Designation: G120 – 15

Standard Practice for Determination of Soluble Residual Contamination by Soxhlet Extraction[1]

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E1235 Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft

E1560 Test Method for Gravimetric Determination of Nonvolatile Residue From Cleanroom Wipers

E1731 Test Method for Gravimetric Determination of Nonvolatile Residue from Cleanroom Gloves

F331 Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Flash Evaporator)

G93 Practice for Cleaning Methods and Cleanliness Levels for Material 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.

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.

---

[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.

Current edition approved Oct. 1, 2015. Published October 2015. Originally approved in 1993. Last previous edition approved in 2008 as G120 – 01(2008). DOI: 10.1520/G0120-15.

[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.

3.1.6 *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 ±5 °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.

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

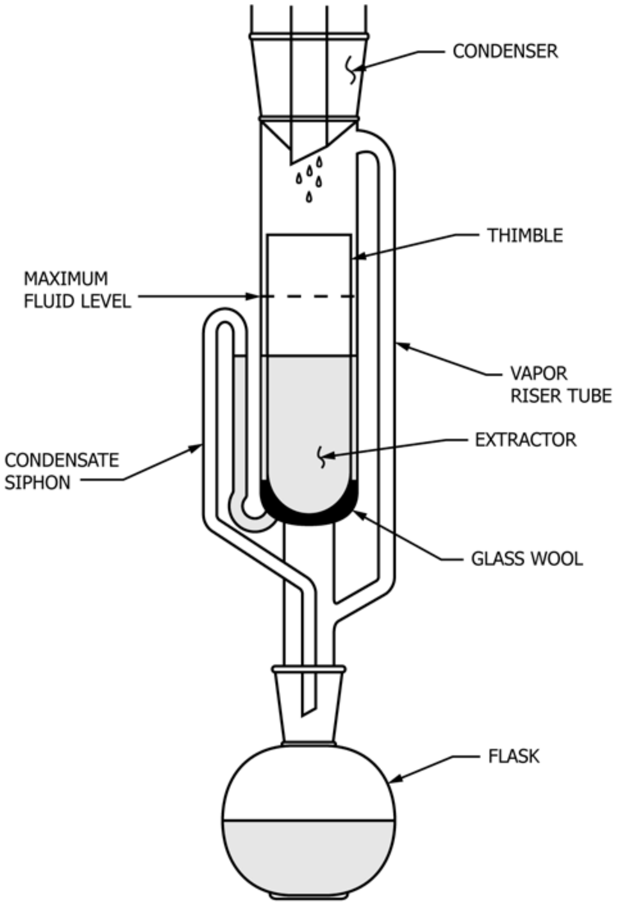

FIG. 1 Soxhlet Extraction Apparatus

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 cm^2, accurately measure and calculate the area (A) in cm^2, 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 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 W_3 in mg/g or as W_4 in mg/cm^2. 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 cm^2.

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 cm^2 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 piece parts, measure and record the total surface area (A) in cm^2 and weight in grams to nearest tenth of a milligram (W_1).

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, cover with a glass wool plug to retain the specimen within the thimble during extraction.

8.2.1 Prior to use, the glass wool used for the plugs shall be washed 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 specimen for six hours.

---

[3] *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.

8.3.1 Extract especially thick or dense materials overnight. If any doubt exists, it is recommended that extractions be performed for progressively longer times (on new specimens from the same lot each time) to verify that the shorter extraction time removed all the extractables. Alternatively, Soxhlet extraction may be repeated several times on the same specimen with a new batch of solvent for each extraction and the NVR graphed from each successive extraction until significant NVR is no longer being removed. The NVR removed by each extraction cycle is added to obtain the total NVR removed.

NOTE 6—The completion of the extraction is verified by *(1)* no further increase in the (combined) NVR weight(s), or *(2)* no further decrease in the dried, extracted specimen weight.

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 Test Method E1235 or Test Method F331.

8.9 The nonvolatile residue may be retained for qualitative analysis to identify contaminant constituents.

9. Calculation

9.1 Calculate the nonvolatile residue as follows:

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

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

where:

NVR = nonvolatile residue, mg/g of specimen,

W_1 = weight of the specimen prior to extraction, (in g), and

W_2 = weight of the nonvolatile residue, (in g).

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

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

where:

NVR = nonvolatile residue, mg/cm_2 of specimen,

A = surface area of the specimen (cm^2), and

W_2 = weight of the nonvolatile residue (g).

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

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

where:

NVR = nonvolatile residue, mg/cm^2 of specimen,

W_1 = weight of the specimen prior to extraction (g),

W_2 = weight of the nonvolatile residue (g), and

W_3 = NVR of new specimen (mg/g).

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

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

where:

NVR = nonvolatile residue, mg/cm^2 of specimen,

W_2 = weight of the nonvolatile residue (g),

A = surface area of the specimen (cm^2), and

W_4 = NVR of new specimen (mg/cm^2).

10. Report

10.1 The report shall include the following:

10.1.1 Identification of the material being extracted (including trade name, proper chemical name, ASTM designation or specification as applicable, 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; and

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

11. Keywords

11.1 contaminant; contamination; extraction; nonvolatile residue; oxygen-enriched; oxygen systems; Soxhlet extraction

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/