Designation: E1235 – 12

Standard Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft[1]

This standard is issued under the fixed designation E1235; 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 nonvolatile residue (NVR) fallout in environmentally controlled areas used for the assembly, testing, and processing of spacecraft.

1.2 The NVR of interest is that which is deposited on sampling plate surfaces at room temperature: it is left to the user to infer the relationship between the NVR found on the sampling plate surface and that found on any other surfaces.

1.3 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E1234 Practice for Handling, Transporting, and Installing Nonvolatile Residue (NVR) Sample Plates Used in Environmentally Controlled Areas for Spacecraft

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 *ISO Standards:*[3]

14644-1 Cleanrooms and Associated Controlled Environments—Part 1: Classification of Air Cleanliness

14644-2 Cleanrooms and Associated Controlled Environments—Part 2: Specifications for Testing and Monitoring to Prove Continued Compliance with ISO 14644-1

14951-3 Space Systems—Fluid Characteristics—Part 3: Nitrogen

2.3 *U.S. Federal Standard:*[4]

FED-STD-209E Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones

2.4 *Institute of Environmental Sciences and Technology:*[5]

IEST-RP-CC001.3 HEPA and ULPA Filters

IEST-RP-CC007.1 Testing ULPA Filters

IEST-RP-CC034.1 HEPA and ULPA Filter Leak Tests

IEST-STD-CC1246 Product Cleanliness Levels and Contamination Control Program

2.5 *American National Standards Institute:*[3]

ANSI/ASME B46.1-2009 Surface Texture (Surface Roughness, Waviness, and Lay)

2.6 *Other:*

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

SMC-TR-95-28, *Nonvolatile Residue Solvent Replacement*, U.S. Air Force Space and Missile Systems Center, 1 March 1995[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *ISO Class N (airborne particulate cleanliness class), n*—level of airborne particulate concentrations as defined in ISO 14644-1 and 14644-2, where 10^N is the maximum allowable concentration (particles/m^3).

3.1.1.1 *Discussion*—The considered particle sizes (lower threshold values) applicable for classification with ISO 14644-1 are limited to the range from 0.1 through 5 µm. Particles larger than 5 µm (macroparticles) may be expressed in accordance with Annex E of ISO 14644-1.

[1] This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the 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.

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

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

[5] Available from Institute of Environmental Sciences, 940 E. Northwest Highway, Mount Prospect, IL 60056.

[6] Available from Committee on Industrial Ventilation, PO Box 16153, Lansing, MI 48901.

3.1.2 *FS209 class, n*—the level of cleanliness specified by the maximum allowable number of particles/ft^3 of air as defined in FED-STD-209E.

3.1.2.1 *Discussion*—This is provided for information and to facilitate the transition to the use of the ISO classification standard (ISO 14644-1).

3.1.3 *bumping, n*—uneven boiling of a liquid caused by irregular rapid escape of large bubbles of highly volatile components as the liquid mixture is heated or exposed to vacuum.

3.1.4 *clean area, n*—a general term that includes cleanrooms, controlled areas, good housekeeping areas, and other areas that have contamination control by physical design and specified operating procedures.

3.1.5 *clean zone, n*—a defined space in which the contamination is controlled to meet specified cleanliness levels.

3.1.5.1 *Discussion*—The clean zone may be open or enclosed and may or may not be located within a cleanroom.

3.1.6 *contaminant, n*—unwanted molecular and particulate matter that could affect or degrade the performance of the components upon which they reside.

3.1.7 *contamination, n*—a process of contaminating.

3.1.8 *controlled area, n*—an environmentally controlled area, operated as a cleanroom, with two prefilter stages but without the final stage of HEPA (or better) filters used in cleanrooms.

3.1.8.1 *Discussion*—Only rough filters (50 to 60 % efficiency) and medium efficiency filters (80 to 85 % efficiency) are required for a controlled area. The maximum allowable airborne particle concentrations are ISO Class 8.5 (FS209 Class 283 000) area for particles 0.5 µm and ISO Class 8 (FS209 Class 100 000) for particles 5.0 µm.

3.1.9 *environmentally controlled areas, n*—a general term that includes cleanrooms, controlled areas, good housekeeping areas, and other enclosures that are designed to provide an environment suitable for people or products.

3.1.9.1 *Discussion*—The environmental components that are controlled include, but are not be limited to, air purity, temperature, humidity, materials, garments, and personnel activities.

3.1.10 *facility (clean facility), n*—the total real property required to accomplish the cleanroom functions.

3.1.10.1 *Discussion*—In addition to the cleanroom and associated clean areas, this includes utility rooms, storage areas, offices, lockers, washrooms, and other areas that do not necessarily require precise environmental control.

3.1.11 *good housekeeping area, n*—an environmentally controlled area without quantitative cleanliness requirements but maintained in a visibly clean condition.

3.1.11.1 *Discussion*—Office, laboratory, and storage areas with air conditioning and janitorial service are typical of good housekeeping areas.

3.1.12 *HEPA (high efficiency particulate air) filter, n*—a filter for air with a removal efficiency in excess of 99.97 % for 0.3-µm particles.

3.1.12.1 *Discussion*—For this application, HEPA filters shall meet the requirements of IEST-RP-CC001.3, IEST-RP-CC007.1, IEST-RP-CC034.1, and 6.4 of this test method.

3.1.13 *molecular contaminant— nonparticulate contaminant, n*—nonparticulate matter.

3.1.13.1 *Discussion*—The molecular contaminant 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.

3.1.14 *NVR (nonvolatile residue), n*—quantity of residual soluble, suspended, and particulate matter remaining after the controlled evaporation of a volatile liquid at a specified temperature.

3.1.14.1 *Discussion*—The liquid is usually filtered through a membrane filter, of a specified size, before evaporation to control the sizes of particles in the NVR. The process used to determine the NVR may affect the quantitative measurement. Process factors include filter size, solvent, and the evaporation temperature and atmosphere. For this reason, the process must be defined as it is in this test method.

3.1.15 *particle (particulate contaminant), n*—a piece of matter in a solid or liquid (droplet) state with observable length, width, and thickness.

3.1.16 *particle size, n*—(*1*) the apparent maximum linear dimension of a particle in the plane of observation, as observed with an optical microscope; (*2*) the equivalent diameter of a particle detected by automatic instrumentation. The equivalent diameter is the diameter of a reference sphere having known properties and producing the same response in the sensing instrument as the particle being measured; (*3*) the diameter of a circle having the same area as the projected area of a particle, in the plane of observation, observed by image analysis; and (*4*) the size defined by the measurement technique and calibration procedure.

3.1.16.1 *Discussion*—Because the particle size is defined by the measurement method, the measurement method and size definition should be stated when specifying or describing particle size.

3.1.17 *azeotropic mixture, n*—a solution of two or more liquids, the composition of which does not change upon distillation. Also known as *azeotrope*.

4. Summary of Test Method

4.1 A stainless steel plate is exposed within an environmentally controlled area for a known time. It is handled and transported in accordance with Practice E1234.

