



Designation: G 136 – 03

Standard Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction¹

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1. Scope

1.1 This practice may be used to extract nonvolatile and semivolatile residues from materials such as new and used gloves, new and used wipes, component soft goods, and so forth. When used with proposed cleaning materials (wipes, gloves, and so forth), this practice may be used to determine the potential of the proposed solvent or other fluids to extract contaminants (plasticizers, residual detergents, brighteners, and so forth.) and deposit them on the surface being cleaned.

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

1.3 The values stated in SI units are standard.

1.4 *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

D 1193 Specification for Reagent Water

E 1235 Test Method for Gravimetric Determination of Non-volatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft

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

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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 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.

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

3.1.2 *contaminate, v*—a process of contaminating.

3.1.3 *nonvolatile residue (NVR), n*—residual molecular and particulate matter remaining following the filtration and controlled evaporation of liquid containing contaminants.

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*—A molecular contaminant may be uniformly or nonuniformly distributed, or be in the form of droplets. Molecular contaminants account for most of the NVR.

3.1.6 *degas, v*—the process of removing gases from a liquid.

4. Summary of Practice

4.1 A material, glove, hand wipe, and so forth, is placed in a container containing the test fluid. This container is then placed in an ultrasonic cleaning bath and treated for a given period of time at the recommended temperature for the test fluid. This results in either a solution if the contaminant is soluble in the test fluid or an emulsion if the contaminant is not soluble in the test fluid. The test fluid may then be analyzed for nonvolatile residue that was extracted from the test specimen.

4.1.1 In the case of aqueous-based agents, the material may be treated in accordance with Specification D 1193 Type II water or Type II water containing an extracting agent.

4.1.1.1 When Type II water is used, the water and material may be analyzed without further treatment. Typical methods of analysis may include weighing the material before and after treatment or more sophisticated analytical procedures such as total carbon (TC) or high-pressure liquid chromatography.

4.1.1.2 When cleaning agents are used, the materials are rinsed with Type II water after the removal from the cleaning bath and then ultrasonically cleaned in reagent water to ensure the removal of the extracting agent. Typical methods of

analysis may include weighing the material before and after cleaning or more sophisticated analytical procedures such as TC or high-pressure liquid chromatography.

4.1.2 In the case of solvent-based agents, the weight of the material before and after cleaning may be determined or the solvents may be analyzed using infrared spectroscopy, gas chromatography, gas chromatography/mass spectroscopy, or the NVR determined using Test Methods E 1235, F 324, or F 331, as appropriate.

5. Significance and Use

5.1 This practice is suitable for the determination of extractable substances that may be found in materials used in systems or components requiring a high level of cleanliness, such as oxygen systems. Soft goods, such as seals and valve seats, may be tested as received. Gloves and wipes, or samples thereof, to be used in cleaning operations may be evaluated prior to use to ensure that the proposed extracting agent does not extract or deposit chemicals, or both, on the surface to be cleaned.

5.2 Wipes or other cleaning equipment may be tested after use to determine the amount of contaminant removed from a surface.

NOTE 1—The amount of material extracted may be dependent upon the frequency and power density of the ultrasonic unit.

5.3 The extraction efficiency has been shown to vary with the frequency and power density of the ultrasonic unit. The unit, therefore, must be carefully evaluated to optimize the extraction conditions.

6. Apparatus

6.1 *Ultrasonic Bath*, with an operating frequency range from 25 to 90 kHz, a typical power range from 10 to 25 W/L, and a temperature controlled bath capable of maintaining a temperature between ambient and 70°C with an accuracy of $\pm 2^\circ\text{C}$ is to be used.

6.2 *Parts Pans*, stainless steel container with volumes between 1 and 4 L are to be used.

6.3 *A Bracket*, to support the sample pans in the ultrasonic bath is to be used.

NOTE 2—The bracket should be designed to hang in the ultrasonic bath without contact with the bottom.

6.4 *Balance*, a minimum capacity of 50 g with an accuracy of 0.1 mg.

7. Reagents

7.1 *Solvents*—the following may be used: tetrachloroethylene (perchloroethylene), trichloroethylene, methylene chloride, and perfluorinated carbon fluids.

NOTE 3—**Warning:** Follow appropriate safe-handling procedures when using the solvents approved for the use application. Many solvents with low TLVs present hazards to personnel working with them as well as to the systems being cleaned. The removal of these solvents from breathing gas systems must be assured. Many solvents are not considered to be compatible with oxygen and must be completely removed from materials before their use in oxygen systems. The preferred solvent removal method shall be determined by the user.

7.2 *Purity of Water*—The water used shall meet the requirements of Specification D 1193, Type II except that the requirement for a maximum TC of 50 kg/L shall not be required.

7.3 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained and that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Detergents to be used shall be identified by the manufacturer and name (registered trademark, if any).

8. Procedure

8.1 Sample Preparations:

8.1.1 Prepare the sample for placement in the ultrasonic bath.

8.1.1.1 To determine the amount of solvent extractable material in a wiping cloth (new or used), cut out a test section approximately 30 cm square, accurately measure and calculate the area (S), in square centimetres, and determine the mass of the section in grams to the nearest tenth of a milligram (mg). Record the area and mass.

8.1.1.2 If the residue is to be determined on used wiping cloths in an effort to assess the cleanliness of a part or system, an extraction and a nonvolatile residue (NVR) or total carbon (TC) analysis shall be performed as described in 8.2-8.5 on an equivalent sample of unused cloth. Record this NVR as M2 in mg/g or as M3 in mg/cm² or as TC in ppm/g or ppm/cm². The NVR or TC value must be subtracted from that determined for the contaminated cloth.

8.1.1.3 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 areas of the glove, the areas that would typically be exposed to the cleaning solvent, determine the mass in grams to the nearest tenth of a milligram, and record the mass (M1). Determine the dimensions of each strip in centimetres (cm) and record the total surface area of the strips (S) in square centimetres.

8.2 *Parts Pan Preparation*—Clean the stainless steel sample parts pans. Conduct the extraction procedure selected without test articles to verify the cleanliness of the parts pans. Use the same volume of cleaning agent for the verification that will be used on the test articles. Determine the amount of NVR or TC for the parts pan using the analysis procedure that will be used on the actual test articles. Record the amount as the blank (B) for the parts pan and cleaning agent.

8.3 Preliminary Procedure:

8.3.1 If an extracting agent is being used that requires dilution or special preparation, carefully follow the manufacturer's instructions. Use Type II water to prepare the aqueous extracting solutions or as the actual extracting agent.

8.3.2 Place the support bracket in the ultrasonic bath, fill with water to the level specified by the manufacturer, heat the ultrasonic bath to the desired temperature, and degas the water for 10 min.

8.3.3 Place the selected parts pan in the support bracket in the ultrasonic bath.

8.4 Extraction Procedure: