



Designation: E 1560 – 95 (Reapproved 2001)

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

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 Water²
- F 24 Test Method for Measuring and Counting Particulate Contamination on Surfaces³
- 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 Particles³

2.2 Military Standards:

- Air Force T.O. 00-25-203 Contamination Control of Aerospace Facilities, U.S. Air Force⁴

MIL-F-51068F Filters, Particulate (High Efficiency, Fire Resistant)^{4,5}

MIL-P-27401 Propellant, Pressurizing Agent, Nitrogen⁴

MIL-STD 105D Sampling Procedures and Tables for Inspection by Attributes⁴

MIL-STD-1246B Product Cleanliness Levels and Contamination Control Program⁴

2.3 Federal Standards:

Fed Spec. O-E-00760 Ethyl Alcohol⁴

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

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

2.4 Other Documents:

Industrial Ventilation, A Manual of Recommended Practice, Latest Edition⁶

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.

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.

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 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 funnel⁸ 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 M Ω /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 $35 \pm 2^\circ\text{C}$, 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 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.