



# Standard Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Flash Evaporator)<sup>1</sup>

This standard is issued under the fixed designation F331; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of nonvolatile matter, that is, residue on evaporation, in solvent extract from aerospace components, using a rotary flash evaporator.

1.2 The procedure for extraction from components is described in practices such as Practice F303. Before subjecting the extract to the following method, it should be processed to remove the insoluble particulate in accordance with Practice F311 (Note 1). Particle count analysis of the removed particulate may then be performed in accordance with Test Method F312. If particulate is not removed from the extract prior to performing this method, this should be noted on the test report.

NOTE 1—Membrane filters with a maximum extractable content of 0.5 weight % should be used on samples to be processed by this test method. Conventional membranes contain 5 to 10 % extractables. For obtaining very low background levels, consideration should be given to using membranes without grid marks.

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

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:*<sup>2</sup>

D1193 Specification for Reagent Water

F303 Practices for Sampling for Particles in Aerospace Fluids and Components

F311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters

<sup>1</sup> 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.

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<sup>2</sup> 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.

F312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters

2.2 *IEST Standard:*<sup>3</sup>

IEST-STD-1246D Product Cleanliness Levels and Contamination Control Program

## 3. Summary of Test Method

3.1 A sample of fluid or the filtrate (Note 1) from a sample of extract from components is evaporated as necessary to approximately 20 mL in a flash evaporator. The residue is then transferred to a foil dish and the evaporation completed by heating to a constant weight.

## 4. Apparatus

4.1 *Oven*, gravity convection provided with suitable thermometer and a temperature range suitable for the solvent being evaporated.

4.2 *Analytical Balance*, single pan or magnetically damped double pan.

NOTE 2—Sensitivity shall be suitable to obtain the required precision noted in 10.1.

4.3 *Evaporator*, flash, batch-type.

4.4 *Graduated Cylinder*.

4.5 *Tongs or forceps*, laboratory, for manipulating weighing foil dishes.

4.6 *Desiccator*, balance, to be placed in balance case.

4.7 *Desiccator*, cooling with plate.

4.8 *Weighing Vessels*, aluminum foil weighing dishes. These may be purchased pre-formed dishes or may be constructed from clean aluminum foil.

4.9 *Pressure Source*, capable of providing 85 KPa (25-in. Hg) for short interval.

4.10 *Distillation Apparatus*, laboratory, all glass (required where solvents with sufficiently low residue are not available). Do not use grease or oil to lubricate glass joints.

<sup>3</sup> Available from Standardization Document Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

## 5. Reagents and Materials

5.1 *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 where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

5.3 *Solvent for Extracting and Rinsing*—The baseline NVR of the solvent shall not exceed 10 % of the requirement being tested.

5.4 *Cleaning Agent*—Liquid surface-active agent. Approved equivalent may be used, provided they can be adequately rinsed from the surface, leaving a negligible residue.

5.5 *Desiccant*, silica gel, indicating.

## 6. Preparation of Apparatus

6.1 Wash the inner walls of the evaporator flask and graduated cylinder thoroughly with a solution of liquid, surface-active cleaning agent in hot water and rinse with tap water (**Note 3**). Dry thoroughly. Double rinse with test solvent.

**NOTE 3**—Distilled or deionized water shall be used in areas in which hardness or contamination increase the blank over the allowable level.

6.2 Soak the weighing vessels in solvent for 1 h. Dry the weighing vessels in the oven at 65°C or 3°C above the evaporation point of the solvent for 1 h. Place the weighing vessels in the desiccator for 30 min minimum. Do not touch foil dishes with the fingers. Use laboratory tongs or forceps as applicable.

