use of a high-pressure nozzle to the entire length of the flex hose. For flex hose tube diameters less than one inch, the use of a high-pressure nozzle is preferred, but verification may be performed by flushing a rinse fluid through the entire length of the flex hose with flex hose agitation.

## 4.3.2 Quantitative Analysis of NVR/Particulate Sample

NVR analysis shall be conducted per ASTM F 331, and particulate analysis shall be conducted per ASTM F 312 test method B. The solvent used for sampling shall be recorded with the test results.

## 4.3.2.1 Counting of fiber contaminants.

When fibers larger than the maximum allowable non-fiber particle size are counted per table I, counting shall be performed by microscopic analysis or by image analysis with visual inspection of the image to verify that fibers have been accurately interpreted by the image analysis system.

#### 4.3.2.2 Measurement of fiber length.

The length of twisted fibers may be measured, when counting by image analysis, by the use of an opisometer or equivalent on the fiber image or, when counting microscopically, may be approximated by measuring the fiber in segments.

## 4.3.2.3 Measurement of fiber width.

The width of a fiber shall be measured as the largest apparent projected width or diameter of the fiber.

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## 4.4 Dryness of Sample

The sample shall consist of a minimum of 5% of items dried, but not less than one sample for each group of 20 or less of items dried. The sample shall be selected at random from production items that have been cleaned in accordance with 3.2, verified in accordance with 3.3, and dried in accordance with 3.4, as applicable. The sample shall be selected in a manner that will provide maximum representation of the affected lot. A lot does not necessarily mean identical parts but does include all hardware processed in one operation. The verification sample and the segment of production that it represents shall be clearly identified as specified by the NASA procuring activity.

Alternately, the drying procedure may be verified for each hardware configuration, and the drying process qualified as specified herein. After qualification of the procedure and equipment for a specific hardware configuration, sampling shall be left to the discretion of the NASA procuring activity. Samples for qualification of the drying process shall be selected as follows:

- a. Select a minimum of 5 cleaned, verified and dried items of each of the hardware configurations to be qualified. Parts shall have been cleaned and dried in accordance with the same procedures and equipment previously approved in accordance with 3.2, 3.3, and 3.4.
- b. Evaluate samples in accordance with 4.4.1 (verification of the drying process) to meet the requirements of 3.4 (dryness).
- c. Upon satisfactory qualification of the drying procedure for each hardware configuration, the established drying cycle requirements shall be implemented. Periodic spot checks shall be made to insure that drying procedures continue to be effective.

## 4.4.1 Verification of the Drying Procedure

- a. Flow pre-filtered drying gas (3.4.1) through or over affected surfaces of the item being tested.
- b. For hardware processed with aqueous media, monitor the dew point of the drying gas entering and leaving the affected item to determine presence of moisture on cleaned and dried surfaces. An increase in moisture content of the drying gas of 5 PPM or greater shall necessitate additional drying prior to packaging or application of protective coverings.
- c. For hardware processed with halogenated solvents, monitor effluent drying gas with a halogen detector, to determine if affected surfaces are free from residual organic solvents. If no measurable concentrations are indicated by the halogen detector, affected surfaces shall be considered free from excessive residual organic solvents. Any measurable

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concentration above ambient in the drying gas shall necessitate additional drying prior to packaging or application of protective coverings.

- d. For hardware processed with alcohols or other hydrocarbons, monitor the effluent drying gas with a hydrocarbon detector. Any measurable concentration above ambient in the drying gas shall necessitate additional drying before packaging or application of protective coverings.
- e. When a flammable solvent is used for cleaning, flushing, or testing of liquid or gaseous oxygen systems or nitrogen tetroxide systems, the residual concentration of flammable solvent shall be verified as within acceptable limits prior to the introduction of flight fluids. This requirement does not apply to solvents used for hand-wiping operations or to piece parts except for assembled components that are required by engineering documentation to have a unit oxygen compatibility test.
  - 1. After purging with an inert gas, a 24 hour "lockup" of the component or assembly shall be conducted, at a minimum temperature of 15 °C.
  - 2. The solvent concentration in lockup gas samples shall not exceed 18 PPM when measured as methane or 10 PPM when measured using an instrument calibrated to the specific solvent utilized and capable of detecting 1 PPM.

