



Standard Test Method for Gravimetric Determination of Nonvolatile Residue From Cleanroom Wipers¹

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ε¹ NOTE—Section 2.4 editorially corrected in January 2012.

1. Scope

1.1 This test method covers the determination of solvent extractable nonvolatile residue (NVR) from wipers used in assembly, cleaning, or testing of spacecraft, but not from those used for analytical surface sampling of hardware.

1.2 The NVR of interest is that which can be extracted from cleanroom wipers using a specified solvent that has been selected for its extractive qualities. Alternative solvents may be selected, but since their use may result in different values being generated, they must be identified in the procedure data sheet.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 *Exception*—The inch-pound units are included for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

E2217 Practice for Design and Construction of Aerospace Cleanrooms and Contamination Controlled Areas

F24 Test Method for Measuring and Counting Particulate Contamination on Surfaces

F50 Practice for Continuous Sizing and Counting of Airborne Particles in Dust-Controlled Areas and Clean Rooms Using Instruments Capable of Detecting Single Sub-Micrometre and Larger Particles

¹ This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 Military Standards:³

MIL-STD-1916 Acceptance on Zero Sampling Inspection

2.3 Federal Standards:³

Fed. Std. 209E Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones⁴

2.4 Other Documents:

IEST-RP-CC001 HEPA and ULPA Filters^{5,6}

IEST-RP-CC005 Gloves and Finger Cots Used in Cleanrooms and Other Controlled Environments⁶

ISO 14644-1 Cleanrooms and Associated Controlled Environments, Classification of air cleanliness⁷

ISO 14644-2 Cleanrooms and Associated Controlled Environments, Specifications for testing and monitoring to prove continued compliance with ISO 14644-1^{5,7}

3. Terminology

3.1 Definitions:

3.1.1 *contaminant*—unwanted molecular or particulate matter that could affect or degrade the performance of the components on which they are deposited.

3.1.2 *contamination*—a process of contaminant transport or accretion or both.

3.1.3 *environmentally controlled area*—cleanrooms, clean facilities, controlled work areas, and other enclosures that are designed to protect hardware from contamination. Cleanliness is achieved by controlling airborne particulate matter, temperature, relative humidity, materials, garments, and personnel activities. Guidelines for controlled areas can be found in Practice E2217.

3.1.4 *high efficiency particulate air (HEPA)*—a term describing filters having an efficiency of 99.97 % for removal of

³ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, <http://dodssp.daps.dla.mil>.

⁴ Fed-Std-209E has been replaced by ISO 14644-1 and -2, but may continue to be used by mutual agreement.

⁵ The use of Di Octyl Phthalate (DOP) in leak testing of filters or filter installation is not acceptable.

⁶ Available from Institute of Environmental Sciences and Technology (IEST), Arlington Place One, 2340 S. Arlington Heights Rd., Suite 100, Arlington Heights, IL 60005-4516, <http://www.iest.org>.

⁷ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

0.3- μm and larger particles. For this application, filters shall meet the requirements of **IEST-RP-CC001.4** and section **6.1** of this test method.

3.1.5 *molecular contaminant (nonparticulate)*—may be in a gaseous, liquid, or solid state. It may be uniformly or nonuniformly distributed or be in the form of droplets. Molecular contaminants account for most of the NVR.

3.1.6 *NVR*—that quantity of molecular matter remaining after the filtration of a solvent containing contaminants and evaporation of the solvent at a specified temperature.

3.1.7 *particle (particulate contaminant)*—a piece of matter in a solid state, with observable length, width, and thickness. The size of a particle is defined by its greatest dimension and is expressed in μm .

4. Summary of Test Method

4.1 A wiper to be tested is placed in a clean blanked container and a measured volume of solvent is added to the container.

4.2 The container is placed in a heated ultrasonic cleaner and agitated by ultrasonic action for a specified period of time and the wiper is removed from the container.

4.3 The solvent in the container is filtered into another clean container and allowed to evaporate to a low volume.

4.4 The solvent is transferred to a clean pre-weighed weighing dish and evaporated to constant weight.

4.5 The results are expressed in milligrams/0.1 square meters of wiper surface area or in mg/unit mass of wiper.

4.6 A control blank shall be run on all solvents, filtration components, and all other equipment associated with the analysis. In the event that more than one determination is run the same day, additional blanks will not be necessary, but will rely on the blank value from the first test.

4.7 NVR samples thus obtained will be saved for analysis to identify contaminant species if a more complete analysis is necessary.

