



Standard Practice for Determination of Soluble Residual Contamination in Materials and Components by Soxhlet Extraction¹

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1. Scope

1.1 This practice describes a procedure for the determination of residual contamination in systems and components requiring a high level of cleanliness, such as oxygen, by Soxhlet extraction.

1.2 This practice may be used for extracting nonvolatile and semivolatile residues from solids such as new and used gloves, new and used wipes, contaminated test specimens or control coupons, small pieces of hardware, component softgoods, etc. When used with proposed cleaning materials (wipes, gloves, etc.), this practice may be used to determine the potential of the proposed solvent to extract contaminants (plasticizers, residual detergents, brighteners, etc.) and deposit them on the surface being cleaned.

1.3 This practice is not suitable for the evaluation of particulate contamination.

1.4 The values stated in SI units are standard. Values in parentheses are for information only.

1.5 *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

E 1235 Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft³

F 324 Test Method for Nonvolatile Residue of Volatile Cleaning Solvents Using the Solvent Purity Meter⁴

F 331 Test Method for Nonvolatile Residue of Halogenated Solvent Extract from Aerospace Components (Using Rotary Flash Evaporator)⁴

G 93 Practice for Cleaning Methods and Equipment Used in

Oxygen-Enriched Environments⁵

3. Terminology

3.1 Definitions:

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

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

3.1.3 *nonvolatile residue (NVR), n*—residual molecular and particulate matter remaining following the filtration of a solvent containing contaminants and complete evaporation of the solvent at a specified temperature.

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

3.1.4.1 *Discussion*—The size of a particle is usually defined by its greatest dimension and is specified in micrometres.

3.1.5 *molecular contaminant (non-particulate contamination), n*—the molecular contaminant may be in a gaseous, liquid, or solid state.

3.1.5.1 *Discussion*—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 *control coupon (witness coupon), n*—a coupon made from the same material and prepared in exactly the same way as the test coupons, which is used to verify the validity of the method or part thereof.

3.1.6.1 *Discussion*—In this test method, the control coupon will be contaminated in the same manner as the test coupons and will be subjected to the identical extraction procedure.

4. Summary of Practice

4.1 The sample is placed in an extraction thimble or between two plugs of glass wool and extracted using an appropriate solvent (see Practice G 93 for recommendations) in a Soxhlet extractor. The solvent is brought to the boiling point; the pure solvent vapors travel to the condenser where they condense and drip into the thimble. When the liquid level in the thimble reaches the top of the Soxhlet siphon, the solvent and extracted soluble contaminant are siphoned back into the boiler. This process is allowed to continue for several hours. The solvent and extract are then concentrated or dried for analysis.

¹ This practice is under the jurisdiction of ASTM Committee G-4 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.01 on Test Methods.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 15.03.

⁴ *Annual Book of ASTM Standards*, Vol 10.05.

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

5. Significance and Use

5.1 It is expected that this test method will be suitable to identify and quantify contaminants found in systems, system materials, and components used in systems requiring a high level of cleanliness, such as oxygen. Softgoods such as seals and valve seats can be tested as received. Gloves and wipes, or samples thereof, to be used in the cleaning operation can be evaluated prior to use to ensure that the proposed cleaning solvent does not extract and deposit chemicals on the surface to be cleaned.

5.2 Wipes or other cleaning equipment can be tested after use to determine the amount of contaminant removed from a surface. This procedure can be used to obtain samples for NVR analysis using contaminated control coupons that were subjected to the cleaning process as controls to validate cleaning operations.

5.3 The selection of the solvent requires some knowledge of the contaminant. If a nonvolatile residue (NVR) analysis is to be performed on the molecular contaminant, the boiling point of the solvent shall be significantly lower than that of the contaminant. For other analytical methods, the tester must know the accuracy of the analytical methods, and the solvent shall be chosen so as not to interfere with the selected analytical method. To identify the composition of the NVR, analytical methods such as infrared spectroscopy or gas chromatography/mass spectroscopy have been used satisfactorily.

6. Apparatus

6.1 *Soxhlet extractor*—500 mL evaporation flask and associated equipment as shown in Fig. 1.

6.2 *Boiling chips*—solvent extracted.

6.3 *Water bath*—heated with temperature control of $\pm 5^\circ\text{C}$.

6.4 *Thimbles*—glass or paper.

6.5 *Heating mantle*—rheostat controlled.

6.6 *Balance*—range to a minimum of 50 g with an accuracy of 0.1 mg.

6.7 *Evaporator*—rotary.

7. Reagents

7.1 Solvents that can be used to prepare standard contaminant solutions include the following: 2-propanol, 2-butanone, hexane, methylene chloride, and perfluorinated carbon fluids.

7.2 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁶ 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.

NOTE 1—**Warning:** Solvents such as 2-propanol and 2-butanone are

⁶ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

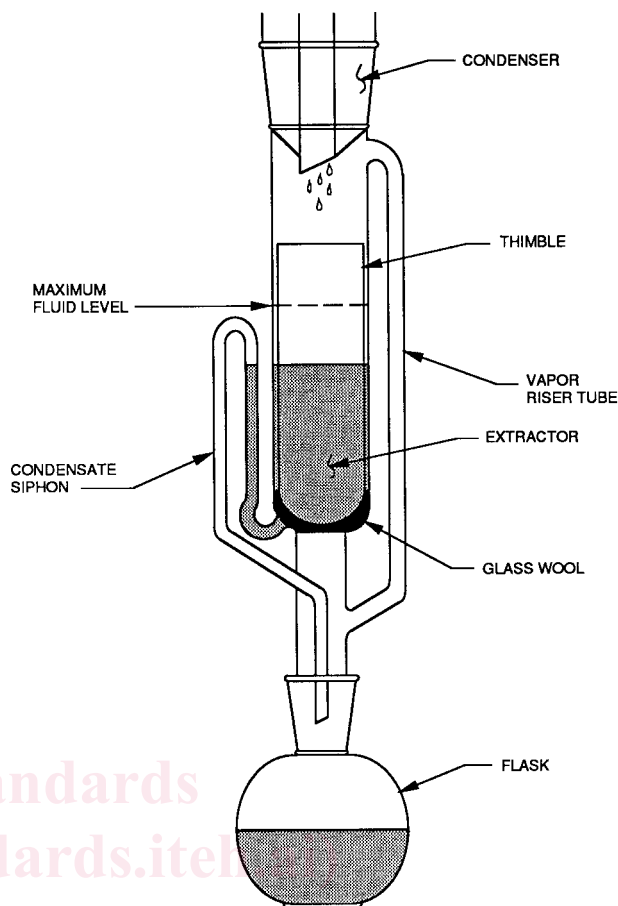


FIG. 1 Soxhlet Extraction Apparatus

highly flammable. The reader should refer to appropriate safe handling procedures.

7.3 *Water*—shall meet the requirements of D 1193, Type II.

8. Procedure

8.1 Prepare the sample for placement in the extractor.

8.1.1 To determine the amount of extractable material in a wiping cloth (new or used), cut out a test section approximately 30 centimetres (cm) square, accurately measure and calculate the area (A) in square centimetres, and weigh the section in grams to the nearest tenth of a milligram (W_1). Record the area and weight.

8.1.1.1 If the NVR is to be determined on a used wiping cloth in an effort to assess the cleanliness of a part or system, a NVR analysis shall be performed on a representative sample of the cloth, with the same surface area, prior to use according to E 1235, F 324, or F 331. Record this NVR as W_3 in mg/g or as W_4 in mg/cm². This NVR value must be subtracted from that determined for the contaminated cloth.

8.1.2 To determine the amount of extractable material in a glove to be used in a cleaning operation, cut several rectangular strips from the fingers and palm, the areas that would typically be exposed to the cleaning solvent, weigh in grams to the nearest tenth of a milligram and record the weight (W_1). Determine the dimensions of each strip in cm and record the total surface area of the strips (A) in square centimetres.

8.1.3 To determine the amount of NVR on control coupons, measure the total contaminated surface area (i.e., front, or