## 7. Procedure

7.1 Determine sample volume (**Note 4**) using a clean graduated cylinder. Transfer (**Note 5**) the sample to a clean flash evaporator flask. Assemble the apparatus. (**Warning**—Do NOT use stopcock grease on the glass joints of the evaporator flask.) Fill the evaporation pan with water, turn on the immersion heater, and adjust the temperature to approximately 50°C (**Note 6**). Turn on the cold water supply and adjust the feed so that running water covers the entire outer surface of the cooling flask (**Note 7**). Start the flash evaporator motor and operate at a pressure of 35 to 80 KPa (vacuum of 10- to 24-in. Hg) (**Note 8**). Continue until volume is reduced to 10 to 20 mL.

**NOTE 4**—The solvent used for testing should be recorded with the collected data for each sample.

**NOTE 5**—All transfers should be accomplished with a double-rinse,

<sup>4</sup> *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.

using three aliquots of a total volume of 20 to 25 mL, which is to be added to the sample.

**NOTE 6**—Use a temperature that is appropriate for the solvent used, but do not exceed the weighing vessel drying temperature in **6.2**.

**NOTE 7**—In some models of flash evaporators a water-cooled condenser is used instead of the cooling flask.

**NOTE 8**—Lower pressure ranges are recommended for solvents such as heptane, acetone, and mixtures of cyclohexane/isopropyl alcohol than for solvents such as straight isopropyl alcohol or ethyl acetate/cyclohexane mixture. Laboratories that have supplies of trichlorotrifluoroethane or trichloroethane/ethanol may use those solvents if they are legally allowed in their jurisdiction.

7.2 Release the vacuum and remove the flask. Using clean tongs, preweigh a foil dish from the desiccator (**Note 9**). Transfer (**Note 5**) the sample from the flask to the preweighed foil dish.

**NOTE 9**—The cover shall be kept securely on the desiccator except when inserting or removing weighing vessels. Desiccant must be replaced when it changes from blue to pink.

7.3 Place the foil dish in the oven until thoroughly dry. Remove and place in the desiccator to cool for 30 min. Remove and weigh to the nearest 0.2 mg.

7.4 If results are in excess of the allowable limits, repeat **7.3**, except place the dish in the oven for an additional 30 min.

7.5 If the blank exceeds 10 % of requirement limit, repeat **7.1-7.4**, using a minimum of 500-mL solvent for blank analysis (**Note 10**). If the blank exceeds 10 % again, the solvent quality is suspect.

**NOTE 10**—Purity of solvents can be improved by distilling it using apparatus called out in **4.10**.

## 8. Calculation

8.1 For extracted samples, report the weight in mg/unit extent in accordance with IEST-STD-1246D or equivalent.

8.2 For solvents and reagents used in this test method, calculate the nonvolatile residue (NVR) in mg/unit volume:

$$\text{NVR} = (A/V) \times \frac{1000 \text{ mL}}{1 \text{ L}} \quad (1)$$

where:

A = weight of residue, mg, and

V = volume of sample, mL.

## 9. Reporting

9.1 NVR results will vary depending on the solvent used for sampling of the contaminant. When reporting test results, the test report must report the solvent used for sampling.

9.2 The surface area of hardware sampled versus the total surface area of the hardware being evaluated will impact the statistical reliability of this test method. When the full surface area of a component is not sampled, the actual or approximate surface area sampled shall be reported. When multiple samples are taken from different locations on a large surface area to provide a more accurate measure of the NVR present, all NVR measurements shall be reported. If a sufficient number of samples are taken, as when qualifying a new cleaning process, statistical deviation shall also be reported.

9.3 The report shall note whether particulate was filtered out prior to the measurement of NVR.

9.4 NVR residue remaining after the completion of this test method may be further analyzed by infrared spectroscopy or other methods if qualitative identification of the contaminant constituents is desired.

## 10. Precision and Bias

10.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.4 mg.

10.2 *Reproducibility*—The averages of duplicate results by different operators in different laboratories should not be considered suspect unless they differ by more than 0.6 mg.

## 11. Keywords

11.1 extractable content; flash evaporator; molecular contamination; nonvolatile residue; NVR; solvent

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