5. Significance and Use

5.1 The NVR obtained by this test method is that amount which is available for release by wipers in normal use.

5.2 Evaporation of the solvent at the stated temperature is to quantify the NVR that can be expected to exist at room temperature, since the slight difference between room temperature and test temperature has not been shown to result in significant variances.

5.3 This test method may be more aggressive than necessary for the evaluation of wipers that will be restricted to dry use only.

5.4 Numerous other methods are being used to determine NVR. This test method is not intended to replace test methods used for other applications.

6. Apparatus and Materials

6.1 *Unidirectional airflow work station*, 100 % exhaust for handling solvents. Must meet the particulate air cleanliness Class 5 (100), or better in accordance with **ISO 14644-1** and **ISO 14644-2** (Fed-Std-209), latest revision when tested in accordance with Practice **F50**. HEPA filters in the work station

must not have been tested with Di-Octyl Phthalate (DOP) at any time. Temperature shall be controlled within a range of 20 to 25°C and relative humidity to less than 60 %.

6.2 *Analytical balance*, 0.01-mg readability, 0.1-mg precision. Capacity to be determined by user.

6.3 *Vacuum filtration system*, 25-mm diameter, consisting of a membrane filter funnel and vacuum pump that will provide a pressure of 250 Torr (20-in. Hg vac.). Other size filtration systems may be used as needed. All items that will come in contact with solvents during analysis shall be of glass, stainless steel, or other material that will not affect the analysis via induced contamination. Any house vacuum system may be used.

6.4 *Solvent resistant membrane filters*, Fluorocarbon, 25-mm diameter, 0.2- μm nominal pore size.⁸ The use of supported membrane filters is not recommended because of possible adverse effects of the solvent on the support media.

6.5 *Teflon-coated tweezers, or hemostat*, unserrated tips.

6.6 *Beakers*, low form glass, 500 ml.

6.7 *Laboratory detergent*, liquid.

6.8 *Methanol*, Reagent grade, A.C.S.

6.9 *Acetone*, Reagent grade, A.C.S.

6.10 *Deionized water*, organic free, Type II per Specification **D1193** with a minimum resistivity of 1.0 M Ω -cm.

6.11 *Gloves*,⁹ Barrier-type, low particle-generating, low out-gassing, per IEST RP-CC005.

6.12 *NVR solvent*, Must be verified to contain no more than 0.35-mg NVR per 300-mL solvent (0.12 mg per 100 mL) when tested in accordance with Section **8** of this test method.

NOTE 1—A solvent should be selected that is representative of the service conditions of the wiper. The solvent used must be recorded in the test report. Historically, a solvent consisting of three parts 1,1,1 trichloroethane and one part ethanol v/v has been used. 1,1,1 trichloroethane is an ozone depleting substance that is no longer available. Examples of solvents currently used are acetone, ethyl acetate, ethyl alcohol, isopropyl alcohol, hexane, and the azeotrope of ethyl acetate/cyclohexane. Ethyl alcohol and isopropyl alcohol are the least effective at removing NVR but are used when they reflect actual solvent usage in service.

NOTE 2—In the event that the solvent does not meet the required purity level, it may be necessary to triple distill it, keeping the temperature of the vapor phase of the distillate no more than 0.2°C higher than the boiling point of the solvent. Higher temperatures will result in the “carryover” of heavier fractions in the vapor phase, which will cause the solvent to fail the required purity tests.

6.13 *Ultrasonic tank*, 5.7-L capacity nominal, with heater capable of maintaining a temperature of 35 $\pm$ 2°C, and cover to position beakers in tank. Other sizes may be used.

⁸ Nuclepore Corp. PTFE Filinert membrane filter, P/N 130606, and Millipore Corp. Fluoropore filter, P/N 02500 have been found to be satisfactory. Other equivalent, solvent-resistant filters may be used. Larger diameter filters to fit larger filter assemblies are acceptable.

The sole source of supply of the apparatus known to the committee at this time is Nuclepore Corp. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁹ Gloves are necessary to protect the analyst from exposure to NVR solvent and to minimize the possibility of introducing any artifacts from the analyst into the sample. Must be resistant to the solvent used and provide a firm grip on items being grasped by the gloves.



6.14 *Evaporating dishes*, aluminum foil, 43-mm diameter, or acceptable equivalent.

6.15 *Drying oven (desiccator)*, cleanroom compatible, stainless steel interior.