4.2 The plate is rinsed with a high purity solvent.

4.3 The solvent is filtered into a beaker, transferred to a preweighed container, and evaporated at or near room temperature, with a final drying at 35°C for 30 min. Alternative evaporation methods are included.

4.4 The NVR sample is weighed after it has equilibrated to room temperature and humidity conditions.

4.5 A blank stainless steel NVR plate is concurrently treated identically to each group of samples to determine solvent background and handling effects.

4.6 A reagent blank for each group of samples is determined.

4.7 Each NVR sample, 0.5 mg or greater, is retained for organic analysis by infrared spectrometry, or other techniques, to identify contaminants.

5. Significance and Use

5.1 The NVR determined by this test method is that amount that can reasonably be expected to exist on hardware exposed in environmentally controlled areas.

5.2 The evaporation of the solvent at or near room temperature is to quantify the NVR that exists at room temperature.

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

6. Apparatus and Materials

6.1 *Analytical Microbalance,* semimicro 5 place, with 30 g or greater tare, no greater than 0.01-mg readability, and ±0.01-mg precision.[7]

6.2 *HEPA Filtered,* ISO Class 5 (FS209 Class 100), or better environment, as defined in ISO 14644-1, unidirectional airflow, clean workstation.

6.3 *HEPA Filtered,* ISO Class 5 (FS209 Class 100), or better environment, as defined in ISO 14644-1, unidirectional air flow, exhausting work station, with 100 % exhaust for handling solvents.

NOTE 1—The exhausting work station is recommended to prevent solvent vapors from entering the laboratory area (see Industrial Ventilation, a Manual of Recommended Practice).

NOTE 2—Verify that the airborne particle concentrations in the work stations are ISO Class 5 FS209 Class 100, or better, per ISO 14644-1, when tested in accordance with Practice F50.

NOTE 3—Verify NVR levels in the work stations are acceptable using the procedures in this standard.

6.4 *HEPA Filters*—All HEPA filters shall be constructed of low outgassing, corrosion-resistant, and fire-resistant materials such as Grade 1 in IEST-RP-CC001.3. Filters with stainless steel or aluminum frames should be considered. The filters shall not be tested with DOP (dioctylphthalate) or other liquid aerosols. Ambient air and solid aerosol test methods are acceptable alternatives to the DOP test. Applicable test methods from IEST-RP-CC007.1 and IEST-RP-CC034.1 shall be considered.

6.5 *Vacuum Filtration System,* consisting of a 47-mm-diameter membrane filter funnel[8] and vacuum pump that will provide a pressure of 30 kPa (250 torr) (a vacuum of 20 in. Hg). See Fig. 1.

6.6 *Solvent-Resistant Filter,* 47-mm diameter,[9] 0.2-µm pore size (nominal) fluorocarbon.

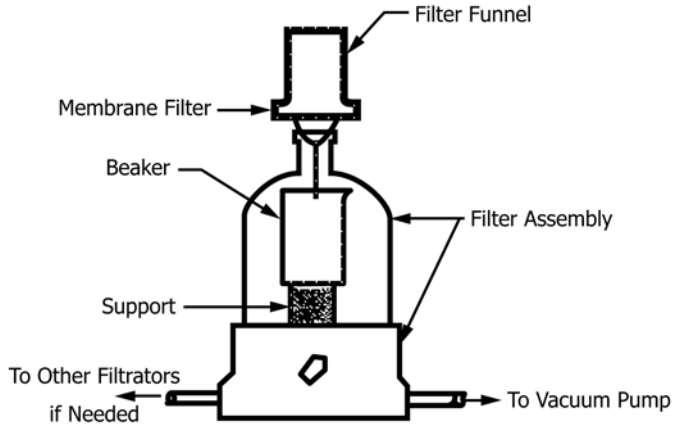

FIG. 1 Vacuum Filtration Apparatus

6.7 *Tweezers or Hemostat,* stainless steel or coated with TFE-fluorocarbon.

6.8 *Beakers,* low-form, glass, 250 mL, etched with an identification number.

6.9 *Evaporating Dish (Petri Dish),* borosilicate glass, approximately 15 g in mass, 60-mm diameter by 12 mm deep, and etched with an identification number.

6.10 *Liquid Laboratory Detergent.*

6.11 *Gloves,* solvent compatible and resistant.[10] (**Warning**—Gloves shall be used to protect the hands from accidental spills of the NVR solvent and minimize contamination of exposed samples. Gloves shall be selected to meet local safety and contamination control requirements.)

6.12 *NVR Plate,* Type 316 corrosion-resistant steel with an area of approximately 0.1 m^2 (1 ft^2). The plate shown in Fig. 2 has been found to be satisfactory. The finish of the sampling surface shall be 0.80 µm (32 µin.) or better per ANSI/ASME B46.1. The plate shall be electropolished and engraved with an identification number.

6.13 *NVR Plate Cover,* Type 316 corrosion-resistant steel. The cover shown in Fig. 3 has been found to be satisfactory. The finish shall be 0.80 µm (32 µin.) or better per ANSI/ASME B46.1. The cover shall be electropolished and engraved with an identification number.

6.14 *Oil-Free Aluminum Foil*[11], to cover the NVR plate if the cover (6.12) is not used.

NOTE 4—The hard cover (6.13) is preferred for ease of handling and possible tearing of the foil resulting in contamination of the NVR plate.

6.15 *Noncontaminating Nylon Bag* to enclose each covered NVR plate.[12]

6.15.1 Bags shall not contain or generate molecular or particulate matter that could contaminate the NVR plate or NVR plate carrier.

[7] Sartorius Model R180D, or equivalent.

[8] Gelman filter funnel P/N 4012/Fisher filtrator assembly Cat. No. 09-788 and Millipore Cat. No. XX1504700 filtration assembly have been found to be satisfactory. Other suitable filtration apparatus may be used.

[9] Millipore Corp. Fluoropore filter Cat. No. FGLP 04700, and Gelman Sciences, Inc. Prod. 66143 PTFE have been found to be satisfactory. Other equivalent solvent resistant filters may be used.

[10] Pioneer green nitrile gloves, Catalog No. A10-1, have been found to be satisfactory.

[11] Fed Spec. Food Service Grade aluminum foil, oil free, Federal Stock No. 8135-00-724-0551 has been found to be satisfactory.

[12] Nylon 6 (heat-sealable Capran 980 from Allied Chemical) has been found to be satisfactory.

