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Standard Test Method for Evaluating the Effectiveness of Cleaning Agents[1]

This standard is issued under the fixed designation G122; 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 correction made in October 2015.

INTRODUCTION

Many systems require a high degree of cleanliness. For example, gaseous and liquid oxygen systems must be clean, particularly of hydrocarbons, to avoid the potential hazard of a reaction and subsequent fire or explosion. Typically, chlorinated solvents have been used to clean systems and equipment that must be free of hydrocarbons and other contaminants. Environmental concerns dictate that suitable replacements are needed. This test method presents a procedure that may be used to evaluate candidate aqueous or non aqueous cleaning agents.

1. Scope

1.1 This test method covers a procedure for evaluating the capability of cleaning agents and processes to remove contamination to the desired level.

1.2 The test coupons provide a relatively rough surface to which contamination can easily adhere.

1.3 The capability of a particular cleaning agent depends upon the method by which it is used and the characteristics of the article being cleaned, such as size, shape, and material. Final evaluation of the cleaning agent should include testing of actual products and production process.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* Specific precautionary statements are given in Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

G94 Guide for Evaluating Metals for Oxygen Service

G121 Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents

2.2 *ANSI Standard:*[3]

D46.1 Surface Texture (Surface Roughness, Waviness, Lay)

3. Terminology

3.1 *Definitions:*

3.1.1 *cleaning effectiveness factor (CEF), n*—the fraction of contaminant removed from an initially contaminated test coupon and determined by gravimetric techniques.

3.1.2 *residual contamination, R_c, n*—the absolute mass of contaminant remaining after the cleaning process and expressed in milligrams per square centimetre of area or optionally as milligrams per square foot.

3.1.3 *surface roughness, R_A, n*—the arithmetic average deviation of the surface profile from the centerline, normally reported in micrometres.

[1] This test method 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 G04.01 on Test Methods.

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

4. Summary of Test Method

4.1 This test method provides quantitative results as to the ability of a specific cleaning agent/process for removing selected contaminants from standard coupons. The coupons used for testing are prepared in accordance with Practice G121. Cleaning is performed using a cleaning tank with or without ultrasonic agitation, elevated temperature or other cleaning enhancement features and depends on the manufacturer's instructions. The effectiveness of the cleaning process is represented as CEF, the cleaning effectiveness factor, that is the fraction of the contaminant removed as determined by gravimetric techniques. A control coupon is used to account for any corrosion or material removal effects due to the cleaning agent/process.

5. Significance and Use

5.1 The purpose of this test method is to define a procedure for evaluating the capability of cleaning agents to clean metallic coupons. Based on the outcome of the testing, suitable cleaning agents may be selected for cleaning in general and for oxygen service in particular.

5.2 The cleaning parameters can be changed and the test method can be repeated. The usual cleaning parameters include cleaning agent concentration, temperature, and time; type and strength of ultrasonic energy or agitation, if used, and others.

NOTE 1—Usual cleaning parameters are based on the manufacturer's recommendations.

6. Apparatus

6.1 *Materials:*

6.1.1 *Test Coupon,* prepared in accordance with Practice G121. The mass of the coupon is approximately 30 to 45 g but will vary significantly for each selected material. Typical materials used in oxygen systems are described in Guide G94.

6.1.2 *Control Coupon*—This is uncontaminated and is subjected to the identical cleaning procedure as the contaminated coupons and serves to evaluate corrosion anderosion of the test coupons.

6.1.3 *Cleaning Agent,* prepared according to the manufacturer's instructions. Specification D1193 Type II water shall be used for preparing aqueous solutions.

6.2 *Equipment:*

6.2.1 *Cleaning Tank,* A vessel of sufficient size to conduct a number of evaluations simultaneously. Testing is enhanced by having automatic temperature and time controls. A cleaning tank with ultrasonics may be used.

6.2.2 *Balance,* accuracy to 0.1 mg. However, 0.01 mg accuracy is desirable to detect contamination levels of 10 mg/m^2 (1 mg/ft^2) or less.

6.2.3 *Beaker Holder*—A device to support beakers in the ultrasonic cleaner tank such that the beakers do not contact the bottom and sides of the tank.

