Designation: G121 – 98 (Reapproved 2015)ε1

Standard Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents[1]

This standard is issued under the fixed designation G121; 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 changes were made throughout in October 2015.

1. Scope

1.1 This practice describes the procedure for the preparation of single- and double-sided contaminated metallic test coupons for the evaluation of cleaning agents. It is applicable for the evaluation of cleaning agents proposed for the cleaning of oxygen-enriched systems and components. It also is applicable to other systems where contamination is a concern.

1.2 Several classes of contaminants most likely to be found in oxygen-enriched systems and components are identified. However, if the user of this practice has identified contaminants not included in these classes, such identified contaminants may be substituted for the preparation of the test coupons.

1.3 Preparation of nonmetallic substrates is not addressed, although similar methodology may be used. Solvent and cleaning agent compatibility with the nonmetallic substrate should be verified prior to the preparation of the test coupons. Typical nonmetallic materials utilized in oxygen systems are contained in Guide G63.

1.4 *This practice may involve hazardous materials, operations, and equipment. This practice does not purport to address all of the safety concerns associated with its use. It is the responsibility of whomever uses this practice to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

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

F303 Practices for Sampling for Particles in Aerospace Fluids and Components

F312 Test Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters

F324 Test Method for Nonvolatile Residue of Volatile Cleaning Solvents Using the Solvent Purity Meter (Withdrawn 1987)[3]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)

G63 Guide for Evaluating Nonmetallic Materials for Oxygen Service

G94 Guide for Evaluating Metals for Oxygen Service

2.2 *ANSI:*

B 46.1 Surface Texture (Surface Roughness, Waviness, and Lay)

3. Terminology

3.1 *Definitions:*

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

3.1.2 *contamination, n*—a process of contaminating.

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

3.1.4 *nonvolatile residue (NVR), n*—residual molecular and particulate matter remaining following the filtration of a solvent containing contaminants and evaporation of the solvent at a specified temperature.

3.1.5 *particle (particulate contaminant), n*—a piece of matter in a solid state with observable length, width, and thickness.

3.1.5.1 *Discussion*—The size of a particle is usually defined by its great dimension and is specified in micrometres.

[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.

Current edition approved Sept. 1, 2004. Published October 2015. Originally approved in 1993. Last previous edition approved in 2010 as G121 – 98 (2010)ε1. DOI: 10.1520/G0121-98R15E01.

[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.

3.1.6 *molecular contaminant (nonparticulate contamination), n*—the molecular contaminant may be in a gaseous, liquid, or solid state.

3.1.6.1 *Discussion*—It may be uniformly or nonuniformly distributed, or be in the form of droplets. Molecular contaminants account for most of the NVR.

3.1.7 *blank, n*—the contamination level of the fluid when the test coupon is omitted.

3.1.7.1 *Discussion*—Sometimes referred to as "background" level.

3.1.8 *control coupon (witness coupon), n*—a coupon made from the same material as the test coupons, but in this test method is not coated with the contaminant.

4. Summary of Practice

4.1 A solution of the contaminant is applied to either one side or both sides of the precleaned test coupons and dried under standard conditions. The amount of contaminant on the test coupons is determined. Nonmetallic material test coupons used as inserts, seats, seals, etc. may also be prepared by this procedure and are evaluated in the as-used condition.

4.2 Three methods of coupon preparation are used:
Method A, NVR sample, single side
Method B, NVR sample, double side
Method C, NVR and particulate sample

5. Significance and Use

5.1 This practice will be suitable to direct the preparation of test coupons with a known amount of contaminant on the surface. A standard test coupon is described and a list of contaminants that have typically been found in oxygen-enriched systems and components is provided.

5.2 These coupons shall be used in the evaluation of cleaning agents for oxygen-enriched systems and components. This will permit direct comparison within and between test facilities.

5.3 Materials used in other fluid handling systems such as nitrogen, helium, hydrogen, gasoline, etc. may also be prepared for evaluation by this practice.

6. Apparatus

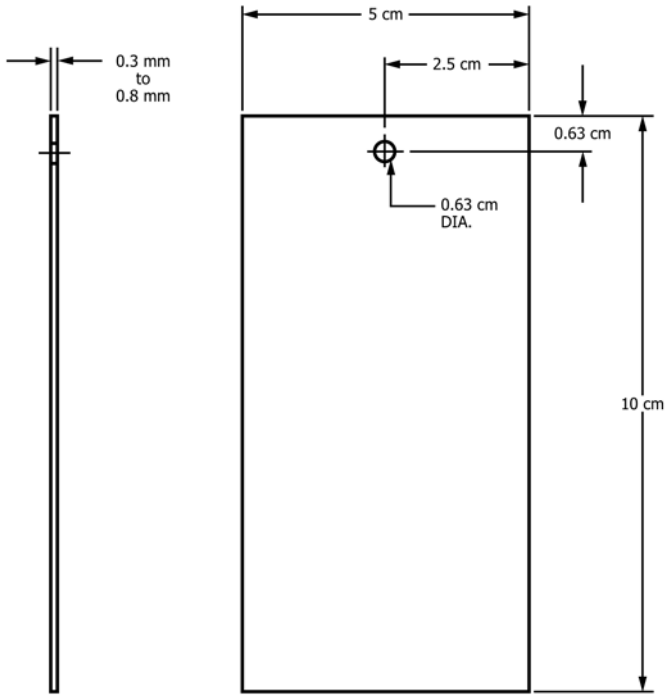
6.1 *Test Coupon*—Metal panels of the same material as the component part to be cleaned. Other alloys that may be used if the specific alloy is unknown are included in Guide G94. The coupon configuration is shown in Fig. 1.