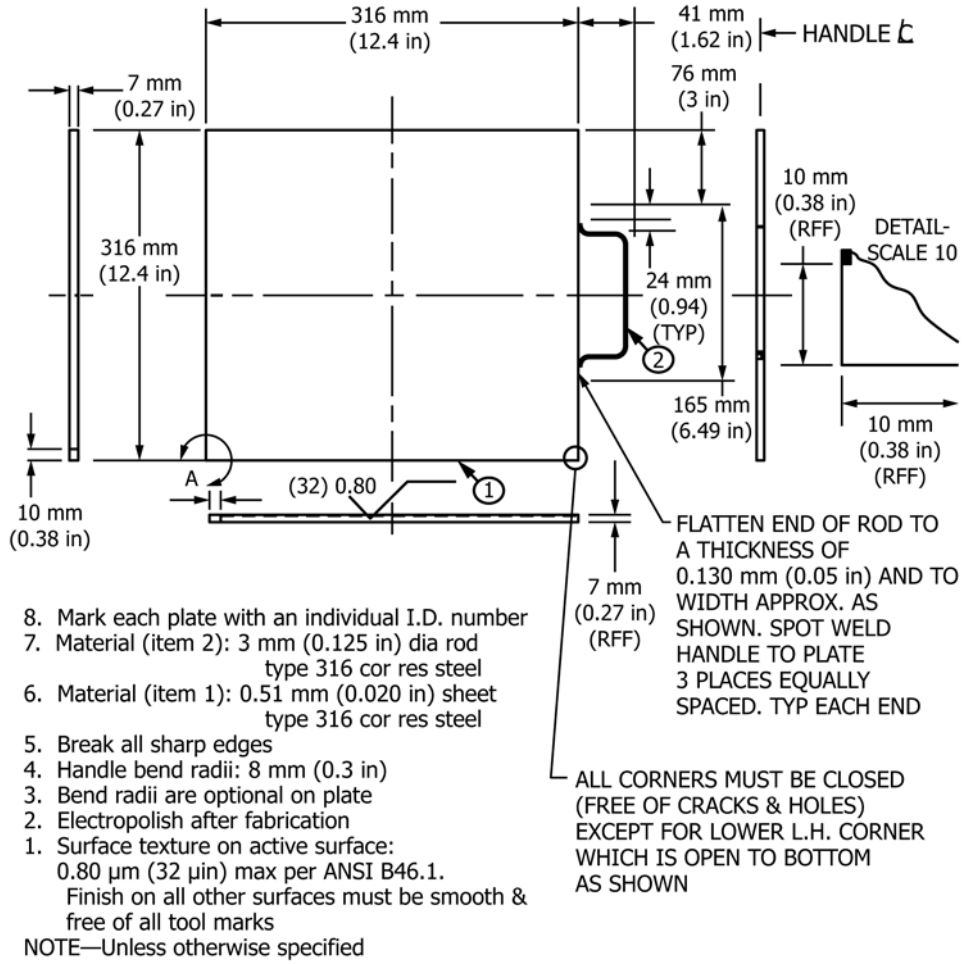

FIG. 2 NVR Collector Plate

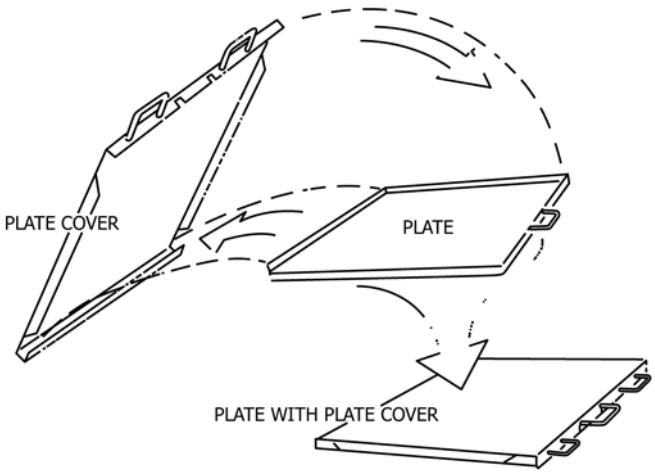

FIG. 3 NVR Plate Cover

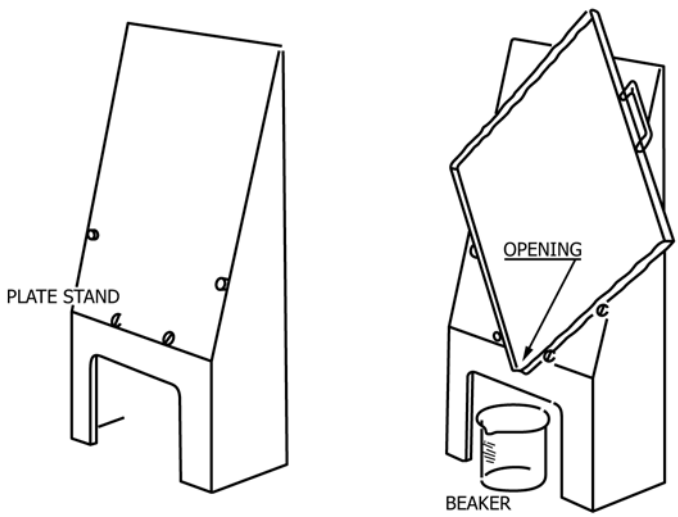

FIG. 4 NVR Plate Stand

6.16 *NVR Plate Carrier*—The sealable, aluminum carrier shown in Fig. 4 has been found to be satisfactory (see Practice E1234).

6.17 *Drying Oven:*

6.17.1 The drying oven shall not produce molecular and particulate contaminants and shall not be used for other operations that could contaminate samples.

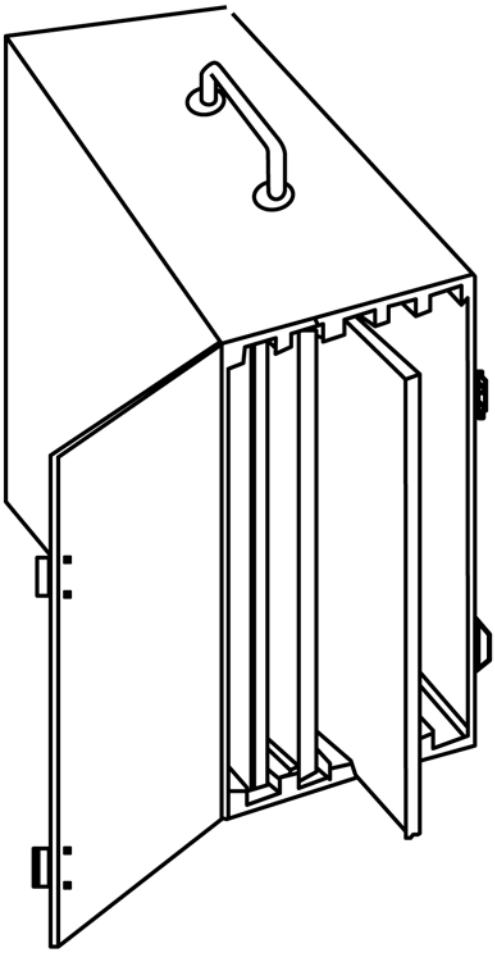

FIG. 5 NVR Plate Carrier

6.18 *Plate Stand*—The plate stand shown in Fig. 5 has been found useful for holding the NVR plate during solvent flushing.

6.19 *Temperature and Relative Humidity Monitors,* as required, to monitor processes that are sensitive to these environments.

