Designation: E 1731 – 95ε1

Standard Test Method for Gravimetric Determination of Nonvolatile Residue from Cleanroom Gloves[1]

This standard is issued under the fixed designation E 1731; 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.

ε[1] NOTE—To bring Subcommittee E21.05's existing standards into compliance with Part H of ASTM's Form and Style Manual, the M designation has been editorially removed in July 2000.

1. Scope

1.1 This test method covers the determination of solvent extractable nonvolatile residue (NVR) from gloves used in cleanrooms where spacecraft are assembled, cleaned, or tested.

1.2 The values stated in SI units are to be regarded as standard.

1.3 The NVR of interest is that which can be extracted from gloves using a specified solvent that has been selected for its extracting qualities, or because it is representative of solvents used in the particular facility. Alternative solvents may be used, 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 Cleanrooms Using Instruments Capable of Detecting Single Sub-Micrometre and Larger Particles[4]

G 120 Test Method for Determination of Soluble Residual Contamination in Materials and Components by Soxhlet Extraction[5]

2.2 *Military Standards[6]:*

Air Force T.O. 00-25-203 Contamination Control of Aerospace Facilities

Mil-F-51068F Filters, Particulate (High Efficiency, Fire Resistant)

Mil-P-27401 Propellant, Pressurizing Agent, Nitrogen

Mil-Std-105D Sampling Procedures and Tables for Inspection by Attributes

Mil-Std-1246B Product Cleanliness Levels and Contamination Control Program

2.3 *Federal Standards[6]:*

Fed Spec O-E-00760 Ethyl Alcohol

Fed Std 209E Airborne Particulate Classes for Cleanrooms and Clean Zones

2.4 *Other Documents:*

IES-RP-CC005.2 Gloves and Finger Cots Used in Cleanrooms and Other Controlled Environments

Industrial Ventilation, A Manual of Recommended Practice

3. Terminology

3.1 *Definitions:*

3.1.1 *contamination, n*—unwanted molecular or particulate matter that could affect or degrade the performance of the components upon which they are deposited.

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

3.1.3 *environmentally controlled area, n*—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 Air Force T.O. 00-25-203 Table 3-1.

3.1.4 *high efficiency particulate air (HEPA), n*—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), n*—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.

[1] This test method is under the jurisdiction of ASTM Committee E-21 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] Discontinued. See *1992 Annual Book of ASTM Standards*, Vol 10.05.

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

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

3.1.6 *NVR, n*—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), n*—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 micrometres.

4. Summary of Test Method

4.1 A glove to be tested is cut into several standard-sized pieces. The pieces are placed in a clean blanked container and a measured volume of solvent is added to the container. (See Note 1.)

4.2 The container is placed in a heated ultrasonic cleaner, or a heated water bath, and heated (and agitated if in an ultrasonic bath) for a specific length of time, after which the pieces of glove are 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 a constant weight.

4.5 The results are expressed in mg/sq cm of glove surface area or in mg/unit mass of glove sections.

4.6 A controlled 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 value from the first test.

4.7 NVR samples thus obtained may be used for analysis such as IR or FTIR if required.

NOTE 1—Some cleanroom gloves are of a coated or layered construction or have different textures applied to the inside and outside surfaces. Because the inside and outside surfaces of these gloves may release different quantities of nonvolatile residue, results using this method may not reflect the actual potential or transfer of contamination from this type of glove to hardware surfaces.

5. Significance and Use

5.1 The NVR obtained by this test method is that amount which is available for release by the gloves onto handled surfaces.

5.2 Evaporation of 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 the test temperature is not likely to result in significant variances.

5.3 Various other methods exist for determining NVR, for example Test Method G 120 and IES-RP-CC005.2. This test is not intended to replace test methods used for other purposes.

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 Fed-Std-209. HEPA filters in the work station must not have been tested with Di-Octly Phthlate (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 *Solvent*, Acetone.

6.3 *Solvent*, Ethanol.

6.4 *Analytical Balance*, 0.01-mg readability, 0.1-mg precision. Capacity to be determined by the user.

6.5 *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 filters may be used as needed. All items that will come in contact with solvents during analysis shall be made of glass, stainless steel, or other materials that will not affect the analysis via induced contamination. Any house vacuum system may be used.

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

6.7 *Teflon-Coated Tweezers, or Hemostat*, unserrated tips.

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

6.9 *Laboratory Detergent*, liquid.

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 megohm-cm.

6.13 *Gloves*, barrier type, low particle-generating, low outgassing, per IES-RP-CC005.2.

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

NOTE 2—Other solvents may be used if they are more representative of service conditions, but the actual solvent used must be reported per Section 11 of this test method.

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 convenient sizes may be used.

6.16 *Evaporating Dishes*, aluminum foil, 43-mm diameter.

6.17 *Drying Oven*, stainless steel interior.