7. Preparation of Equipment

7.1 All operations shall be performed in a work station in accordance with section 6.1.

7.2 Wash all glassware, filter funnels, weighing dishes, and associated tools in a 3 % solution of liquid detergent in deionized water. Rinse with deionized water for a period of 1 minute, followed by rinsing with acetone or methanol, then with the NVR solvent described in section 6.10. Dry in a cleaned oven for 1 h at 35°C, remove, and store in a desiccator until used.

7.3 All items, such as glassware, funnels, and so forth, that will come in contact with the NVR solvent during analysis, will be blanked in accordance with Section 8 of this test method before use.

8. NVR and System Blank

8.1 The NVR of the solvent, and all glassware or other items that will come in contact with the solvent during the analysis, shall be determined before use. The only exception is when several tests are to be run consecutively, in which case, the blank only needs to be determined once for a batch. It must be remembered that this solvent will absorb moisture from the atmosphere every time the storage container is opened, so large quantities should not be processed at one time.

8.1.1 Pour 300 mL of solvent into a 500-mL beaker cleaned per 7.2.

8.1.2 Perform analysis per Section 9.

8.1.3 The NVR blank shall be less than 0.35 mg per 300 mL.

8.1.4 Record the results of the blank analysis on the test report form.

8.1.5 Solvents that do not meet the NVR requirements may be redistilled and retested.

8.1.6 Only verified clean, non-contaminating metals, glass, or fluorocarbon containers are acceptable for storage of blanked solvent.

9. Procedure

9.1 All operations shall be performed in a work station in accordance with section 6.1.

9.2 Assemble the filtration assembly according to manufacturer's instructions.

9.3 When reporting results on the basis of mg per unit area, place enough wipers to provide a combined surface area of 0.1 m², minimum, in a precleaned 500-mL beaker.¹⁰ For example, a wiper measuring 6 by 6 in. will have a surface area of 36 in.², thus requiring four wipers to provide the necessary surface area. Do not cut wipers to simplify calculations or for any other purpose. The wipers must be tested as received from the supplier. (No attempt shall be made to determine the surface

area of each individual fiber in a woven product.) When reporting results on the basis of mg per unit mass, weigh a sample of the wipers to be tested (similar to above sample size) to an accuracy of 0.1 mg. Place in beaker as above.

9.4 Add 300-mL blanked NVR solvent to beaker. Cover beaker with a watchglass to minimize contamination of sample by fallout.

NOTE 3—Wipers larger than 0.1 m² will require 500 mL of solvent to ensure complete wetting of the sample.

9.5 Place beaker in ultrasonic tank that has been filled with fluid heated to 35 ± 2°C and install tank cover to position the beaker in the tank. Typically, the fluid used is D.I. water.

9.6 Agitate for 30 min. This agitation is necessary to ensure that all available NVR is contacted by solvent and removed from the wiper being tested.

9.7 Remove beaker from tank and extract wiper(s) using precleaned tongs. Hold wipers over the beaker until dripping ceases. Place damp wiper on a tray or rack to dry. When the item is dry, as determined by a lack of solvent odors, it will be placed in a clean Nylon plastic envelope, and saved for future reference, or discarded at the option of the analyst. No further analysis will be performed on this sample.

9.8 Filter the solvent into a precleaned and blanked beaker that has been installed in the filtration assembly.¹¹

9.9 When all the solvent has been filtered, rinse the filter assembly with solvent (exact volume of rinse is not critical). The filter membrane may be removed from the filter assembly and analyzed in accordance with Appendix X1 to determine particle release from the wipers.

9.10 Place the beaker in HEPA-filtered airflow at ambient temperature. Position the beaker near or directly under the airflow. Allow to evaporate to approximately 10 mL. It may be necessary to cover the beaker partially with a watchglass to prevent introduction of extraneous material during evaporation.

9.11 Transfer solvent to a clean, preweighed weighing dish. Rinse beaker with 10- to 20-mL volumes of solvent and add to weighing dish. This shall be repeated three times. Total rinse volume shall not exceed 50 mL.

9.12 Allow the solvent in the weighing dish to evaporate in the laminar airflow bench until no visible solvent remains.

9.13 Place the weighing dish in the drying oven at 35 ± 2°C for 30 min.

9.14 Remove dish from oven, protect from contamination, and allow to equilibrate to room conditions.

9.15 Weigh the dish and contents. Record weight in log book.

9.16 Return dish to oven for 30 min.

9.17 Reweigh dish. Continue equilibrating and reweighing until readings have stabilized (repeat weights that do not vary by more than 0.1 mg). Record the results in the test report form.

9.18 Retain NVR if further analysis is necessary to identify contaminants.

¹⁰ It is assumed that selection of samples from a shipment has been made in accordance with MIL-STD-1916 to provide a truly representative sample.

