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.

Designation: G136 − 03 (Reapproved 2016)$^{\varepsilon 1}$

Standard Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Editorial changes made throughout in May 2017.

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:*[2]

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)[3]

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.

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

[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] The last approved version of this historical standard is referenced on www.astm.org.

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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 bath capable of maintaining a temperature between ambient and 70°C with an accuracy of ±2°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 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 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/cm^2 or as TC in ppm/g or ppm/cm^2. 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 The ratio of extraction agent to part's surface area shall not exceed 1000 mL/0.1 m^2; the preferred ratio is 500 mL/0.1 m^2.

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.

8.6.3 Wash the parts pan and parts with a total of 500 mL of fresh Type II water in three roughly equal portions and discard unless Type II water was used as the extracting agent. If Type II water was used as the extracting agent, combine the three portions with the water from 8.6.2, and set aside as the sample for analysis. If a surface active compound was used, repeat the procedures in 8.3 – 8.5 using Type II water and use the Type II water as the sample for analysis.

8.6.4 Determine the NVR of the sample using TC or high pressure liquid chromatography (see 4.1.1.2).

9. Report

9.1 Report the following information:

9.1.1 Identification of the part or material being cleaned (including tradename, part number, serial number, proper chemical name, ASTM designation, lot number, batch number, and manufacturer).

9.1.2 Cleaning reagent;

9.1.3 Cleaning time;

9.1.4 Cleaning temperature;

9.1.5 Frequency of the ultrasonic bath, kHz;

9.1.6 Power density of the ultrasonic bath, W/L;

9.1.7 Volume of extracting agent used, mL;

9.1.8 Mass (M1) of parts extracted, g;

9.1.9 Mass (M2) of material extracted from unused wipes, mg/g, or (M3), mg/cm^2, or TC in ppm/g or ppm/cm^2;

9.1.10 Mass (M4) of NVR determined using Test Methods E1235, F324, or F331;

9.1.11 Blank (B) for the parts pan and agent, mg; and,

9.1.12 Surface area (S), cm^2.

10. Keywords

10.1 contaminant; contamination; extraction; nonvolatile residue; oxygen systems; total carbon (TC); ultrasonic extraction

APPENDIX

(Nonmandatory Information)

X1. SELECTION OF ULTRASONIC BATHS

X1.1 *Introduction*—This appendix describes technical information useful to the user in the selection of ultrasonic baths for aqueous extraction and cleaning applications. The following information was graciously provided by Blackstone Ultrasonics[4] and is reprinted here with their permission.

X1.2 Designing an immersible ultrasonic transducer system requires that several factors be taken into account. Each case is unique. The following list will give the reader some idea of the parameters that should be defined. Later, each will again be considered as to its effect on the design of the system.

X1.2.1 *The Tank:*

X1.2.1.1 *Volume*—cubic measure or gallons.

X1.2.1.2 *Shape*—length, width and depth.

X1.2.1.3 *Internal Features*—heaters, agitators, linings, submersible pumps, etc.

X1.2.1.4 *Cleaning Zone*—parts placement and racking.

X1.2.2 *The Parts Being Cleaned:*

X1.2.2.1 *Size*—physical dimensions.

X1.2.2.2 *weight*—weight/density.

X1.2.2.3 *Number per Load or per Unit of Time*—Parts per rack or basket, parts per hour.

X1.2.2.4 *Complexity*—holes, blind holes, internal surfaces, hems, etc.

X1.2.2.5 *Ratio of Part Surface Area to Part Size*—Solid cube versus typical heat exchanger.

X1.2.3 *The Contaminant Being Removed:*

X1.2.3.1 *Removal Difficulty*—Light oil versus buffing compound.

X1.2.3.2 *Thickness of Buildup*—Holes plugged solid versus surface coat.

X1.2.3.3 Solubility of the contaminant and its ability to absorb ultrasound.

X1.2.4 *Process Parameters:*

X1.2.4.1 Typical cleaning time.

