



Designation: G120 – 15 (Reapproved 2023)

Standard Practice for Determination of Soluble Residual Contamination by Soxhlet Extraction¹

This standard is issued under the fixed designation G120; 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 practice describes a procedure for the determination of residual contamination in materials to be used within or in contact with hardware requiring a high level of cleanliness, such as components for oxygen service, 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 wipers, contaminated test specimens or control coupons, small piece parts (metallic or nonmetallic), etc. When used with proposed consumable cleaning materials (wipers, gloves, etc.), this practice may be used to determine the potential of the proposed solvent to extract contaminants (plasticizers, residual detergents, brighteners, etc.) from the cleaning material 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This practice is under the jurisdiction of ASTM Committee G04 on Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres and is the direct responsibility of Subcommittee G04.02 on Recommended Practices.

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2. Referenced Documents

2.1 ASTM Standards:²

- D1193 Specification for Reagent Water
- E1235 Test Method for Gravimetric Determination of Non-volatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft
- E1560 Test Method for Gravimetric Determination of Non-volatile Residue From Cleanroom Wipers
- E1731 Test Method for Gravimetric Determination of Non-volatile Residue from Cleanroom Gloves
- F331 Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Flash Evaporator)
- G93 Guide for Cleanliness Levels and Cleaning Methods for Materials and Equipment Used in Oxygen-Enriched Environments
- G127 Guide for the Selection of Cleaning Agents for Oxygen-Enriched Systems
- G136 Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction
- G144 Test Method for Determination of Residual Contamination of Materials and Components by Total Carbon Analysis Using a High Temperature Combustion Analyzer

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

3.1.3.1 *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.

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.

3.1.4 *molecular contaminant, n*—non-particulate contamination.

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

3.1.4.2 *Discussion*—Molecular contaminants account for most of the NVR.

3.1.5 *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.

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

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

4. Summary of Practice

4.1 The specimen 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 and 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.

5. Significance and Use

5.1 It is expected that this practice 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 components for oxygen service. Nonmetallic piece parts such as seals and valve seats can be tested as received. **Warning**—If parts being tested are to be subsequently installed in an oxygen-enriched system, residual extraction solvent remaining in the part due to inadequate drying may increase the susceptibility of the system to ignition. The extraction solvent shall be thoroughly removed from the parts prior to service.

5.2 Processing materials such as gloves and wipers, 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 that may be deposited as residues on the surface to be cleaned.

NOTE 1—Test methods that do not require Soxhlet equipment, such as Test Methods E1560 and E1731, may be suitable alternatives for evaluation of processing materials. Test Method G144 with Practice G136 may be suitable for use when the material to be tested is not degraded by sonication. However, results from this test method cannot be directly compared to results from other test methods. Soxhlet extraction may be more aggressive than other extraction methods.

5.3 Wipers or other cleaning supplies can be tested before and after use to determine the amount of contaminant removed from a surface. The type of contaminant removed from the surface may also be determined by qualitative analysis of the extracted NVR using analytical methods such as Fourier Transform Infrared (FTIR). 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.

5.4 The selection of the solvent requires some knowledge of the contaminant (see Practice G93 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.

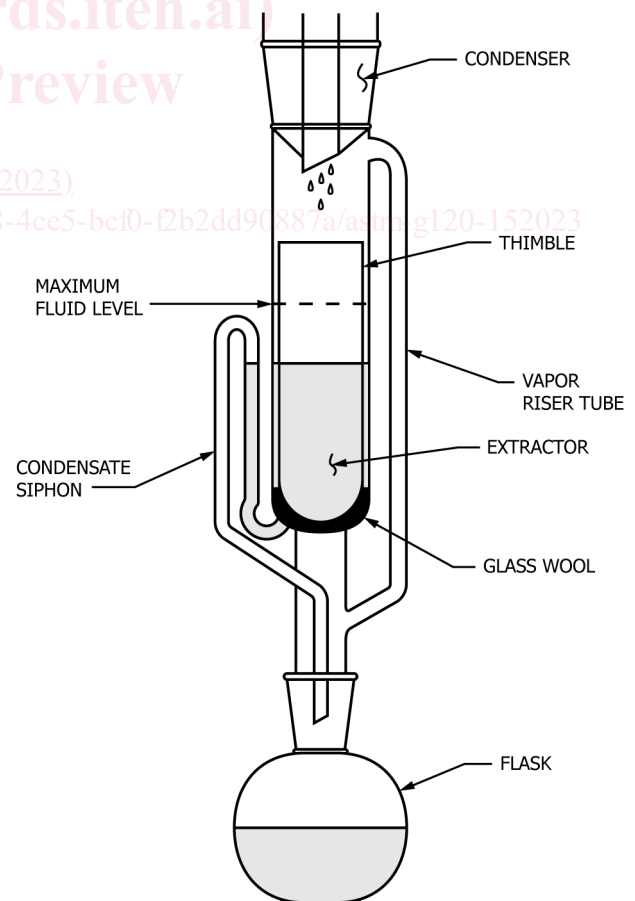


FIG. 1 Soxhlet Extraction Apparatus

6.7 *Evaporator*—rotary.

