Designation: G131 – 96 (Reapproved 2016)^{ε1}

Standard Practice for Cleaning of Materials and Components by Ultrasonic Techniques¹

This standard is issued under the fixed designation G131; 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.

ε¹ NOTE—Editorial corrections were made throughout in May 2017.

1. Scope

- 1.1 This practice covers a procedure for the cleaning of materials and components used in systems requiring a high level of cleanliness, such as oxygen, by ultrasonic techniques.
- 1.2 This practice may be used for cleaning small parts, components, softgoods, etc.
 - 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. Specific precautionary statements are given in Note 1.
- 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
- F311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters
- F324 Test Method for Nonvolatile Residue of Volatile

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.

- 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)
- G121 Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents
- G122 Test Method for Evaluating the Effectiveness of Cleaning Agents

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 applying a contaminant.
- 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, which is used to verify the validity of the method or part thereof.
- 3.1.3.1 *Discussion*—In this practice, the control coupon will be contaminated in the same manner as the test coupons and will be subjected to the identical cleaning procedure.
 - 3.1.4 degas, v—the process of removing gases from a liquid.
- 3.1.5 *nonvolatile residue (NVR), n*—residual molecular and particulate matter remaining following the filtration and controlled evaporation of a solvent containing contaminants.
- 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 A part, material or component is placed in a container containing the cleaning agent. This container is then placed in an ultrasonic cleaner and treated for a given period of time at

³ The last approved version of this historical standard is referenced on www.astm.org.

the recommended temperature for the cleaning agent. This results in a solution if the contaminant is soluble in the test fluid or a emulsion if the contaminant is not soluble in the test fluid. The cleaning solution combined with the rinse solutions may then be analyzed for particulate, NVR, or total carbon (TC).

- 4.1.1 In the case of aqueous-based agents, the parts are rinsed after the removal from the cleaning bath and ultrasonically cleaned in reagent water to provide a solution for TC analysis using G TC.
- 4.1.2 In the case of solvent-based agents, the parts are rinsed with fresh solvent, which is collected and combined with the solvent used in the cleaning process, and the NVR determined using Test Method E1235, Test Method F324, or Test Method F331, as appropriate.
- 4.1.3 Particulate analyses may be performed by filtering the final cleaning solution. The particles captured by the filter are then counted using Practice F311.

5. Significance and Use

- 5.1 This practice is suitable for the removal of contaminants found on materials, parts, and components used in systems requiring a high level of cleanliness, such as oxygen. Parts shall have been precleaned to remove visible contaminants prior to using this procedure. Softgoods such as seals and valve seats may be cleaned without precleaning.
- 5.2 This procedure may also be used as the cleanliness verification technique for coupons used during cleaning effectiveness tests as in Test Method G122.
- 5.3 The cleaning efficiency has been shown to vary with the frequency and power density of the ultrasonic unit. Low frequencies in the 20 to 25 kilohertz range have been found to damage soft metals such as aluminum and silver. Therefore, the specifications of the unit and the frequencies available must be considered in order to optimize the cleaning conditions without damaging the parts.

6. Apparatus

- 6.1 *Ultrasonic Cleaner*, with an operating frequency range between 25 and 90 kHz, a typical power range between 10 and 25 W/L, and a temperature-controlled bath capable of maintaining a temperature between ambient and 70°C with an accuracy of 2°C.
- 6.2 Parts Pans, stainless steel container with volumes between 1 and 4 L.
- 6.3 *Bracket*, stainless steel device capable of supporting the parts pans in the ultrasonic bath.

 ${\it Note}\ 1$ —The bracket should be designed to hang in the ultrasonic bath without contact with the bottom.

7. Reagents

- 7.1 Solvents such as the following may be used: tetrachloroethylene (perchloroethylene), trichloroethylene, methylene chloride, and perfluorinated carbon fluids.
- Note 2—Warning: Solvents such as tetrachloroethylene (perchloroethylene), trichloroethylene, and methylene chloride have relative low threshold limit values and the user should refer to appropriate safe

handling procedures, particularly in open tanks. Many solvents are not considered to be compatible with oxygen and must be completely removed from cleaned components prior to the use of these components in oxygen systems. The preferred method of removal shall be determined by the user.

- 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.⁴ 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. Detergents used shall be identified by manufacturer and name (registered trademark, if any).
- 7.3 *Purity of Water*—The water used shall meet the requirements of Specification D1193, Type II.

8. Procedure

- 8.1 Sample Preparation:
- 8.1.1 If cleanliness verification is to be performed on control or test coupons, prepare the coupons in accordance with Practice G121.
- 8.1.2 If cleanliness verification is to be performed on small parts, measure the total surface area (S) in square centimetres or the mass in grams, or both, as applicable, to the nearest tenth of a milligram (MI). Record the surface area (S) and mass (MI).
 - 8.2 Preliminary Procedure:
- 8.2.1 If a cleaning agent is being used that requires dilution or special preparation, carefully follow the manufacturer's instructions. Use Type II water to prepare the aqueous cleaning agent solutions or as the actual cleaning agent.
- Note 3—It has been found that many common hydrocarbon based lubricants are effectively removed to acceptable levels using Type II water at 50 to 55 °C. Contaminants more difficult to remove, such as fluorinated or silicone based lubricants, have typically been found to require the use of surface active agents. Use Test Method G122 to evaluate the cleaning effectiveness of the proposed cleaning agent.
- 8.2.2 Fill the ultrasonic bath to the level specified by the manufacturer with water. Place the support bracket in the ultrasonic bath, heat the ultrasonic bath to the desired temperature, and degas the water for 10 min.
- 8.2.3 Clean the stainless steel sample parts pan to be used. Conduct the sampling procedure using the selected cleaning agent without parts to verify the cleanliness of the parts pan. Use the same sampling and analysis procedures that will be used on the actual parts. Determine the contamination level of the parts pan, the blank value (B), which shall be less than the allowable contamination level for the items being cleaned or extracted. If the contamination level of the parts pan is greater than that specified for the parts, reclean the parts pan until the contamination level is less than the allowable contamination specified for the parts.

