



Standard Practice for Determination of Soluble Residual Contamination in 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.), from the cleaning materials 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.

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 331 Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Rotary Flash Evaporator)³

G 93 Practice for Cleaning Methods for Material and

Equipment Used in Oxygen-Enriched Environments⁴

3. Terminology

3.1 Definitions:

3.1.1 *contaminant, n*—unwanted molecular and particulate matter that could affect the performance of the components or materials 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, n*—non-particulate contamination.

3.1.5.1 *Discussion*—A molecular contaminant may be in a gaseous, liquid, or solid state and may be uniformly or nonuniformly distributed.

3.1.5.2 *Discussion*—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 contaminants are extracted using an appropriate solvent 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

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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 14.04.

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.

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 contaminants and residues 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 (see Practice G 93 for recommendations). 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.

6.8 500 mL concentrator flask.

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

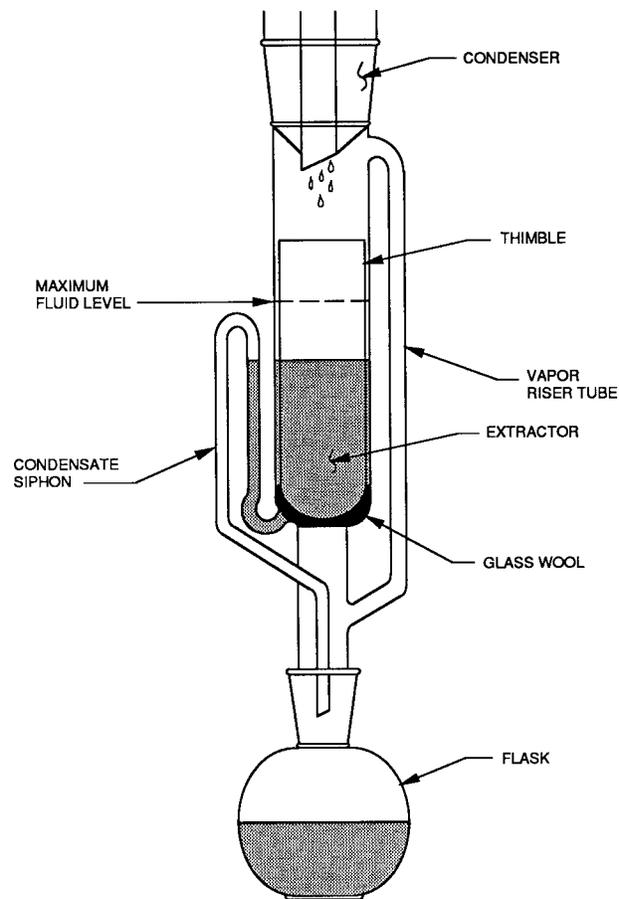


FIG. 1 Soxhlet Extraction Apparatus

such specifications are available.⁵ 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 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 an unused representative

⁵ *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 *Analytical 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.

sample of the cloth, with the same surface area, prior to use according to E 1235 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 front, back, and sides) in square centimetres and weigh in grams to the nearest tenth of a milligram. Record the surface area (A) and weight (W_1).

8.1.4 To determine the amount of NVR on small parts, measure and record the total surface area (A) in square centimetres and weight in grams to nearest tenth of a milligram (W_1) to be evaluated.

8.2 Place the material to be extracted in an extraction thimble or between two glass wool plugs in the extractor.

8.2.1 The glass wool used for the plugs shall be washed prior to use with the solvent to be used for the extraction.

8.3 Place 300 mL of the extraction solvent into the 500 mL flat bottom boiling flask containing one or two clean boiling chips. Place the flask in a heating mantle (or water bath that can be heated 15 to 20°C above the boiling point of the solvent) and attach the extractor to the flask. Attach the condenser to the extractor, turn on the cooling water to the condenser, bring the flask to a boil and extract the sample for six hours.

8.3.1 Extract especially thick or dense materials overnight. If any doubt exists, it is recommended that extractions be performed for different times (on new samples each time) to verify that the shorter extraction time removed all the extractables.

8.4 Allow the extract to cool after the extraction is complete.

8.5 Assemble the rotary evaporator.

8.6 Carefully pour the extract from the 500 mL boiling flask into the 500 mL concentrator flask. Rinse the 500 mL flask with 25 to 50 mL portions of fresh reagent grade extraction solvent and add the rinse solution to the concentrator flask. Repeat the rinse process two more times to complete the quantitative transfer.

8.7 Attach the concentrator flask to the rotary evaporator. Partially immerse the flask in a water bath approximately 15 to 20°C above the boiling point of the solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the flask from the water bath and allow the flask to cool for at least ten minutes at room temperature.

8.8 Determine the weight (W_2) of the nonvolatile residue in grams to the nearest tenth of a milligram using E 1235 or F 331.

9. Calculation

9.1 Calculate the nonvolatile residue as follows:

9.1.1 For new materials, wipes, gloves, etc., the total NVR per unit weight is:

$$\text{NVR} = \frac{1000 \cdot W_2}{W_1} \quad (1)$$

where:

NVR = nonvolatile residue, mg/g sample,
 W_1 = weight of the sample prior to extraction, g, and
 W_2 = weight of the nonvolatile residue, g.

9.1.2 For new materials, wipes, gloves, etc., NVR by unit area is:

$$\text{NVR} = \frac{1000 \cdot W_2}{A} \quad (2)$$

where:

NVR = nonvolatile residue, mg/cm² of sample,
 A = surface area of the sample, cm², and
 W_2 = weight of the nonvolatile residue, g.

9.1.3 For used material, the total NVR per unit weight is:

$$\text{NVR} = \frac{1000 \cdot W_2}{W_1} - W_3 \quad (3)$$

where:

NVR = nonvolatile residue, mg/cm² of sample,
 W_1 = weight of the sample prior to extraction, g,
 W_2 = weight of the nonvolatile residue, g, and
 W_3 = NVR of new sample, mg/g.

9.1.4 For used material, the total NVR per unit area is:

$$\text{NVR} = \frac{1000 \cdot W_2}{A} - W_4 \quad (4)$$

where:

NVR = nonvolatile residue, mg/cm² of sample,
 W_2 = weight of the nonvolatile residue, g,
 A = surface area of the sample, cm², and
 W_4 = NVR of new sample, mg/cm².

10. Report

10.1 The report shall include the following:

10.1.1 Identification of the material being extracted (including tradename, proper chemical name, ASTM designation, lot number, batch number, and manufacturer),

10.1.2 Extraction solvent,

10.1.3 Extraction time,

10.1.4 Evaporation temperature,

10.1.5 Method used to determine the weight of the NVR,

10.1.6 Value of the NVR (using formula 1, 2, 3 or 4).

11. Precision and Bias

11.1 *Precision*—The precision of the procedure in Practice G 120 is being determined.

11.2 *Bias*—Since there is no accepted reference material for determining the bias for the procedure in Practice G 120 bias has not been determined.

12. Keywords

12.1 contaminant; contamination; extraction; nonvolatile residue; oxygen systems; Soxhlet extraction



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