



Designation: **G136 – 03 (Reapproved 2016) G136 – 03 (Reapproved 2016)<sup>ε1</sup>**

## Standard Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction<sup>1</sup>

This standard is issued under the fixed designation G136; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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<sup>ε1</sup> NOTE—Editorial changes made throughout in May 2017.

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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 to be regarded standard. No other units of measurement are included in this 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.*

1.5 *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.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1193 Specification for Reagent Water

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

F324 Test Method for Nonvolatile Residue of Volatile Cleaning Solvents Using the Solvent Purity Meter (Withdrawn 1987)<sup>3</sup>

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

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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.

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<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

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 **D1193** 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 **E1235**, **F324**, or **F331**, 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~~ 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~~ 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 **D1193**, 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~~ ~~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~~ ~~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/cm<sup>2</sup> or as TC in ppm/g or ppm/cm<sup>2</sup>. 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:*

8.4.1 Place the material or part(s) to be extracted in the stainless steel parts pan.

8.4.2 Pour a measured amount of the extracting agent into the stainless steel parts pan sufficient to cover the parts. Cover the parts pan with aluminum foil or a stainless steel lid, place the parts pan and parts in the bracket in the ultrasonic bath, adjust the water level in the bath such that it is above the extracting agent level in the parts pan, and allow the extracting agent and bath temperature to equilibrate to the desired temperature. Alternatively, preheat the parts pan and extracting agent prior to the placement of the materials or parts into the parts pan. Then cover the parts pan with foil and place the parts pan into the bracket in the bath and allow the extracting agent to equilibrate to the temperature of the bath.

8.4.2.1 ~~Extraction~~—The ratio of extraction agent to ~~parts~~ ~~part's~~ surface area ratio shall not exceed 1000 mL/0.1 m<sup>2</sup>; the preferred ratio is 500 mL/0.1 m<sup>2</sup>.

8.4.3 Subject the parts to the ultrasonic bath for 10 min. Perform the sampling procedure as soon as possible, with a maximum time limit of 120 min after turning off the ultrasonic bath.

### 8.5 *Sampling Procedure for Solvent Extracted Parts:*

8.5.1 Remove the parts pan from the ultrasonic bath and remove the cover. Swirl the parts pan to thoroughly mix the solvent.

8.5.2 After swirling, quickly decant the solvent from the parts pan.

8.5.3 Wash the parts pan and parts with a total of 500 mL of fresh solvent in three roughly equal portions, combine with the solvent from 8.5.2, and set aside as the sample for NVR analysis.

8.5.4 Determine the mass (M4) of the nonvolatile residue in milligrams to the nearest tenth of a milligram using Test Methods **E1235**, **F324**, or **F331**. Ensure that the reported NVR is adjusted by subtracting the NVR of an equivalent volume of “blank” solvent.

### 8.6 *Sampling Procedure for Aqueous Extracted Materials and Parts:*

8.6.1 Remove the parts pan from the ultrasonic bath and remove the cover. Swirl the parts pan to mix the extracting agent.

8.6.2 After swirling, quickly decant the extracting agent from the parts pan.