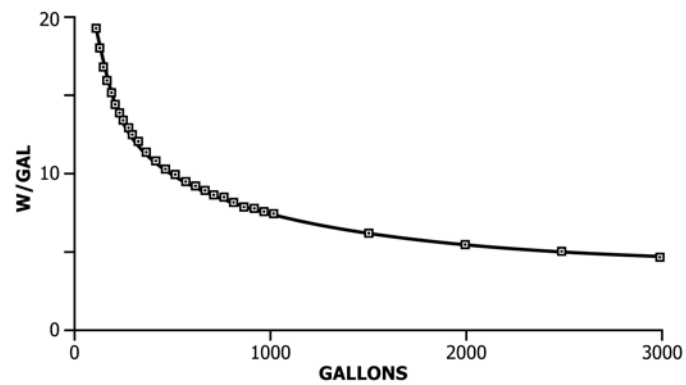
X1.2.4.2 Temperature.

X1.2.4.3 Chemical and concentration.

X1.3 System design includes determination of the number of transducers to be used and their placement in the cleaning tank for maximum cleaning effectiveness.

X1.3.1 *Determination of the Ultrasonic Power Required* (see Fig. X1.1):

X1.3.1.1 Several schemes have been devised for determining the total ultrasonic power required in an ultrasonic cleaning system. Most center around watts of ultrasonic power per some unit of measure. Watts per gallon, watts per square inch of tank bottom, and watts per square inch of surface being cleaned are the ones most often utilized. Meaningful argument can be made for and against each individually, but in practice, all must be used in combination to come up with a properly powered system.



NOTE 1—Over 3000 gal, a minimum of 5 W/gal is recommended.

FIG. X1.1 Watts of Ultrasonic Power Required for Given Tank Volumes

X1.3.1.2 Watts per gallon of cleaning solution is a good starting point for the determination of the number of transducers for a given cleaning system. It is relatively easy to express and calculate. The number of gallons is calculated based on the volume of the tank (7.5 gallons per cubic foot) then divided into the total ultrasonic power. The result is watts per gallon.

X1.3.2 *Determining the Number of Transducers Required* (see Fig. X1.2):

X1.3.2.1 Once the approximate number of watts per gallon has been determined, calculation of the number of transducers required is an easy matter. One simply multiplies the number of gallons times the number of watts per gallon required for that number of gallons and then divides by the number of watts per transducer and rounds to the nearest whole number to find the

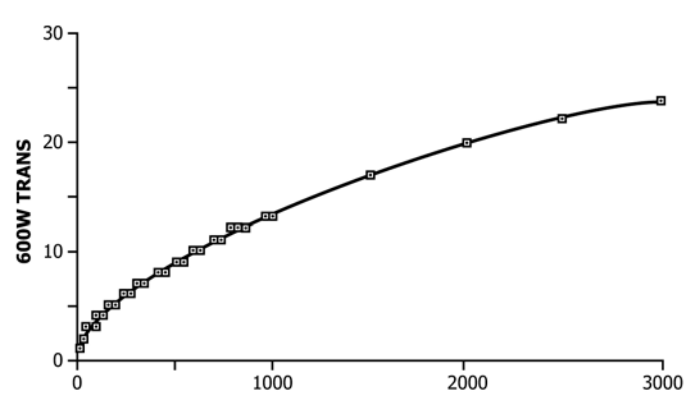


FIG. X1.2 Number of Transducers Required for Given Tank Volume

[4] Blackstone Ultrasonics, P.O. Box 220, 9 North Main St., Jamestown, NY 14702-0220.

number of transducers. The following chart has been developed based on the power per transducer being 600 W.

X1.3.2.2 The number of watts per gallon required in a cleaning system diminishes as the size of the tank is increased. Small ultrasonic cleaners with a capacity of one or two pints may be powered with the equivalent of up to several hundred watts per gallon while a system with several thousand gallons of cleaning solution may be very effective with as little as 3 or 4 W/gal.