6.20 Vacuum oven evaporation system (Method 2), consisting of a vacuum oven, a two-stage vacuum pump, and vacuum gage. The vacuum oven shall be controllable to within ±5°C over an operating range of 25 to 100°C. Fig. 6 shows a typical vacuum oven evaporation system. Two solvent traps cooled with isopropanol/dry ice baths, collect the solvent vapors to prevent release into the atmosphere, protect the vacuum pump, and allow recycling of the solvent.

6.21 Automatic, controlled environment (nitrogen atmosphere) evaporator capable of controlling to a temperature of 37°C[13] (Method 3). Fig. 7 shows a typical arrangement.

6.22 600-mL (450-mL capacity) graduated, borosilicate glass tubes, 75-mm diameter, 150 mm high with 2-mL stems, to fit in the temperature controlled block in the evaporator[14] (Method 3).

[13] RapidVap N_2, Model 79100, evaporation system, with No. 79065 sample block, Labconco Corp., 8811 Prospect Ave., Kansas City, MO 641132-2696 has been found to be satisfactory.

[14] Catalog No. 79138-00 borosilicate glass tubes (6) with 2-mL stems to fit the RapidVap Model 79100 has been found to be satisfactory.

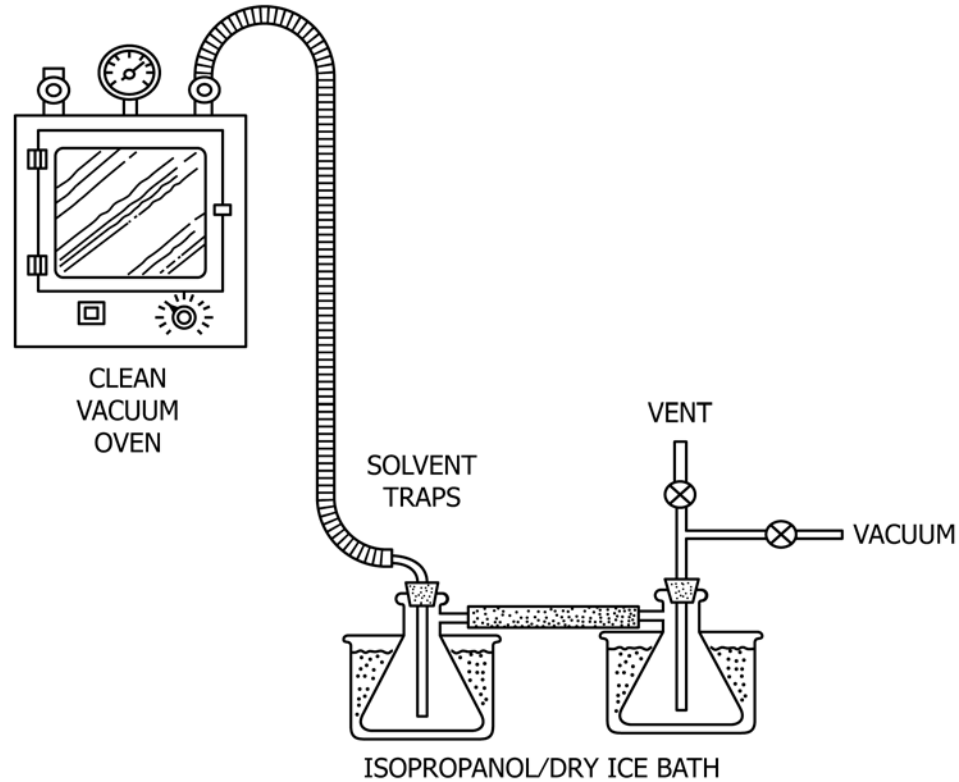

FIG. 6 Vacuum Oven Evaporation System

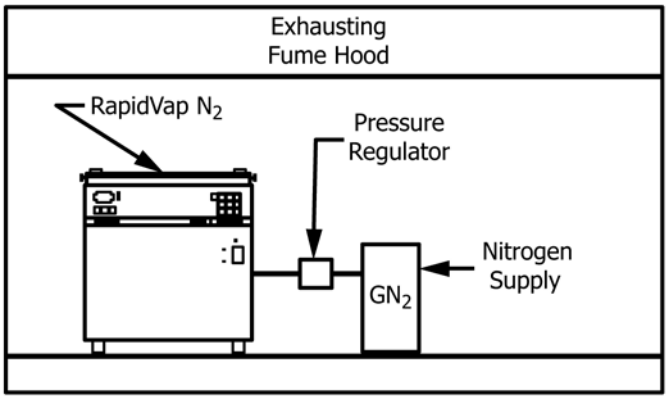

FIG. 7 Automatic Evaporator System

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical reagents of the American Chemical Society, when such specifications are available.[15]

7.2 *Purity of Water*—Deionized, organic-free water such as reagent water, Type II in accordance with Specification D1193 with a minimum resistivity of 1.0 MΩ-cm.

7.3 *Acetone,* reagent grade.

7.4 *Methanol Absolute,* reagent grade.

7.5 *Nitrogen,* ISO 14951-3, Type I (gaseous), Grade B or better.

7.6 *NVR Solvent* (see Note 5)—Methylene chloride, used in Test Method E1235-95, is toxic[16], and is being phased out for many applications. Methylene chloride has been replaced in this revision of Test Method E1235. The replacement solvents were selected based on tests and analyses performed by The Aerospace Corporation and described in SMC-TR-95-28. The following solvents are acceptable:

7.6.1 *HPLC (High-Purity Liquid Chromatography) Grade Ethyl Acetate*[17]—The solvent shall be certified to contain <1-ppm (<1-mg/L) NVR using the procedure in Section 10.

NOTE 5—Ethyl acetate is an organic solvent, and as such, presents some degree of physical and health hazard. Use of ethyl acetate should be according to the recommendations provided in the Material Safety Data Sheet.[18]

7.6.2 *Cyclohexane/Ethyl Acetate Azeotrope Mixture*—This is an azeotrope consisting of 44 to 46 % cyclohexane[19] and 54 % ethyl acetate[20] by mass or 53 % cyclohexane and 47 % ethyl acetate by volume.[21,22] The solvent shall be certified to contain <1-ppm (<1-mg/L) NVR using the procedure in Section 10.

NOTE 6—Cyclohexane/ethyl acetate azeotrope is an organic solvent, and as such, presents some degree of physical and health hazard. Use of cyclohexane should be according to the recommendations provided in the Material Safety Data Sheet.[23] No commercial sources have been located for this solvent mixture. Users have blended their own mixtures using cyclohexane and ethyl acetate.

NOTE 7—Other solvents may be used to perform these measurements. The use of different solvents may be required because of incompatibilities of these solvents with surfaces being sampled or for other operational reasons; however, the results may be different because the performance characteristics of the solvents are different. This means that comparisons of NVR data determined with different solvents may not be possible.

CERTIFIED FOR SAMPLING	
Inspector:	Date:
LAB W.O. No.:	Date Sampled
Measurement: NVR	Quantity: (1) mg/
Item: (2)	Serial No.:

(1) The quantity of NVR can be "mg/L", "mg/0.1 m², etc.