7. Preparation of Equipment

7.1 All operation shall be performed in the work station per 6.1.

7.2 Wash all glassware, filter funnels, weighing dishes, and the associated tools (see Note 3). Rinse with deionized water for a period of 1 min followed by rinsing with acetone or methanol, then with acetone (or other NVR solvent) as described in 6.14. Dry in a cleaned oven for 1 h at 35 to 40°C, remove and store in a dessicator 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.

NOTE 3—A 3 % solution of liquid detergent in deionized water has been found to be effective.

8. NVR and System Blank

8.1 The NVR of the solvent, and all glassware and 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 may absorb moisture from the atmosphere, so it should be kept covered and small quantities 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 system blank shall be less than 0.35 mg/300 mL.

8.1.4 Record results of blank analysis in log book.

8.1.5 Solvents that do not meet the NVR requirements shall either be redistilled and retested or 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 filtration assembly according to manufacturer's instructions.

9.3 Cut and place in a beaker at least two sections 5 by 5 cm from each glove, preferably from the palm and the back of the glove. Cut up at least two gloves, or enough surface area for 0.2 m, minimum, counting both sides of the glove, in a precleaned 500-mL beaker. Test the gloves as received from the supplier. When reporting results on the basis of mg/unit mass, weigh a sample of cut sections of gloves to an accuracy of 0.01 mg. Place in 500-mL beaker as above.

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

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

9.6 Ultrasonic agitate for 15 min. This agitation is necessary to assure that all available NVR is contacted by the solvent and removed from the glove segments being tested.

9.7 Remove beaker from tank and extract glove sections using precleaned tongs. Hold the glove segments over the beaker until dripping ceases. Place the damp glove segments on a tray or rack to dry. When they are dry, as determined by lack of solvent odors, either discard, or store in a clean nylon bag, at the option of the analyst. No further analysis is performed on these samples.

9.8 Place the vessel in HEPA-filtered airflow at ambient temperature. Position the beaker near or directly under the airflow. Allow evaporation to approximately 10 mL. It may be necessary to cover the vessel partly with a watchglass to protect against fallout during evaporation.

9.9 Transfer solvent to a clean, preweighed weighing dish. Rinse beaker with 10 mL of solvent and add the wash solvent to the weighing dish. Repeat this three times. Total rinse volume shall not exceed 30 mL.

9.10 Allow to evaporate in the laminar flow bench until no visible solvent remains.

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

9.12 Remove the dish from the oven, protect contents from contamination, and allow to equilibrate to room ambient conditions.

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

9.14 Return dish to dessicator for 30 min.

9.15 Reweigh the dish. Continue equilibrating and reweighing until weight stabilizes (weights vary by 0.1 mg or less).

9.16 Retain NVR if further analysis as by infrared (IR) spectroscopy is necessary to identify the contaminants.

9.17 Record results in log book.

10. Calculation of NVR

10.1 NVR in mg/0.1 m^2.

$$NVR = A^*(-)\frac{20^*(B + C)}{D} \quad (1)$$

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 glove sections in m^2.

10.2 NVR in mg/unit mass

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

where:

E = mass of sample and weighing dish, mg;
F = mass of weighing dish, mg;
G = mass of solvent, mg; and
H = mass of glove sections.

11. Reporting Results

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

11.2 The estimated accuracy of the NVR determination shall be noted. This is based upon 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.

12. Precision and Bias

12.1 Precision and bias have not yet been determined.

13. Additional Tests

13.1 In addition to NVR, gloves may be tested for particle counts by either IES-RP-CC005.2 or by optical measurement of particles captured on the membrane filter when the solvent is filtered per 9.8 and 9.9.

13.2 NVR transfer from gloves to other surfaces may be determined by cutting samples from the fingers measuring 10 × 10 cm total. Place these sections between two clean pieces of aluminum foil or precleaned polished aluminum plates. Apply a load of 1000 kg or 10 kg/cm^2. Hold the pressure for 1 h minimum, 90 min maximum. Release the pressure. Wash the sides of the aluminum foil that were in contact with the glove sections with acetone. Use 25 cm^3 of solvent for each rinsing, collecting the solution in a clean 500-mL beaker. Rinse at least three times but no more than five times. Evaporate the solvent per Section 9. Weigh the solvent residue per 9.11-9.17. Analyze the solvent by IR infrared spectroscopy or FTIR to

NVR Reporting Form - Data Sheet

Sample identification _____
Glove Description, type _____
Size, color, other features _____
Date Received _____ Date Tested _____
Requester _____ Charge No. _____
Solvent: Acetone___ Ethanol___ Other_____

Results per unit area:			Results per unit mass	
Mass of dish + solvent	(A)	_____	(E)	_____
Mass of weighing dish	(B)	_____	(F)	_____
Blank of solvent	(C)	_____	(G)	_____
NVR extracted from sample	(D)	_____	(H)	_____

Normalized results NVR/unit surface area _____

Comments:_____
_____

Analyzed by _____ Date _____

FIG. 1 Test Reporting Form

determine the nature of any contaminants. Calculate NVR per Section 10 and report results per Section 11.