¹¹ The filter membrane used to filter the sample before evaporation may be used to determine particle counts on wipers. If this is planned, a 47-mm membrane and filtration apparatus should be used.



9.19 Calculate as shown below and record results on the test report form.

10. Calculation of NVR

10.1 NVR in mg per 0.1 m²

$$\text{NVR} = \frac{A - (B + C)}{D} \quad (1)$$

where:

A = mass of sample dish and residue, mg (from section 9.17);

B = mass of weighing dish, mg (from 8.1.4);

C = mass of blank of solvent, mg; and

D = surface area of wiper, in 0.1 m².

10.2 NVR in mg per unit mass

$$\text{NVR} = \frac{E - (F + G)}{H} \quad (2)$$

where:

E = mass of sample and weighing dish, mg (from section 9.17);

F = mass of weighing dish, mg (from 8.1.4);

G = mass of blank of solvent, mg; and

H = mass of wiper.

11. Reporting Results

11.1 The report shall use the Test Reporting Form shown in Fig. 1.

11.2 The estimated accuracy of the NVR determination shall be noted. This is based on the accuracy and precision of the balance, NVR background from the solvent and blank samples, and any other factors observed during the test operations. Explanation, if required, shall be included under Comments.

11.3 Other comments might include observations made such as condition of packaging, evidence of improper handling, color of the solvent rinse, and any anomalies that may have occurred during analysis.

12. Precision and Bias

12.1 Precision and bias have not yet been determined.

13. Keywords

13.1 cleanroom wipers; non-volatile residue; NVR; wipers

TEST REPORTING FORM
NVR DATA SHEET

SAMPLE IDENTIFICATION			
MATERIAL TYPE			
SIZE, COLOR, ETC.			
SPECIFICATION			
DATE RECEIVED		DATE TESTED	
REQUESTER	DEPT	TEL NO	
TYPE OF SOLVENT USED			

RESULTS PER UNIT AREA:

MASS OF DISH AND RESIDUE	(A)		MG
MASS OF WEIGHING DISH	(B)		MG
BLANK OF SOLVENT	(C)		MG
SURFACE AREA	(D)		MG
NVR EXTRACTED FROM SAMPLE TO SPEC			MG/0.1 m ²
	YES		
	NO		

RESULTS PER UNIT MASS:

MASS OF DISH AND RESIDUE	(E)		MG
MASS OF WEIGHING DISH	(F)		MG
MASS OF SOLVENT	(G)		MG
MASS OF WIPER	(H)		MG
NVR EXTRACTED FROM WIPER TO SPEC			MG/GRAM
	YES		
	NO		

COMMENTS (use additional sheets if necessary)

ANALYZED BY

PRINT NAME

SIGNATURE

FIG. 1 Test Reporting Form—NVR Data Sheet

APPENDIX

(Nonmandatory Information)

X1. PARTICLE COUNTS ON SAMPLES FROM CLEANROOM WIPERS

X1.1 The appendix lists the parameters for performing particle counts on samples collected from cleanroom wipers, as an adjunct to the analysis for nonvolatile residue.

X1.2 *Equipment Needed:*

X1.2.1 *Microscope*, compound binocular, brightfield illumination, numerical aperture of 0.65 or better at 40×. Capable of providing total magnification of 40 and 100× (total = objective lens × body tube lens × eyepiece).

X1.2.2 *Ocular micrometer*, 5 mm long, divided into five major units, with each major unit being subdivided into ten subunits. To be installed in the eyepiece of the microscope.

X1.2.3 *Stage micrometer*, 2 mm long marked at 0.1- and 0.01-mm intervals. (To be used for calibration of ocular micrometer.)

X1.2.4 *Mechanical counter*, five sections, resettable.

X1.2.5 *Plastic Petri dishes*, Petrislides Millipore Corp. or equivalent.

X1.3 In practice, the membrane used to filter the solvent is removed from the filter apparatus and placed in a “Petrislide” for analysis. The membrane is analyzed using microscope techniques, in accordance with Test Method F24 to size and count particles removed from the wiper during sample collection. Particles are counted in five size ranges:

5 to 10 μm
10 to 25 μm
25 to 50 μm
50 to 100 μm
>100 μm

X1.4 Results are reported on the basis of counts/0.1 m².

X1.5 In the event that the number of particles on the membrane is too excessive to count, a separate sample must be run, and a 30-mL or larger aliquot of the extraction solvent is filtered to perform particle counts. Test results must be corrected to compensate for the size of the aliquot used.



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