**NOTE:** The lockup will typically be performed at ambient temperature. For systems with small internal volumes, a clean sampling reservoir may be added to increase the volume of gas in the system.

## 4.5 Acceptance Inspection

Acceptance inspection shall be performed as specified herein. Alternate acceptance inspection procedures, qualified by the user, shall be approved by the NASA procuring activity.

## 4.5.1 Visual Inspection

Surfaces of all components that will contact the respective service medium shall be visually inspected at a distance of 0.6 to 1.2 meters (2 to 4 feet) under a minimum incident light level of 500 lumens/meter<sup>2</sup> (50 foot-candles). An external light, ultraviolet light, or borescope may be required to examine internal surfaces. The presence of contamination shall require test/evaluation to determine acceptance or rejection. Scale-free discoloration of a surface due to welding and passivation shall be permitted.

**NOTE:** When inspection of piece parts at the minimum inspection distance is impractical, closer inspection is permitted. When interior volumes of tanks do not provide sufficient access to

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physically conduct an inspection within the defined VC range, the inspection shall be conducted at a distance that deviates from the defined range only to the extent required to physically perform the inspection.

#### 4.5.2 Acidity/Alkalinity Evaluation

External and internal surfaces which have been cleaned with aqueous media shall be tested for pH while the component is still wet from final rinse, or if dry, by wetting the surface of the component with a few drops of water (see 3.2.4.3). The pH indicating paper or other approved indicator shall be sensitive to the pH range of 6.0 to 8.0.

#### 4.5.3 Particle/NVR Cleanliness

Hardware shall be tested and accepted for visual inspection per 4.5.1 and acidity/alkalinity per 4.5.2, if applicable, prior to being submitted for tests. Sampling and analysis shall be conducted per Sections 4.3.1 and 4.3.2. Alternate methods may be used after qualification and written approval by the NASA procuring activity in accordance with 6.1.

Test fluids shall be selected by the user, approved by the NASA procuring activity, and shall be specified in the cleaning and verification process instructions. Test fluids used to verify the particulate cleanliness requirements shown in Table I shall meet the particle requirements 3.2.4.1 as a minimum. Test solvents used to verify nonvolatile residue shall have a maximum NVR background level of 3 mg per 100 ml for sampling to level B or 0.4 mg per 100 ml for sampling to Level A. To meet these levels, test fluids may require filtration and/or distillation prior to use. The selected solvent shall be chemically compatible with the component per section 3.2.6 and effective at removing contaminants of concern.

#### 4.5.4 Dryness

Hardware shall be tested for dryness per the procedures described in Section 4.4.1 to meet the requirement of section 3.4.

Visual inspection for dryness is allowed where appropriate, provided the user receives approval from the NASA procuring activity.

#### 4.5 Rejection

FAILURE TO MEET ANY REQUIREMENT OF SECTION 3.0 SHALL BE CAUSE FOR REJECTION

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#### 5. PACKAGING

## **5.1 Packaging of Components**

All significant surfaces or openings to significant surfaces shall be protected from contamination by double bagging the surfaces or openings with approved coverings (5.3.1), secured by heat sealing, closing with tape (5.3.2), or other approved method.

## **5.1.1** Application of Tape

Tape shall not contact or otherwise contaminate significant surfaces and, when feasible, shall not contact other product surfaces cleaned to visibly clean levels.

#### **5.1.2 Packaging of Fluid Line Connections**

Unless impractical due to small component size or shape, unmated fluid line connections shall be individually packaged and integrity sealed to facilitate clean mating operations at the assembly stage.

## 5.1.3 Outer Bags

Protected components shall be placed in visually clean outer bags meeting the requirements of 5.3.1 and shall be sealed with tamper-evident integrity seals.