(2) The item can be "NVR plate", "plate carrier", "NVR solvent", etc.

FIG. 8 Typical Certification Tag

8. Cleaning of Equipment

8.1 All operations, except weighings, shall be performed in a unidirectional air flow, clean work station {ISO Class 5 (FS209 Class 100) environment as defined in ISO 14644-1} or an equivalent cleanroom or clean zone.

8.2 Clean the glassware, tools, plate cover, and NVR plates by washing twice with a strong liquid detergent in water followed by a deionized water rinse. Then rinse the object with acetone, then with methanol, and finally with the NVR solvent described in 7.6. Allow glassware to air dry in the clean work station. Certify cleanliness by analysis.

8.3 Verify that the carrier is visually clean. If cleaning is required, clean to Level 100A per IEST-STD-CC1246.

8.4 Cover the beakers and other equipment in oil free aluminum foil (6.14). Store until required.

[15] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[16] Material Safety Data Sheet No. 310, Genium Publishing Corp., 1145 Catalyn St., Schenectady, NY 12303.

[17] Burdick & Jackson catalog No. 100, B&J Brand ethyl acetate has been found to be satisfactory.

[18] Material Safety Data Sheet No. 437, Genium Publishing Corp., 145 Catalyn St., Schenectady, NY 12303.

[19] Burdick & Jackson catalog No. 053, B&J Brand cyclohexane has been found to be satisfactory.

[20] Burdick & Jackson catalog No. 100, B&J Brand ethyl acetate has been found to be satisfactory.

[21] CRC Handbook of Chemistry and Physics, 52nd Ed, The Chemical Rubber Co., Cleveland, OH.

[22] Data Source For Homogenious Azeotropes at the University of Edinburgh.

[23] Material Safety Data Sheet No. B&J 0053, Burdick & Jackson.

8.5 Certify the cleanliness of the NVR plate by performing an NVR measurement in accordance with Section 10 using 60 mL of NVR solvent. Note the results in the certification tag (Fig. 8). The measured NVR shall be no more than 0.1 mg.

8.6 An NVR plate also may be considered as being certified clean if the previous sampling measurement is within 0.1 mg of the sample blank and the reagent blank is less than 0.05 mg (this is equivalent to a solvent NVR background of approximately 1 mg/L).

8.7 Install the NVR plate cover (6.13) on the NVR plate or enclose in the oil-free aluminum foil (6.14).

8.8 Enclose the covered NVR plate in a noncontaminating nylon bag (6.15) and install in the carrier immediately to avoid contamination. Place the lid on the carrier and fasten securely.

8.8.1 A certification decal or tag (Fig. 8) shall be packaged with each NVR plate.

8.9 Seal the carrier in a noncontaminating bag.

8.9.1 The noncontaminating nylon bag shall be used when the carrier is to be removed from a controlled environment for transport.

8.10 Affix the proper quality control decal or tag (Fig. 8) to the carrier in accordance with Practice E1234 and local requirements.

8.11 Prepare a paper "traveler" (Fig. 5 in Practice E1234) in accordance with Practice E1234.

8.12 Attach the paper "traveler" to the outer bag or container.

8.13 Store the carrier inside the noncontaminating nylon bag in a good housekeeping area until required for use. Reclean the NVR plates in accordance with 8.2 for acceptability after six months or as determined by local conditions.

NOTE 8—An acceptable storage time for the NVR plates before requiring recertification should be determined because local conditions can affect the process.

9. Postexposure Handling

9.1 Storage of the carrier, following the return of the exposed samples, shall be in a good housekeeping area, or cleaner environment.

9.2 Remove the NVR plate carrier from the outer plastic bag immediately before placing the carrier into a ISO Class 5 (FS209 Class 100) or better unidirectional air flow work station.

9.3 Clean the outside of the NVR plate carrier if required.

9.4 Remove the NVR sample plates individually, including the blank, and perform a visual inspection of each. Record observations on the "traveler" form (see Fig. 5 of Practice E1234) accompanying the NVR plate carrier. Replace plates in the carrier.

NOTE 9—Large particles of organic materials on the plate will affect the NVR measurements if the NVR solvent extracts soluble matter from the particles. Large particle fallout is more severe on horizontally mounted than on vertically mounted plates. To reduce possible errors, the plates may be flushed with clean, dry nitrogen. If a nitrogen flush is used, all plates, including the blank, shall be flushed.

10. NVR Solvent Certification

10.1 Procure the NVR solvent in accordance with 7.6.

10.2 The NVR of each bottle shall be determined upon opening in accordance with 10.7.

10.3 All operations, except weighings, shall be performed in a unidirectional air flow, clean work station {ISO Class 5 (FS209 Class 100) environment as defined in ISO 14644-1} or an equivalent cleanroom or clean zone.

10.4 Solvent operations shall be performed in the exhausting work station (6.3), which is an ISO Class 5 (FS209 Class 100) or cleaner environment as defined in ISO 14644-1.

10.4.1 Handle ethyl acetate and the cyclohexane/ethyl acetate azeotrope with caution and observe appropriate precautions.

10.5 Document all appropriate information using the NVR Analysis Data and Summary Sheet, Figs. 5 and 6.

10.6 Preweigh the certified borosilicate glass petri dish (6.9) using the microbalance (6.1) to the nearest 0.01 mg. Record the mass.

10.6.1 The relative humidity shall not vary by more than ±10 % and the air temperature by more than ±3°C during all weighings. The air velocities during weighings shall not be large enough to disturb the balance.

10.7 Determine the NVR in 100 mL of solvent by one of the following methods.

10.7.1 *Method 1—Air Evaporation:*

10.7.1.1 Pour 100 mL of solvent into a beaker, certified clean in accordance with 8.2.

10.7.1.2 Position the beaker near or under the exhausting flow bench filter bank and allow the solvent to evaporate to dryness.

10.7.1.3 Transfer the contents of the beaker to the preweighed petri dish using a rinse of 5 mL of certified solvent.

10.7.1.4 Allow to evaporate in the exhausting flow bench until no visible solvent remains in the petri dish.

10.7.1.5 Weigh the petri dish and contents on the microbalance.

10.7.1.6 Repeat the weighings after 15 min. If no change in mass is detected, equilibration has been achieved and the evaporation is complete. "No change" is considered to be less than 0.1 mg.

10.7.1.7 The NVR shall be less than 0.1 mg (a solvent NVR of less than 1 mg/L).

10.7.2 *Method 2: Vacuum Oven Evaporation System* (see Fig. 6.):

10.7.2.1 Pour 100 mL of solvent into a beaker, certified clean in accordance with 8.2.

10.7.2.2 Place the beaker in the vacuum oven.

10.7.2.3 Set the oven temperature to 25 ± 5°C.

10.7.2.4 Turn the vacuum pump on and allow the pressure to gradually drop to approximately 30 kPa (250 torr) (vacuum to gradually increase to approximately 500 mm of mercury).

