Designation: E 1560 – 95 (Reapproved 2001)

Standard Test Method for Gravimetric Determination of Nonvolatile Residue From Cleanroom Wipers[1]

This standard is issued under the fixed designation E 1560; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1.3 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.

1.4 *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:*

D 1193 Specification for Reagent Water[2]

F 24 Test Method for Measuring and Counting Particulate Contamination on Surfaces[3]

F 50 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[3]

2.2 *Military Standards:*

Air Force T.O. 00-25-203 Contamination Control of Aerospace Facilities, U.S. Air Force[4]

MIL-F-51068F Filters, Particulate (High Efficiency, Fire Resistant)[4,5]

MIL-P-27401 Propellant, Pressurizing Agent, Nitrogen[4]

MIL–STD 105D Sampling Procedures and Tables for Inspection by Attributes[4]

MIL-STD-1246B Product Cleanliness Levels and Contamination Control Program[4]

2.3 *Federal Standards:*

Fed Spec. O-E-00760 Ethyl Alcohol[4]

Fed Spec. O-T-620 1,1,1-Trichloroethane, Technical, Inhibited (Methyl Chloroform)[4]

Fed. Std. 209E Airborne Particulate Classes for Cleanrooms and Clean Zones[4]

2.4 *Other Documents:*

Industrial Ventilation, A Manual of Recommended Practice, Latest Edition[6]

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 Table 3-1 of Air Force T.O. 00-25-203.

3.1.4 *high efficiency particulate air (HEPA)*—a term describing filters having an efficiency of 99.97 % for removal of 0.3-μm and larger particles. For this application, filters shall meet the requirements of 2.3 and 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

[1] This specification 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] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 15.03.

[4] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, Attn: NPODS.

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

[6] Available from Committee on Industrial Ventilation, American Conference of Governmental Industrial Hygienists, 1330 Kemper Meadow Dr., Suite 600, Cincinnati, OH 45240.

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 dimensions 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 preweighed weighing dish and evaporated to constant weight.

4.5 The results are expressed in milligrams/0.1 square metres 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 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 M3.5 (100), or better in accordance with Federal Standard 209, latest revision. 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 50 %.

6.2 *Solvent*, 1,1,1-Trichloroethane per 2.10.

6.3 *Solvent*, Ethanol, per 2.11.

6.4 *Analytical balance*, 0.1-mg readability, 0.1-mg precision.[7] Capacity to be determined by user.

6.5 *Vacuum filtration system*, 25-mm diameter, consisting of a membrane filter funnel[8] and vacuum pump that will provide a pressure of 250 Torr (20-in. Hg vac.). See Fig. 1. Other size filters 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 contaminants.

6.6 *Solvent resistant membrane filters*, Fluorocarbon, 25-mm diameter, 0.2-µm nominal pore size.[9] The use of supported membrane filters is not recommended because of possible adverse effects of the solvent on the support media.

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

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

6.9 *Laboratory detergent*, liquid.[10]

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

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

6.12 *Deionized water*, organic free, Type II per Specification D 1193 with a minimum resistivity of 1.0 MΩ/cm.

6.13 *Gloves*,[11] Barrier-type, low particle-generating, low outgassing, per I.E.S. Recommended Practice RP-CC-005-88.

6.14 *NVR solvent*, consisting of three parts 1,1,1-Trichloroethane and one part ethanol v/v. 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—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.

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

6.16 *Evaporating dishes*, aluminum foil, 43-mm diameter.[12]

6.17 *Drying oven*, [13] cleanroom compatible, stainless steel interior.

7. Preparation of Equipment

7.1 All operations shall be performed in a work station per 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

[7] Analytical Balance, Mettler Model AE 240, Sartorius Model 2405, Sartorius Model 2434, and Cahn Model TA 4100 or similar units are acceptable.

[8] Vacuum System, Fisher Scientific, P/N 09-750 is acceptable. Other makes and models may be used.

[9] 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.

[10] A 3 % solution of Micro-clean, Liqui-nox, Joy, or similar products is acceptable.

[11] 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 solvent resistant and provide a firm grip on items being grasped by the gloves.

[12] Evaporating dishes, VWR Cat. No. 25433-008 are satisfactory. Other equivalent brands may be used.

[13] Drying oven, stainless steel interior, Blue M Corp. Model OV-8A or equivalent is acceptable.

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 _____ PER 6.14 _____ OTHER _____

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		_____	MG/0.1 m^2
TO SPEC	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		_____	MG/GRAM
TO SPEC	YES	_____	
	NO	_____	

COMMENTS (use additional sheets if necessary) _____

ANALYZED BY _____
PRINT NAME _____
SIGNATURE _____

FIG. 1 Test Reporting Form—NVR Data Sheet

min, followed by rinsing with acetone or methanol, then with the NVR solvent described in 6.12. 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 per 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 NVR blank shall be less than 0.35 mg per 300 mL.

8.1.4 Record results of blank analysis in laboratory log book.

8.1.5 Solvents that do not meet the NVR requirements shall either be redistilled and retested or can be appropriately marked and set aside for other uses, including cleaning purposes.

8.1.6 Only verified clean, noncontaminating 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 per 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 m^2, minimum, in a precleaned 500-mL beaker.[14] 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 2—Wipers larger than 0.1 m^2 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.

[14] It is assumed that selection of samples from a shipment has been made in accordance with MIL STD 105 to provide a truly representative sample.

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.[15]

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 to evaporate in the laminar airflow bench until no visible solvent remains.

9.13 Place the weighing dish in 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 desiccator 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).

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

9.19 Record test results in log book.

10. Calculation of NVR

10.1 NVR in mg per 0.1 m^2

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

where:
A = mass of sample dish and residue, mg;
B = mass of weighing dish, mg;
C = mass of solvent, mg; and
D = surface area of wiper, in 0.1 m^2.

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;
F = mass of weighing dish, mg;
G = mass 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.

[15] 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 should be used.

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 F 24 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 m^2.

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.