#### **5.1.4 Inert Storage Package**

Interiors of the bags and parts shall be purged with a drying gas meeting the requirements of 3.4.2.1; bags shall be completely sealed to assure an inert storage package.

#### **5.1.5** Prevention of Damage

Sealed bags shall be over packed as necessary to prevent damage during storage and handling.

## **5.1.6** Moisture Barrier

At least one layer of the packaging system shall be a moisture barrier.

## **5.1.7** Other Packaging Methods

Other packaging materials compatible with the applicable service media may be used, however, gas purging and over packaging requirements shall be as listed above. If alternate methods are used, prior approval of materials and procedures shall be obtained from the NASA procuring activity.

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#### 5.18 Environmental Requirements for Packaging

Packaging operations involving cleaned and verified components should be accomplished within the same environmentally controlled area in which verification was performed. However, where impractical to package in the same environment, the environment shall not adversely impact the function of the hardware/component. Outer wrapping and over packing may be performed outside the controlled area.

#### **5.1.9** Use of Desiccants

Desiccants (5.3.3), when required for additional corrosion protection, shall not be placed in such a manner as to contaminate cleaned surfaces. Provisions shall be made for monitoring desiccants prior to use to assure dehydrating effectiveness. Even non-dusting desiccants may be a source of contamination for precision-cleaned components, therefore desiccants placed inside the inner bag or package shall be sealed within a clean water-vapor permeable film. Desiccants shall be packaged with a visual humidity indicator.

#### 5.2 Protection of Tanks and Containers

Immediately after drying, openings shall be covered with approved pre-cleaned dry covers (5.3.1) secured in a manner to prevent detachment or damage during handling, storage, or shipment. Covers shall be designed to prevent recontamination of tank interior. If exposed to uncontrolled environments, a positive pressure purge shall be maintained until the system is closed.

#### **5.3 Packaging Materials**

#### **5.3.1 Protective Materials and Devices**

Protective materials and devices that serve as contamination barriers shall be specified in the cleaning process document.

- 5.3.1.1 <u>Cleanliness of Materials</u>. Prior to use, protective packaging materials or devices shall be cleaned and dried to a level compatible with the component end item cleanliness requirements.
- 5.3.1.2 <u>Durability of Materials</u>. Under normal usage, materials or devices shall be lint-free, and shall not delaminate, peel, disintegrate, slough or otherwise deteriorate in a manner that will contaminate the cleaned item.
- 5.3.1.3 Oxygen Service. Inner packaging materials that protect the wetted surfaces of parts for liquid or gaseous oxygen service shall be compatible with LO2/GO2. Fluorohalocarbon films such as Aclar 22A, 22C, and 33C, or polyfluoroethylenepropylene (PFEP) film conforming to AMS 3647 are acceptable. This requirement applies to covers for the openings of tanks, feedlines, tubes, valves, and other fluid system components and to the inner bag for components

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where covers for openings are not used or where the part is to be fully immersed in the service fluid.

## **5.3.2** Tape

Tape used for precision-cleaned packaging shall be constructed of materials that do not outgas, shed particulate, or degrade during normal use.

#### 5.3.3 Desiccants

Desiccants used for packaging of precision-cleaned components shall meet the requirements of MIL-D-3464 Type II (non-dusting).

## 5.4 Marking/Identification

Unless otherwise specified by the NASA procuring activity, cleaned items shall be identified per MIL-STD-129 with appropriate certification tags and shall contain as a minimum the following information:

- a. Part or identification number.
- b. Contractor identification
- c. Contractor cleaning and packaging procedure identification
- d. Date of cleaning
- e. Title, date, and number of this standard
- f. Service medium or intended use of component
- g. Manufacturer's serial number
- h. Acceptance stamps
- i. Cleanliness level

## 6. NOTES

## **6.1** Approval of Alternate Verification Procedures

Alternate verification procedures may be used only upon qualification and written approval by the NASA procuring activity. For qualification of alternate procedures, the following methodology is required; in all instances statistically significant data shall be provided to the NASA procuring activity before alternative verification methods can be considered.