10.7.2.5 If no bumping or boiling occurs, the pressure may gradually be decreased to 7 kPa (50 torr), which is typical of a mechanical pumping system.

10.7.2.6 Allow the solvent to evaporate until about 10 mL remains.

10.7.2.7 Increase the pressure in the vacuum oven to ambient atmosphere with clean, dry air or nitrogen.

10.7.2.8 Quantitatively transfer the solvent to a clean, dried, and weighed petri dish. On emptying the contents of the beaker, do a final rinse with 5 mL of certified solvent and add to the petri dish.

10.7.2.9 The petri dish is returned to the vacuum evaporation system, and evaporation is continued until no visible solvent remains in the dish.

10.7.2.10 Weigh the petri dish and contents on the microbalance.

10.7.2.11 Repeat the weighings after 15 min. If no change in mass is detected, equilibration has been achieved and the evaporation is complete. "No change" is considered to be less than 0.1 mg.

10.7.2.12 The NVR shall be less than 0.1 mg (a solvent NVR of less than 1 mg/L).

10.7.3 *Method 3: Automatic Evaporation System:*

10.7.3.1 Pour 100 mL of solvent into a borosilicate glass tube (6.20), certified clean in accordance with 8.2.

10.7.3.2 Place the borosilicate glass tube in the RapidVap temperature controlled block and close the lid.

10.7.3.3 Set the RapidVap operating conditions as follows:

Time:	15 min (nominal)
Vortex speed:	90 % (nominal)
Temperature:	37°C
GN purge pressure:	30 kPa gage (5 psig)

NOTE 10—The time and vortex speed should be adjusted so that there is no spillage from the glass tube.

10.7.3.4 Start the evaporation process by depressing the run button.

10.7.3.5 Allow the solvent to evaporate until the apparatus stops.

10.7.3.6 Extract the residual, approximately 1 mL of solvent, with a Pasteur pipet into a preweighed petri dish.

10.7.3.7 Rinse the stem of the borosilicate glass tube with 1 mL of solvent two times using the Pasteur pipet and add to the same petri dish. The dish now contains approximately 3 mL of solvent.

10.7.3.8 Transfer the petri dish to the RapidVap.

10.7.3.9 The petri dish needs to be supported on the temperature controlled block because the dish diameter is less than the hole diameter and prevented from moving during rotation. Also, it has been found useful to extend the nitrogen flow from each stainless steel tube closer to the petri dish. Fluorocarbon tubes 3-mm I.D. by 5-mm O.D. by 100 mm long (⅛-in. I.D. by 3⁄16-in. O.D. by 4 in. long) has been found to be satisfactory. The tubes shall not touch the solvent or dishes.

10.7.3.10 Set the following operating conditions:

Time:	15 min (nominal)
Vortex speed:	30 % (nominal)
Temperature:	37°C
GN purge pressure:	30 kPa gage (5 psig)

NOTE 11—The time and vortex speed should be adjusted so that there is no spillage from the petri dish.

10.7.3.11 Start the evaporation process by depressing the run button.

10.7.3.12 After evaporation is completed, leave the petri dish in the RapidVap for 30 min at 37°C.

10.7.3.13 Remove the petri dish and allow to equilibrate in the unidirectional flow bench for a minimum of 30 min.

10.7.3.14 Weigh the petri dish and contents on the microbalance in a clean environment.

10.7.3.15 Repeat the weighings after 15 min. If no change in mass is detected, equilibration has been achieved and the evaporation is complete. "No change" is considered to be less than 0.1 mg.

10.7.3.16 The NVR shall be less than 0.1 mg (1 mg/L).

10.8 Label the outside of the solvent bottle with the NVR result and date.

10.9 Repeat the solvent certification in three months for any unused solvent.

10.10 Out-of-specification NVR solvent (>0.1-mg NVR) (>1 mg/L) may be set aside, appropriately marked, used for cleaning purposes, or distilled and recertified.

10.11 Only clean, certified, noncontaminating metals, glass, and fluorocarbons are acceptable materials for containers that are used to hold the NVR solvent.

10.12 Secondary containers shall be certified clean in accordance with 8.2.

11. Method 1: Analysis Using Manual Evaporation

11.1 All operations, except weighings, shall be performed in an ISO Class 5 (FS209 Class 100) or cleaner environment, (as defined in ISO 14644-1), unidirectional air flow, clean work station or an equivalent cleanroom or clean zone.

11.2 Solvent operations shall be performed in the exhausting work station (6.3), which is an ISO Class 5 (FS209 Class 100) or better environment as defined in ISO 14644-1.

11.2.1 Handle ethyl acetate and the cyclohexane/ethyl acetate azeotrope mixture with caution and observe appropriate precautions.

11.2.2 The air shall not contain molecular species that can contaminate the samples and laboratory apparatus. Filters and equipment shall not be tested with DOP (dioctylphthalate), other liquid aerosols, or smoke.

11.3 The air temperature shall be monitored and controlled to ±3°C within the range of 20 to 25°C.

11.4 The relative humidity shall be monitored and controlled at 60 ± 5 %.

11.5 Monitor temperature and humidity and note deviations from the required conditions. The effects of temperature and humidity on the NVR results will be evaluated using this data and as a part of a round-robin test program.

NOTE 12—The evaporation of the NVR solvent results in a temperature drop and possible condensation of water, from the air, into the solvent. This effect can be eliminated by evaporating to dryness in the beaker or evaporating in a dry atmosphere. See the alternate evaporation procedures.

11.6 Assemble the filtration assembly in accordance with Fig. 1.

11.7 Flush the assembly with 20 mL of certified NVR solvent and discard the solvent.

11.8 Select several 250-mL beakers (6.8) and petri dishes (6.9) that have been certified clean in accordance with 8.2.

11.9 Weigh the clean evaporation (petri) dishes and record the number of each dish along with its mass, to the nearest 10 µg, on the Test Report Form (see Fig. 8).

11.10 Keep an historical record of the masses of each petri dish so that changes or errors can be easily detected.

11.11 Remove one NVR plate from the plate carrier and slide the plate out of the plastic bag. Save the bag for reuse. Discard any bag that is torn or visually unsuitable.

11.12 Record all appropriate information onto the Test Report Form.

11.13 Verify that the certification tag agrees with the information on the paperwork, for example, the tracking number, plate ID number, cover ID number, date, and so forth.

11.14 Carefully remove the plate cover from the NVR plate while in the unidirectional airflow bench. If aluminum foil is used to cover the plate, carefully remove the foil without touching the sampling surface of the NVR plate. Discard the aluminum foil.

NOTE 13—Be careful to hold the NVR plate only by its handle.

11.15 Place the NVR plate on the plate stand (see Fig. 5) with the corner drain over a certified beaker.

11.16 Record the beaker identification number on the Test Report Form.

11.17 Rinse the NVR plate with 30 mL of NVR solvent, and repeat with 20 mL of solvent, thoroughly rinsing the entire sampling surface area of the plate. (**Warning**—Take care not to contaminate the NVR solvent. Keep container tightly closed when not in use.)