6.8 500 mL concentrator flask.

NOTE 2—Larger extraction systems may be used for batch extraction of wipers to be used to sample NVR on surfaces or to extract NVR from larger components. The item to be extracted must be small enough to be fully immersed in solvent within the extraction chamber.

7. Reagents

7.1 Examples of solvents commonly used for Soxhlet extraction are ethanol, 2-propanol, 2-butanone, hexane, dichloromethane, propanone, ethyl acetate, and the azeotrope of ethyl acetate/cyclohexane. Different solvents will vary in removal efficiency for different types of contaminants and should be selected based on the contaminants of greatest concern. The solvent used shall be recorded in the test report.

Warning—Solvents such as ethanol, 2-propanol, 2-butanone, hexane, and ethyl acetate are highly flammable. The reader should refer to appropriate safe handling procedures.

NOTE 3—Hydrofluorocarbons and hydrofluoroethers may be acceptable choices but are less effective at removing hydrocarbon-based NVR than ethyl acetate or hexane.

NOTE 4—Ethanol and 2-propanol are the least effective at removing NVR but may be used when they reflect actual solvent usage in service.

7.1.1 When testing processing materials such as gloves and wipers, a solvent that is representative of the service conditions of the material should be selected.

7.1.2 Many polymeric materials are incompatible with solvents. Refer to Guide **G127** for guidance on material compatibility. Extraction may also leach desirable additives, altering the performance of the polymer. Gloves and wipers that are attacked by an extraction solvent should not be used with that solvent in production.

7.1.3 Nonmetallic piece parts should be tested using solvents that are known to be compatible with the material. Cracking, discoloration, or significant weight loss are evidence of solvent attack. Continued release of NVR in successive extraction cycles may also indicate that the material is incompatible with the extraction solvent.

7.1.3.1 For extraction of unfilled nonmetallic material, there must be sufficient insolubility between the material and extraction solvent such that there is no dissolution of the material.

7.1.3.2 For extraction of filled nonmetallic material, the filler must not be soluble in the extraction solvent at the extraction temperature and sufficient insolubility or crosslinking must be present to prevent migration of filler during the extraction, such that the extractant remains clear and free of filler.

7.1.3.3 To evaluate surface contamination on a nonmetallic piece part that is not compatible with extended exposure to the solvent but is compatible with brief exposure, the surface of the part may be sampled by the wipe method described in section **5.3** followed immediately by drying.

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.³ 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. Alternatively, the solvent may be purified by distillation prior to use.

7.3 *Water for extraction*—shall meet the requirements of **D1193**, Type II.

8. Procedure

8.1 Prepare the specimen 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 cm², accurately measure and calculate the area (A) in cm², and weigh the section in grams to the nearest tenth of a milligram (W₁). 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 sample of the cloth, with the same surface area, prior to use according to Test Method **E1235** or Test Method **F331**. Low-linting cotton cleanroom wipers have been found to be acceptable for NVR wipe sampling. Monofilament polyester wipers, while less absorbent, release fewer fibers than cotton wipers and may be preferred in critical applications. For greater accuracy, the wiping cloths should be pre-extracted as a batch prior to use. Record this NVR as W₃ in mg/g or as W₄ 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 (W₁). Determine the dimensions of each strip in cm and record the total surface area of the strips (A) in cm².

NOTE 5—Some gloves are of a coated or layered construction or have different textures applied to the inside and outside surfaces. Because the inside and outside surfaces of these gloves may release different quantities of nonvolatile residue, results using this method may not reflect the actual potential for transfer of contamination from this type of glove to hardware surfaces.

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 cm² and weigh in grams to the nearest tenth of a milligram. Record the surface area (A) and weight (W₁).

8.1.4 To determine the amount of NVR on small piece parts, measure and record the total surface area (A) in cm² and weight in grams to nearest tenth of a milligram (W₁).

8.2 Place the material to be extracted in an extraction thimble or between two glass wool plugs in the extractor. If flotation of the specimen occurs in the extraction thimble,

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.