⁴ 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 Analar 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 Cleaning Procedure:
- 8.3.1 Place the material or part(s) being cleaned in the stainless steel parts pan.
- 8.3.2 Pour a measured amount of the cleaning agent into the stainless steel cleaning pan sufficient to cover the parts. Cover the parts pan with aluminum foil or a stainless steel lid, place the parts pan in the bracket in the ultrasonic bath, adjust the water level in the bath such that it is above the cleaning agent level in the parts pan, and allow the cleaning agent and bath temperature to equilibrate to the desired cleaning temperature. Alternatively, preheat the parts pan and cleaning agent prior to the placement of the materials or parts into the parts pan. Then cover the parts pan with foil and place into the bracket in the bath and allow the cleaning agent to equilibrate to the temperature of the bath.
- 8.3.2.1 Cleaning agent to parts surface area ratio shall not exceed 1000 mL/0.1 m²; the preferred ratio is 500 mL/0.1 m².
- 8.3.3 Clean the parts in the ultrasonic bath for 10 min. If an aqueous detergent or surfactant solution was used for cleaning, thoroughly rinse the parts with Type II water and then perform the ultrasonic procedure with fresh Type II water. Perform the sampling procedure as soon as possible within a maximum time limit of 120 min after turning off the ultrasonic cleaner.
 - 8.4 Sampling Procedure for Solvent Cleaned Parts:
- 8.4.1 Remove the parts pan from the ultrasonic bath and remove the cover. Swirl the parts pan to mix the solvent.
- 8.4.2 After swirling, quickly decant the solvent from the parts pan.
- 8.4.3 Wash the parts pan and parts with 500 mL of fresh solvent in three roughly equal portions and combine with the solvent decanted from 8.4.2. Determine the particulate contamination analysis using Practice F311. Use the filtrate from the particulate analysis as the sample for NVR analysis.
- 8.4.4 Determine and record the mass (M²) of the nonvolatile residue in milligrams to the nearest tenth of a milligram using

- Test Method E1235, Test Methods F324, or F331. Ensure that the reported NVR is adjusted to subtract the NVR of an equivalent volume of "blank" solvent.
- 8.5 Sampling Procedure for Aqueous Cleaned Materials and Parts
- 8.5.1 Remove the parts pan from the ultrasonic bath and remove the cover. Swirl the parts pan to mix the Type II water.
- 8.5.2 After swirling, quickly decant the Type II water from the parts pan.
- 8.5.3 Wash the parts pan and parts with 500 mL of Type II water in three roughly equal portions and combine with the Type II water from 8.5.2.
- 8.5.4 Use the combined volumes of water from 8.5.3 to determine the TC of the sample using G TC.

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 cleaner, W/L;
 - 9.1.7 Volume of cleaning reagent used;
 - 9.1.8 Mass (M1) of parts cleaned, g;
 - 9.1.9 Surface area, cm²; and
 - 9.1.10 Mass (M2) of thr NVR, g.

10. Keywords

10.1 cleaning; contamination; contaminant; nonvolatile residue; NVR; oxygen systems; TC; total carbon; ultrasonic cleaning

APPENDIX

(Nonmandatory Information)

X1. SELECTION OF ULTRASONIC BATHS

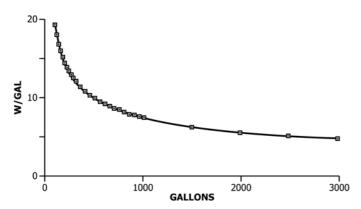
- X1.1 Introduction—This appendix describes technical information useful in the selection of ultrasonic baths for aqueous extraction and cleaning applications. The following information was graciously provided by Blackstone Ultrasonics⁵ and is reprinted here with their permission.
- X1.2 Designing an 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 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.

⁵ Blackstone Ultrasonics, P.O. Box 220, 9 North Main St., Jamestown, NY 14702-0220.

- 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 most be used in combination to come up with a properly powered system.
- 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 gal/ft³) then divided into the total ultrasonic power. The result is W/gal.
- 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



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

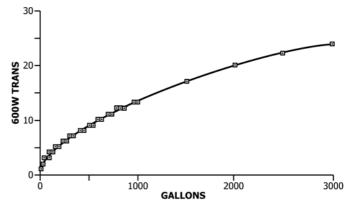


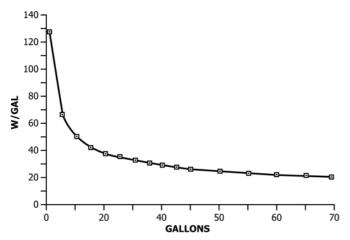
FIG. X1.2 Number of Transducers Required for Given Tank Volume

number of transducers. Fig. X1.2 was 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 pt 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.

X1.4 Other Considerations:

- X1.4.1 It was stated earlier that the measure of watts per gallon in a cleaning tank, although it is a good starting point for determining the ultrasonic power required, is not sufficient without considering a number of 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



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 gal.

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 \times 1 ft \times 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 will be required. A system used to clean small parts such as kitchen utensils (forks, spatulas, etc.) hung 200 per racks 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 mentioned at the outset of this standard.

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