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## **6.1.1 Sample Selection**

Hardware and contaminant(s) must be representative and reasonably reflect worst case configuration/conditions.

## **6.1.2** Qualification Test for Alternate Verification Test Methods

- a. Prepare a set of non-flight test items of representative hardware and apply representative contaminants. Verify hardware cleanliness on 50% of test items, using the standard test method specified in section 4. An accepted aggressive NVR solvent such as ethyl acetate, cyclohexane, hexane, CFC-113, HCFC-225, or trichloroethylene shall be used for the baseline NVR test method.
- b. Verify hardware cleanliness of the balance of the contaminated test items using the proposed alternative verification test method.
- c. Statistically equate verification equivalence. The mean and variance of the proposed alternate process shall be provided. The contamination level results and variance of the proposed process shall be statistically equivalent to or less than the baseline process. Variance shall be determined from a minimum of seven (7) configuration tests.

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## APPENDIX A. ACRONYMS

COPV Composite Over-Wrapped Pressure Vessels

GO2 Gaseous Oxygen

GSE Ground Support Equipment

HEPA High Efficiency Particulate Air

LO2 Liquid Oxygen

MSDS Materials Safety Data Sheet

NVR Nonvolatile Residue

PFEP Polyfluoroethylenepropylene

PPM Parts Per Million

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#### APPENDIX B. DEFINITIONS

**Cleaning** - Removal of particulate and incompatible materials from significant surfaces of components and systems.

**Contaminant** - Any material that could chemically interact or mechanically interfere with the component or system function

**Corrosion** - The gradual destruction of a materials by chemical reaction with their environment or substances contacting the material.

**Crazing** (non-metallics) - The phenomenon that produces a network of fine cracks on the surface of a material.

**Embrittlement** – A significant decrease of ductility of a material, which makes the material brittle.

**Fiber** - A particle having a length to width ratio of 10 as a minimum i.e., length-to-width ratio of 10 - to 1 or greater.

**Hydrolysis** (non-metallics) - The chemical breakdown of a compound due to reaction with water.

**Inert Storage Package** - A barrier material used to encase a cleaned item to maintain item cleanliness level and which, when properly sealed, cannot introduce contaminants to the protected item.

**Integrity Seal** - Decal or other device applied to a package or closure in such a manner that it becomes visually damaged or destroyed when the package or closure is opened, providing evidence of tampering or unauthorized access.

**Leaching** - The process of a solute becoming detached or extracted by way of a solvent.

**Micron** - 0.001 millimeter

Milligram (mg) - 0.001 gram

**Nonvolatile Residue** (**NVR**) - The quantifiable substance remaining after filtration and controlled evaporation of final flush.

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**Particle** - A minute quantity of matter, metallic or non-metallic, with observable length, width and thickness.

**Procuring activity** - The first tier NASA customer of the user of this standard.

**pH** - A unit of measure from 0-14 representing acidity/alkalinity of an aqueous solution.

**Residue** – The material remaining after evaporation

**Reversion** (non-metallics) – The returning of a material to a previous state.

**Scale** – Surface oxidation in the form of partially adherent layers of corrosion products, left on metals by casting or heat treatment in air or other oxidizing atmosphere.

**Significant Surface** - All component surfaces that may come into contact with the respective service medium (wetted areas).

**Silting** - An accumulation of minute particles in the size range normally not counted but of sufficient quantity to interfere with sample analysis.

**Stress Corrosion Cracking** - The growth of crack formation in a corrosive environment.

**Visually Clean** - An article is classified visually clean when free of dirt, scale, oil, or other contamination when viewed at a distance of 0.6 to 1.2 meters (2 to 4 feet) under a minimum incident light level 500 lumens/meter<sup>2</sup> (50 foot-candles) with normal or corrected vision. A borescope is allowed to aid visual examination.

