



Designation: E1560 – 11^{ε1} E1560 – 18

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

This standard is issued under the fixed designation E1560; 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.

^{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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

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

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.

Current edition approved Dec. 1, 2011; Nov. 1, 2018. Published January 2012; November 2018. Originally approved in 1993. Last previous edition approved in 2006; 2011 as E1560 – 06; E1560 – 11^{ε1}. DOI: 10.1520/E1560-11.10.1520/E1560-18.

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.

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.

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 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- μ m nominal pore size. 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*,⁸Barrier-type, low particle-generating, low outgassing, 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 $35 \pm 2^\circ\text{C}$, and cover to position beakers in tank. Other sizes may be used.

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.⁹ For example, a wiper measuring 6 by 6 in. will have a surface area of 36 in.², thus

⁸ 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.

⁹ 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.