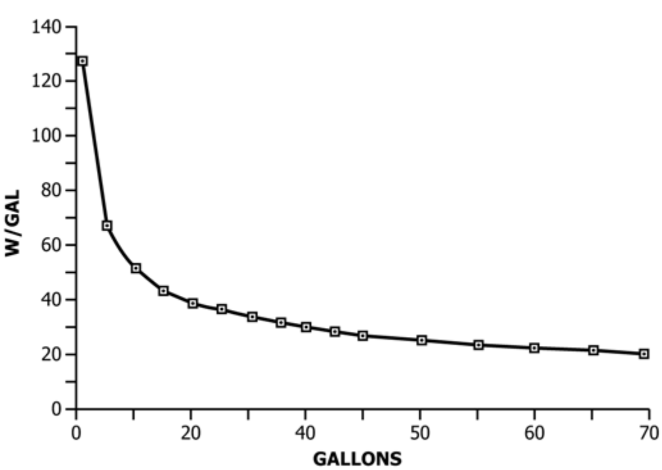
X1.3.2.3 *This phenomenon can be attributed to several factors:*

(1) In a large tank, less energy is absorbed into the tank walls which have proportionately less surface area than those in a smaller tank.

(2) In a large tank, ultrasonic energy travels unimpeded through the volume of liquid for greater distances and is reflected by large flat surfaces including the sides and bottom of the tank as well as the liquid/air interface at the top. In a small tank, frequent and inefficient reflection may lead to rapid dissipation of energy due to dampening effects and destructive interference.

(3) In small tanks, the loading factor (ratio of the volume or surface area of the parts being cleaned to the volume of the tank) is generally higher leading to greater utilization of the energy available. Similar loading factors are not achieved in typical large cleaning systems.

X1.3.2.4 Taking the above into account, the Fig. X1.3 was developed as a guideline for the number of watts per gallon required for tanks up to 100 gal. The numbers are based on watts being the average RMS input watts to the transducer(s) at an ultrasonic frequency of between 20 and 40 kHz. The chart assumes a cleaning operation requiring average ultrasonic power and average tank loading.



NOTE 1—This curve was generated by taking a number of known successful installations and fitting a curve to the data. As tank capacity is extended further, the number of watts per gallon required continues to decrease at a diminishing rate.

FIG. X1.3 Watts per Gallon Required for Given Tank Volume Up to 100 Gallons

X1.4 *Other Considerations:*

X1.4.1 It was stated earlier that the measure of watts per gallon in a cleaning tank is a good starting point for determining the ultrasonic power required but not sufficient without considering other factors.

X1.4.2 *Tank Geometry*—The geometry of a cleaning tank can be such that even with the number of transducers required to give the recommended number of watts per gallon, the volume of the tank will not be adequately provided with ultrasonic energy. One example is a very narrow, long tank. Assume a tank 1 ft × 1 ft × 10 ft. Although three transducers would supply sufficient ultrasonic power for this 75-gal cleaning tank, the energy would not be adequately distributed due to the length of the tank. In this case, the geometry of the tank requires at least four transducers to give an even distribution of ultrasonic energy.

X1.4.3 *Tank Construction*—Tanks with complex interior surfaces or linings require added power. These features tend to absorb ultrasonic energy and prevent effective reflection. In some instances the addition of a special reflecting surface on the wall opposite the ultrasonic transducers is indicated to enhance reflections.

X1.4.4 *Tank Loading Factor*—The greater the load in a tank, the more power required. A system used to clean small parts such as kitchen utensils (forks, spatulas, etc.) hung 200 per rack will require less ultrasonic power than the same size system used to clean racks of 20 or 30 zinc die castings weighing 10 lb each. The key factors here are the weight of the parts and the number being cleaned at one time. A heavily loaded tank may require up to twice the power of one with a lower loading factor.

X1.4.5 *The Parts Being Cleaned*—The nature of the parts being cleaned can have a great bearing on the amount of power required in a cleaning system. Simple parts with relatively little surface area are easiest to clean. As complexity grows effective cleaning requires higher ultrasonic intensity. Blind holes and internal cavities provide the first level of complexity and may require up to a 25 % increase in power over the level required for the simplest of parts. As the ratio of surface area to volume increases, cleaning becomes much more difficult. A typical heat exchanger including fins is representative of such a part configuration and may require 50 % more power than the simplest parts. It is a case such as this which may support the validity of the watts per square inch of surface being cleaned measure.

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