7. Test Procedure

7.1 Prepare a minimum of six test coupons by Practice G121.

7.2 Indicate the masses of coupons in grams as *MXy* where *X* is the coupon designation (number, letter, or name) and $y = 1$ indicates a clean coupon, $y = 2$ indicates a contaminated coupon and $y = 3$ indicates a coupon after cleaning.

7.3 Designate one coupon as the control coupon to undergo cleaning without contamination.

7.4 Measure the mass of the control and test coupons (recording them as *MX*1 as previously defined).

7.5 Contaminate five test coupons in accordance with Practice G121.

7.6 Measure the mass of all contaminated test coupons (recording them as *MX*2 as previously defined).

7.7 Process the control coupon in the test cleaning solution separately from the contaminated test coupons.

7.8 The contaminated test coupons can be processed in independent beakers held in the cleaning tank or as a batch in a single beaker.

7.9 Clean the test and control coupons in the candidate cleaning agent by the manufacturer's procedure or selected procedure.

7.9.1 Prepare the cleaning agent in accordance with the manufacturer's recommendations.

7.9.2 Select beakers of suitable size to accommodate the test coupons and fit the beaker holder.

7.9.3 Wash the beakers thoroughly with a solution of liquid, surface-active cleaning agent in hot water and rinse with type II water.

7.9.4 Fill the beakers with the cleaning agent solution to a level that will ensure the test coupons are submerged.

7.9.5 Fill the cleaning tank to its operating level with the transfer fluid and preheat to desired test temperature.

7.9.6 Place the beakers in the beaker holder in the tank so that the liquid levels in the tank and beakers are approximately equal.

7.9.7 Allow the temperatures of the tank fluid and cleaning agent in the beakers to equilibrate at the desired temperature.

7.9.8 Suspend the test coupons and control coupon in the cleaning agent, using a wire hook of the same material as the coupon or a compatible material. Position the coupons such that they do not touch the beaker or one another.

7.9.9 Begin agitation or sonication in the cleaning process and start the timer.

7.9.10 Upon completing the required cleaning time, discontinue the agitation or sonication, and remove the coupons from the cleaning agent.

7.9.11 Rinse the test coupon in accordance with the manufacturer's recommendations.

7.9.12 Allow the suspended coupons to dry overnight or in a forced convection oven for one hour.

NOTE 2—**Warning:** Do not place test coupons directly in the oven after application of the solution containing the contaminant. A fire may result if the solvent is flammable or rapid evaporation of the solvent may cause spattering of the contaminant thereby reducing the amount of contaminant on the test coupon. It is recommended that the test coupons be air dried until no traces of a liquid phase are visible.

7.9.13 Determine the final mass of each test coupon (recording them as *MX*3 as previously defined), including the control coupon.

8. Calculation

8.1 *Validation of Procedure*—Examine the control coupons to determine whether they lost mass (such as might occur if there was corrosion occurring, if the coupons were dissolving, or if the standard cleaning procedure used prior to contamination had left residue on the coupons); gained mass (such as might occur if the solution was plating a material on their surfaces, or was depositing contaminant rather than removing it) or exhibited the same mass. The simplest valid test procedure is one in which there is no change in the control-coupon's mass to within the measurement error of the balance.

8.1.1 If the control coupon is designated *MC*, and, if $|MC3 - MC1| <$ balance error, then the experiment is valid. Proceed to calculate a cleaning effectiveness factor.

8.1.2 If $|MC3 - MC1|$ is greater than the balance error, the test may be considered to be suspect and the reason for the mass change should be investigated.

8.2 *Cleaning Effectiveness Factor (CEF):*

8.2.1 The cleaning effectiveness factor indicates the fractional contaminant that was removed during cleaning (for example, $CEF = 0.9$ indicates that 90 % of the contaminant was removed).

$$\bullet CEF = \frac{MX2 - MX3}{MX2 - MX1} \quad (1)$$

where:

$MX2 - MX3$ = the mass of contaminant removed, and
$MX2 - MX1$ = the mass of contaminant applied.