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#### APPENDIX C. NVR SWAB SAMPLING TEST PROCEDURE

Solvents used for this procedure shall meet NVR requirements of 2.0 mg per 500 ml, maximum, and material compatibility requirements as described in Section 3.2.6 Solvents should be selected based on their ability to solvate credible, expected non-volatile residues. The temperatures used for solvent drying, evaporation and distillation should be selected based on evaluation of the Safety Data Sheet (SDS) and manufacturer technical data sheets. The same solvent will normally used for swab sampling and swab extraction, but it is not a requirement.

**EQUIPMENT** 

<u>Item</u> <u>Description</u>

Oven Stainless steel lined, 50 °C, minimum capability

Analytical Balance Capable of 0.1 mg precision

**Tongs** 

Vacuum Source Capable of pulling 12.3 psig, for performing filtration

Vacuum Funnel

Vacuum Flask 1000 mL

Soxhlet Extractor 200 mL minimum volume

Wash Bottles 500 mL, compatible with sampling and extraction solvent

**Rotary Evaporator** 

Evaporating Flask 1000 mL, for Rotary Evaporator

Water heating bath For Rotary Evaporator

Beakers 500 mL, 1000 mL

Desiccator Two required

**MATERIALS** 

<u>Item</u> <u>Description</u>

Weighing Dish Disposable aluminum

Wipes Kim Wipes or equivalent

Filter Membranes 0.45 µm, compatible with sampling and extraction solvent

Aluminum Foil

Gloves Nitrile or as required by SDS

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Swabs Compatible with sampling and extraction solvent

#### **SOLVENTS**

ItemDescriptionEthanol200 proofAcetone> 99.7%Hexanes> 99.9%

Extraction and sampling NVR content less than 2.0 mg per 500 mL; distillation may be

required to achieve this NVR level

#### **INSTRUCTIONS**

To protect personnel, operations involving solvent should be conducted in a fume hood equipped with an exhaust blower capable of producing a minimum face velocity of 100 ft/min. The exhaust blower must be in continuous operation during solvent handling.

Adequate ventilation and personal protective equipment is required during swab sampling and processing. If adequate ventilation is not available, the use of a respirator with cartridges for organic vapors is highly recommended.

## DETERMINE NVR CONTENT OF SAMPLING AND EXTRACTION SOLVENT

- 1. Triple wash all surfaces that will contact the test solvent with 1) ethanol, 2) acetone, 3) hexanes, and 4) solvent that is known to meet the NVR requirement of 2.0 mg per 500 mL maximum, henceforth referred to as verified solvent.
- 2. After washing, dry each piece of equipment with dry nitrogen (GN<sub>2</sub>) or missile grade air (MGA).
- 3. Triple wash a new aluminum weighing dish with 1) ethanol, 2) acetone, 3) hexanes, and 4) verified solvent.
- 4. Place the weighing dish in an oven at 50 °C for a minimum of 1.5 hours.
- 5. Remove the weighing dish from the oven and place it in a desiccator. Allow the dish to cool for at least 30 minutes.
- 6. Filter 500 mL of the test solvent, or analyte, through a 0.45 µm filter into a clean vacuum flask.
- 7. Transfer the filtered analyte to a clean 1000 mL evaporating flask.
- 8. Rinse the vacuum flask with 10 mL verified solvent and pour the contents into the 1000 mL evaporating flask.

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- 9. Evaporate the filtered analyte using the rotary evaporator until approximately 10-20 mL remain.
- 10. Using a pair of clean tongs, remove a clean weighing dish from the desiccator and weigh it on the analytical balance. Record the weight.
- 11. Transfer the 10-20 mL sample from the 1000 mL evaporating flask to the weighing dish.
- 12. Rinse the 1000 mL evaporating flask with 10 mL verified solvent and pour the contents into the weighing dish.
- 13. Using a pair of clean tongs, place the weighing dish in an oven for a minimum of 1.5 hours at a temperature that is no more than 3 degrees C above the boiling point of the solvent.
- 14. Using a pair of clean tongs, remove the weighing dish from the oven and place into a second desiccator to cool for at least 30 minutes.
- 15. Weigh the dish again and record the weight.
- 16. Subtract the tare weight from the second weight then record the result as mg NVR per volume of solvent evaporated.