11.18 Filter the collected solvent through the 0.2-µm filter using the vacuum filtration assembly shown in Fig. 1.

11.19 Follow with a rinse of 5 mL of solvent through the filter assembly.

11.19.1 Replace the filter membrane with a new filter following each individual filtration. The filter assembly shall not be used for other laboratory procedures because of potential contamination.

11.20 Collect the filtrate in a certified clean, 250-mL, identified beaker.

11.21 Evaporate the filtrate using one of the following three methods:

11.21.1 *Method 1: Manual Evaporation Method:*

11.21.1.1 Place the 250-mL beaker containing the filtrate in a HEPA-filtered, unidirectional air flow, exhausting work station. Position the beaker near or under the flow bench filter bank. Allow the filtrate to evaporate to dryness.

11.21.1.2 Quantitatively transfer the residue to a clean, dry, preweighed, evaporating (petri) dish with small amounts of certified NVR solvent. Record the added amount of solvent to the nearest 1 mL.

NOTE 14—The total volume of solvent used for rinsing should be approximately the same for each sample and blank.

11.21.1.3 Place the evaporating dish and contents in the exhausting work station. Allow to evaporate until no visible, liquid solvent remains.

11.21.1.4 Place the evaporating dish and residue in a drying oven at 35 ± 2°C for a minimum of 30 min.

11.21.2 *Method 2: Evaporation Using Vacuum Oven Evaporation System:*

11.21.2.1 This procedure uses a vacuum oven to increase evaporation rates so as to shorten processing times. (See Fig. 6.)

11.21.2.2 Place the 250-mL beaker containing the filtrate in the vacuum oven.

11.21.2.3 Set the oven temperature to 25 ± 5°C.

11.21.2.4 Turn the vacuum pump on and allow the pressure to drop gradually to approximately 30 kPa (250 torr) (vacuum to increase gradually to approximately 500 mm of mercury).

11.21.2.5 If no bumping or boiling occurs, the pressure may gradually be decreased to 7 kPa (50 torr), which is typical of a mechanical pumping system.

11.21.2.6 Allow the solvent to evaporate until about 10 mL remains.

11.21.2.7 Increase the pressure in the vacuum oven to ambient atmosphere with clean, dry air or nitrogen.

11.21.2.8 Quantitatively transfer the solvent to a clean, dried, and weighed petri dish. On emptying the contents of the beaker, do a final rinse with 5 mL of certified solvent and add to the petri dish.

11.21.2.9 The petri dish is returned to the vacuum evaporation system, and evaporation is continued until no visible solvent remains in the dish.

11.21.3 *Method 3: Evaporation Using Automatic Evaporator (RapidVap):*

11.21.3.1 This procedure uses an automatic evaporator to increase evaporation rates so as to shorten processing times.[24]

11.21.3.2 Fig. 7 shows a typical laboratory arrangement using the RapidVap apparatus (6.19). The RapidVap shall be located in an exhausting hood to prevent certified NVR solvent vapor from entering the laboratory.

11.21.3.3 Quantitatively transfer the solvent, without losing any solvent, to a clean, dry, 600-mL RapidVap glass tube (6.22). Aid the transfer by rinsing with small amounts of NVR solvent added to the 250-mL beaker. Record the added amount of solvent to the nearest 1 mL.

NOTE 15—The total volume of solvent used for rinsing should be approximately the same for each sample and blank.

11.21.3.4 Six borosilicate glass tubes can be processed in each run in the RapidVap evaporator.

11.21.3.5 Place each borosilicate glass tube in the RapidVap temperature controlled block and close the lid.

11.21.3.6 Set the RapidVap operating conditions as follows:

Time:	15 min (nominal)
Vortex speed:	90 % (nominal)
Temperature:	37°C
GN purge pressure:	200 kPa gage (30 psig)

[24] This procedure is written to the RapidVap N_2, Model 79100, evaporation system, Labconco Corp., 8811 Prospect Ave., Kansas City, MO 641132-2696. Other automatic evaporators may be satisfactory, but the procedure should be tailored to the apparatus that is used.

NOTE 16—The time and vortex speed should be adjusted so that there is no spillage from the petri dish.

11.21.3.7 Start the evaporation process by depressing the "run button."

11.21.3.8 Allow the solvent to evaporate until the apparatus stops. At this time, there should be 1 mL of solvent remaining in the tube stem.

11.21.3.9 Extract the residual, approximately 1 mL, of solvent, from each glass tube with a Pasteur pipet into individual, preweighed petri dishes.

11.21.3.10 Rinse the stem of each borosilicate glass tube with 1 mL of solvent two times using the Pasteur pipet and add each to the original petri dishes. Each dish now contains approximately 3 mL of solvent.

11.21.3.11 Transfer each petri dish to the RapidVap.

11.21.3.12 The petri dish needs to be supported on the temperature-controlled block because the dish diameter is less than the hole diameter and prevented from moving during rotation. Also, it has been found useful to extend the nitrogen flow from each stainless steel tube closer to the petri dish. Fluorocarbon tubes 3-mm I.D. by 5-mm O.D. by 100 mm long (⅛-in. I.D. by 3⁄16-in. O.D. by 4 in. long) has been found to be satisfactory. The tubes shall not touch the solvent or dishes.

11.21.3.13 Set the following operating conditions:

Time:	15 min (nominal)
Vortex speed:	30 % (nominal)
Temperature:	37°C
GN purge pressure:	200 kPa gage (30 psig)

NOTE 17—The time and vortex speed should be adjusted so that there is no spillage from the petri dish.

11.21.3.14 Start the evaporation process by depressing the run button.

11.21.3.15 After evaporation is completed, leave each petri dish in the RapidVap for 30 min at 37°C.

11.21.3.16 Remove each petri dish and allow to equilibrate in the unidirectional flow bench for a minimum of 30 min.

11.22 Weigh the petri dish and contents to the nearest 0.01 mg using the microbalance and record on the Test Report Form. Perform weighings in a clean environment, such as a containment weigh station or cleanroom, and return the dish and contents to the unidirectional air flow work station.

11.22.1 The objective is to provide a clean, low air velocity environment for the weighings so as not to disturb the microbalance. The relative humidity shall not vary by more than ±5 % and the air temperature by more than ±3°C during all weighings. The air velocities during weighings shall not be large enough to disturb the balance.

11.23 Record temperature and relative humidity and note deviations from the required conditions. The effects of temperature and humidity on the NVR results will be evaluated using this data and as a part of a round-robin test program.

11.24 Repeat the weighings after 15 min. If no change in mass is detected, the evaporation and equilibration are complete. No change is considered to be within ±0.01 mg.

11.25 If the weighings show mass changes, return the evaporating dish and contents to the containment weigh station and repeat the weighings in 15 min. Repeat the above procedure until the samples have equilibrated.

11.26 Retain the NVR sample for further analysis if it is 0.5 mg or greater or if requested to do so by the requester.

11.27 The NVR plate may be accepted as certified for sampling if the resulting NVR is less than 0.1 mg.

11.28 If the resulting NVR is greater than 0.1 mg, repeat the steps in 11.2 – 11.32 and Section 12 until the NVR is less than 0.1 mg.

