



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

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## 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 Practices F 303 and Practice F 305. In cases in which analysis of particulate contamination is also required, before subjecting the extract to the following method, it should be processed in accordance with Practice F 311 (Note 1). Particle count analysis should then be performed in accordance with Methods F 312. Identification of particulate material, if required, may be performed by Test Method F 314.

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 *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<sup>2</sup>
- D 2021 Specification for Neutral Detergent, 40 Percent Akybenzene Sulfonate Type<sup>3</sup>
- F 303 Practices for Sampling Aerospace Fluids from Components<sup>4</sup>
- F 305 Method of Sampling Particulates from Reservoir-Type Pressure-Sensing Instruments by Fluid Flushing<sup>5</sup>
- F 311 Practice for Processing Aerospace Liquid Samples for

Particulate Contamination Analysis Using Membrane Filters<sup>6</sup>

F 312 Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters<sup>6</sup>

F 314 Test Methods for Identification of Metallic and Fibrous Contaminants in Aerospace Fluids<sup>7</sup>

2.2 *Military Standard*:<sup>8</sup>

MIL-STD-1246 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 9.1.

4.3 *Evaporator*, flash, batch-type.

4.4 *Graduated Cylinder*.

4.5 *Tongs*, 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.

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

4.10 *Bottles*, sample.

4.11 *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>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> Withdrawn. See 1999 *Annual Book of ASTM Standards*, Vol 15.04.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 15.03.

<sup>5</sup> Withdrawn. See 1988 *Annual Book of ASTM Standards*, Vol 15.03.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>7</sup> Withdrawn. See 1988 *Annual Book of ASTM Standards*, Vol 14.02.

<sup>8</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>9</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 D 1193.

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 conforming to Specification D 2021. 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 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.

<sup>9</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.

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, 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 trichlorotrifluoroethane, heptane, acetone, and mixtures of trichloroethane/ethanol or cyclohexane/isopropyl alcohol than for solvents such as straight isopropyl alcohol or ethyl acetate/cyclohexane mixture.

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 4.11.

## 8. Calculation

8.1 For extracted samples, report the weight in mg/unit extent in accordance with MIL-STD-1246 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. Precision and Bias

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

9.2 *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.

## 10. Keywords

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

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