#### **SWAB PREPARATION**

- 1. All solvent used in this procedure shall meet the NVR requirement of 2.0 mg per 500 mL maximum, determined as described in the section above and henceforth referred to as verified solvent.
- 2. Triple wash all surfaces that will contact the samples with 1) ethanol, 2) acetone, 3) hexanes, and 4) verified solvent.
- 3. After washing, dry each piece of equipment with GN<sub>2</sub> or MGA.
- 4. Add 600 mL of verified solvent to a 1000 mL beaker. Place a swab in the solvent.
- 5. Bring the solvent to a boil for 10 minutes. Agitate the swab with a clean pair of tongs.
- 6. Remove the swab from the beaker and place it in a second beaker containing 600 mL of verified solvent.
- 7. Repeat the boiling process.
- 8. Transfer the swab into a Soxhlet extractor vessel. Add 200 mL of verified solvent and reflux for 2 hours.
- 9. Transfer the refluxed solvent to a clean beaker and analyze it for NVR content per the section above.
- 10. Repeat the extraction process until two identical results are obtained.

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11. Remove the swab from the Soxhlet extractor using a pair of clean tongs. Wrap the swab in clean aluminum foil.

#### **SWAB SAMPLE COLLECTION**

- 1. Using a pair of clean tongs, remove the clean, blanked swab from the aluminum foil wrapper. The swab should not be handled with ungloved hands.
- 2. Saturate the swab with solvent that is known to meet the NVR requirement of 2.0 mg per 500 mL maximum, henceforth referred to as verified solvent.
- 3. Swab the surface of interest, turning the swab often to expose a clean area to the surface. If possible, at least 3-ft<sup>2</sup> should be sampled.
- 4. Return the swab to the aluminum foil wrapper without drying the swab, and enclose it securely.
- 5. Record the sample area in square feet and label the swab package.

## SWAB SAMPLE ANALYSIS

- 1. Using a pair of clean tongs, place the swab in a Soxhlet extractor vessel.
- 2. Add 200 mL of solvent that is known to meet the NVR requirement of 2.0 mg per 500 mL maximum, henceforth referred to as verified solvent, to the bottom flask of the Soxhlet extractor.
- 3. Heat the bottom Soxhlet flask to refluxing temperature, and then allow the solvent to siphon at least 10 times.
- 4. Transfer the solvent to a clean beaker.
- 5. Filter the 200 mL of solvent through a 0.45 µm filter into a clean vacuum flask.
- 6. Transfer the sample to a clean 1000 mL evaporating flask.
- 7. Rinse the vacuum flask with 10 mL of verified solvent and pour the contents into the 1000 mL evaporating flask.
- 8. Evaporate the sample using a rotary evaporator until approximately 10-20 mL remain.
- 9. Using a pair of clean tongs, remove a clean weighing dish from the desiccator and weigh it on the analytical balance. Record the tare weight and sample identification.
- 10. Transfer the sample from the 1000 mL evaporating flask to the weighing dish.
- 11. Rinse the 1000 mL evaporating flask with 10 mL verified solvent and pour the contents into the weighing dish.

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- 12. Using a pair of clean tongs, place the weighing dish in an oven for a minimum of 1.5 hours at a temperature that is no more than 3 degrees C above the boiling point of the solvent.
- 13. Using a pair of clean tongs, remove the weighing dish from the oven and place into a second desiccator to cool for at least 30 minutes.
- 14. Weigh the dish again and record the weight.
- 15. Subtract the tare weight from the second weight, then subtract the weight of NVR present in the solvent used to process the sample. Report this result as mg NVR per surface area flushed.