11.29 Visually inspect the NVR plate cover for cleanliness and damage. Clean per Section 8. Damaged plates shall be repaired or discarded.

11.30 Cover the certified NVR plate with the clean cover or the oil-free aluminum foil (6.14).

11.30.1 If aluminum foil is used, it shall be placed tightly over the plate so that the foil does not sag and contact the sampling surface of the plate. Also, the foil must be snug so that the covered plate will fit in the carrier without tearing the foil.

11.31 Prepare the certification tag (Fig. 8) and attach to or place into the nylon plastic bag along with the covered NVR plate.

11.32 Store in the carrier ready for use.

12. Blank Correction

12.1 A blank sample shall be run with each batch of samples.

12.2 The blank sample shall be an NVR plate that is treated identically to the exposed NVR plates, except for exposure to the cleanroom environment, to determine solvent background and handling effects.

12.3 Perform the NVR analysis using one of the methods specified in Section 12.

12.4 Calculate the NVR of the blank NVR plate, using Fig. 9, as follows:

$$m_b = m_f - m_i \,(\text{mg}) \tag{1}$$

where:

m_f = mass of petri dish plus residue, mg;
m_i = mass of petri dish, clean, mg; and
m_b = mass of blank NVR, mg.

12.5 Record the blank NVR in the proper place in Fig. 9, NVR Sample Blank and Solvent Blank form.

13. Calculation

13.1 Calculate the NVR mass per unit area (mg/0.1 m^2) for each NVR plate, using Fig. 10, NVR Test Report Data Sheet, as follows:

$$\text{NVR} = \frac{(m_f - m_i) - m_b}{10A}\,(\text{mg}/0.1\ \text{m}^2) \tag{2}$$

where:

m_f = mass of petri dish plus residue, mg;
m_i = mass of petri dish, clean, mg;
m_b = mass of blank NVR, mg; and
A = area of NVR plate, m^2.

SOLVENT NVR

				Solvent Blank
1	Sample No.			
2	Solvent			
3	Solvent Volume	(V)	mL	
4	Beaker ID			
5	Petri Dish ID			
6	Final Mass, Dish	(m_f)	mg	
7	Temp./Humidity		°C / %	
8	Initial Mass. Dish	(m_i)	mg	
9	Temp./Humidity		°C / %	
10	Measured Solvent NVR ($m_f - m_i$)		mg	
11	Solvent NVR	$10^3(m_f - m_i)/V$	mg/L	

SAMPLE BLANK NVR

				Sample Blank
12	Sample No.			
13	NVR Plate S/N			
14	NVR Plate Area	(A)	m^2	
15	Solvent			
16	Solvent Volume	(V)	mL	
17	Beaker ID			
18	Petri Dish ID			
19	Final Mass, Dish	(m_f)	mg	
20	Temp./Humidity		°C / %	
21	Initial Mass. Dish	(m_i)	mg	
22	Temp./Humidity		°C / %	
23	NVR Blank	$m_b = (m_f - m_i)$	mg	
24	Blank NVR per area	(m_b/A)	mg/0.1 m^2	

Laboratory Analysis by (print name) _____ **Date** _____

Signature _____

FIG. 9 NVR Sample Blank and Solvent Blank

Sample location _____

Sample number _____ NVR Plate S/N _____

Installed _____ Date _____ Time _____

Removed _____ Date _____ Time _____

Day Mo. Yr.

Requester Name _____ Tel. _____

Exposure time (t) Hrs Wk s

_____ _____

Sample blank number _____ Blank NVR plate S/N _____

Beaker ID _____ Petri Dish ID _____

_____ _____

Lab. temperature _____ °C Lab. rel. humidity _____ %

Mass of dish+residue (sample)	(m_f)	_____	mg
Mass of same dish, clean (sample)	(m_i)	_____	mg
NVR, total	$(m_f - m_i)$	_____	mg
Blank NVR (Item 23 from Fig. 8) (Note 1)	(m_b)	_____	mg
NVR, net	$(m_f - m_i) - m_b$	_____	mg
Area of NVR plate (Note 2)	(A)	_____	m^2
NVR per area	$\frac{(m_f - m_i) - m_b}{10A}$	_____	mg/0.1 m^2
Reagent blank (Item 11 from Fig. 8)		_____	mg/L
NVR per area per time (NVR/t)	State time	_____	mg/0.1 m^2- ____

Comments(use additional sheets if necessary) _____

_____

_____

_____

_____

Laboratory Analysis by (print name) _____ | Date _____

Signature _____

Notes: (1) This assumes that the blank NVR plate has the same area as the sample NVR plate. If the areas are different, subtract the blank NVR after normalizing values to mg/0.1 m^2.

(2) Use the actual area in m^2 for A. For example, a 1 ft^2 plate has an area of 0.0929 m^2.

FIG. 10 NVR Test Report Data Sheet

NOTE 18—When other than a 0.1-m^2 plate is used, use the correct area in square metres. If a 1-ft^2 plate is used, for example, the area is 0.0929 m^2.

NOTE 19—Fig. 10 assumes that the blank NVR plate has the same area as the NVR sample plate. If the areas are different, subtract the blank NVR from the sample NVR after normalizing to an area of 0.1 m^2.

13.2 The results for cleanrooms are usually normalized to one month (four weeks); however, other time periods may be used.

NOTE 20—This procedure defines a month to be four consecutive weeks of seven days each.

14. Report

14.1 The report shall use the Test Report Forms shown in Figs. 10 and 11.

14.2 Note exposure time in days.

14.3 State the normalized NVR time (week, month, and so forth).

14.4 State the estimated accuracy for the NVR for the same time period used in 13.2.

14.5 The kinds of comments to be stated shall include observations made during NVR plate installation and removal, condition of the NVR plate (extraneous debris and other material or evidence of improper handling), color of the solvent rinse, and anomalies observed during the analysis.

15. Quality Control

15.1 Develop a quality control program to validate periodically the laboratory performance of this procedure and conformance to the other provisions of this procedure.

15.2 The quality control program shall include but not be limited to the following.

15.2.1 Maintain and calibrate instruments.

15.2.2 Monitor the laboratory environment (temperature, relative humidity, and air cleanliness).

15.2.3 Process plates with known NVR quantities through the laboratory along with clean, blank NVR plate.

16. Precision and Bias

16.1 Precision and bias have not yet been determined.

17. Keywords

17.1 gravimetric determination; nonvolatile residue

Number of samples in batch		
Time for weighing of batch		**hours**
Temperature at start of weighing		**°C**
Temperature at end of weighing		**°C**
Relative humidity at start of weighing		**%**
Relative humidity at end of weighing		**%**
Reagent blank, NVR solvent		**mg/L**

Sample Number	NVR (mg/0.1 m^2−mo)		
		±	
		±	
		±	
		±	
		±	

FIG. 11 Summary NVR Data Sheet