8.2.2 Calculate the *CEF* for each test coupon.

8.2.3 Calculate the average *CEF* by arithmetic mean.

8.3 *Residual Contamination (RC):*

8.3.1 A cleaning agent does not necessarily remove a fixed fraction of the contamination on a given surface. In some cases, it cleans a surface to a constant residual cleanliness level. For example, sometimes the cleaned surface will exhibit a layer of organic material that has remained after a fluid vehicle has dried, and a constant *RC* for varying initial contamination levels suggests this may be happening.

8.3.2 Calculate the contaminated area (*S*) of each coupon in square centimeters.

8.3.3 Calculate the residual contamination that is $(MX3 - MX1)$ in grams.

8.3.4 Using the equation $RC = (MX3 - MX1)/S$, calculate the value of *RC* for each coupon (milligrams/centimetre^2).

8.3.5 Determine an average *RC* in mg/cm^2.

8.3.6 As an option, *RC* can be calculated in mg/ft^2.

9. Report

9.1 Because of the many variables involved in conducting a cleaning test program, it is necessary that all data be carefully documented.

9.2 Report the following information, as applicable:

9.2.1 Date of test,
9.2.2 Technician,
9.2.3 Contaminant identification, and
9.2.4 *Coupon Data Refer to Practice* G121*:*
9.2.4.1 Identification number of each coupon,
9.2.4.2 Material,
9.2.4.3 Surface roughness, (R_A) micrometres, and
9.2.4.4 Coupon Contaminated surface areas.
9.2.5 *Cleaning Data:*
9.2.5.1 Cleaning agent identification,
9.2.5.2 Concentration of cleaning agent,
9.2.5.3 pH of diluted cleaning agent,
9.2.5.4 Ultrasonic, soak, or agitation,
9.2.5.5 Time,
9.2.5.6 Temperature,
9.2.5.7 Level of ultrasonic frequency (kHz), and
9.2.5.8 Power density in watts per litre.
9.2.6 *Rinsing Data:*
9.2.6.1 Agent,
9.2.6.2 Time,
9.2.6.3 Temperature,
9.2.6.4 Number of rinses, and
9.2.6.5 Agitation method (if any).
9.2.7 *Drying Data:*
9.2.7.1 Method,
9.2.7.2 Time, and
9.2.7.3 Temperature.
9.2.8 *Test Data:*
9.2.8.1 Initial mass of each coupon, including control coupon, *MX*1 and *MC*1,
9.2.8.2 Mass of each coupon with contaminant, *MX*2,
9.2.8.3 Mass of each cleaned coupon after cleaning, *MX*3, and
9.2.8.4 Mass of control coupon after cleaning, *MC*3.
9.2.8.5 Report $|MC3 - MC1|$ and give comparison to balance error.
9.2.8.6 Report $(MX2 - MX3)$ and $(MX2 - MX1)$ and *CEF* for each test coupon.
9.2.8.7 Report average *CEF*.
9.2.8.8 Report area of contamination in square centimetres.
9.2.8.9 Report $(MX3 - MX1)$ and *RC* in milligrams/square centimetre.
9.2.8.10 Report average *RC* (mg/cm^2).
9.2.8.11 Report average *RC* in milligrams/foot^2 (optional).

10. Precision and Bias

10.1 An interlaboratory study of the cleaning effectiveness factor (CEF) was conducted using the general test protocol of Practice E691 in six participating laboratories with three materials. However, not every laboratory evaluated every material.[4]

10.1.1 The terms repeatability limit and reproducibility limit in Table 1 are used as specified in Practice E177.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure for measuring the CEF in this test method, bias has not been determined.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:G04-1001.

TABLE 1 CEF—Precision[A]

Material	CEF Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
A	0.9866	0.0154	0.0197	0.0432	0.0552
B	0.9531	0.0404	0.0507	0.1131	0.1420
C	0.4074	0.1008	0.1146	0.2821	0.3208

[A] The table was calculated using the relationship: Limit = 2.8 × standard deviation.

11. Keywords

11.1 cleaning agents; cleaning evaluation; cleaning process; contaminant; oxygen; oxygen systems; reagent; solvent