NOTE 1—The surface finish of the test coupon should be the same as the part to be cleaned.

6.2 *Balance-Range* to a minimum of 50 g with an 0.1-mg accuracy capable of weighing to ±0.1 mg.

6.3 *Oven-Convection,* capable of maintaining 50°C ± 5°C.

6.4 *Spray Applicator*—Capable of applying an even coat of contaminant; that is, an artist's airbrush, perfume atomizer, or a spray device such as that used with window or tile cleaners has been found to apply an even coating of the contaminant in a controlled manner.



NOTE: ALL SHARP EDGES SHOULD BE BROKEN AND DEBURRED.

FIG. 1 Standard Test Coupon

6.5 Other standard equipment such as a vacuum filtration system, solvent resistant filters, gloves, laboratory glassware, syringes, pipettes, desiccator, laboratory tongs, tweezers, and wire.

7. Reagents

7.1 Contaminant materials-general classes of materials that have typically been found in oxygen-enriched systems and components as a result of the manufacturing, assembly, fabrication, and construction processes include:
silicone oils and greases,
fluorinated aerospace fluids and greases,
petroleum based oils and greases,
ester based oils and greases,
phosphate esters,
waxes,
chlorotrifluoroethylene based oils and greases,
inks,
cutting oils, and
dye penetrants.

7.2 Solvent-reagent-grade used to prepare standard solutions of contaminants which may include the following: 2-propanol, 2-butanone, hexane, Type II reagent water, or better, in accordance with Specification D1193, and perfluorinated carbon fluids. (**Warning—**Solvents such as 2-propanol hexane and 2-butanone are highly flammable. The reader should refer to appropriate safe handling procedures.)

7.3 Desiccant—for example, silica gel.

7.4 Particulate contaminant—fine (0- to 80-µm), or coarse (0- to 200-µm) dusts[4] available commercially.

8. Procedure

8.1 *Coupon Preparation:*

8.1.1 The test coupons shall be numbered and precleaned prior to use; record the number. Determine the surface roughness, R_a, of representative coupons of each alloy being evaluated per ANSI B 46.1 and record. Determine the surface area to be contaminated (S) of each of the test coupons and record. Attach a handling wire through the hole at the top of each of the test coupons. The precleaning procedure shall be performed in an ultrasonic cleaner with the coupons immersed in type II reagent water for 10 min. Process a control coupon with the coupons to be contaminated for each cleaning agent to be evaluated. The coupons shall be dried and stored in a desiccator until needed.

8.1.2 Verify that the test coupons have an NVR of 10 mg/sq m (1 mg/sq ft) or less using Test Methods E1235, F324, or F331 and have no particles larger than 300 µm when evaluated using Test Methods F312. Determine the blank NVR values and particle counts on the solvents used to prepare the contaminating solutions as recommended in Practice F303. These values must be taken into account when preparing the standard contaminating solutions, especially when very dilute solutions are desired. Rinse each coupon being prepared for contamination with the solvent to be used in the contaminant solution. Allow the coupons to dry overnight or in an oven at 40 to 50°C for 1 h, cool to room temperature in a desiccator, and weigh. Reweigh the test coupon at intervals, 1 h typically, until a constant weight, ±0.1 mg, is achieved. Weigh, record the weight (W_1) in grams to the nearest 0.1 mg, and store in a desiccator until needed.

NOTE 2—It is recommended that coupons be prepared in lots of ten or more to ensure the necessary sensitivity for the verification.

8.1.3 After cleaning, the test coupons should be handled only with laboratory tongs or tweezers by the handling wire or a hook on the coupon itself.

8.2 *Method A, Nonvolatile Residue (NVR) Sample, Single Side:*

8.2.1 Prepare a standard solution containing the desired contaminant, in a suitable solvent for that contaminant. As an alternative, a mixture of as many contaminants as desired may be prepared in a suitable solvent. The concentration of the solution, typically between 1 and 100 mg/mL, should be adjusted to reflect the worst expected contamination level in the system to be cleaned. In some cases, i.e. oils, dye penetrants, or inks, it may not be necessary to use a solvent in the preparation of the coupons to achieve the desired level of contamination.

8.2.2 Place the test coupon(s), number side down, on a horizontal surface and carefully apply the solution containing the contaminant to the upper surface of the coupon using a pipette, syringe, brush, or spray applicator. Record the number of the test coupon and the surface area (S) in square centimeters of each coupon that was coated.

8.2.3 Allow the contaminant to dry overnight or in an oven at 40 to 50°C for 1 h, cool to room temperature in a desiccator, remove from the desiccator, and weigh. Reweigh the test coupon at intervals while storing the coupons in a desiccator between weighings, 4 h typically, until a constant weight is achieved. Record the final weight (W_2) in grams to the nearest 0.1 mg. It may be necessary to make additional applications to achieve the desired contamination level. (**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.)

8.2.4 Test coupons shall be used immediately after final weighing.

8.3 *Method B, NVR Sample, Double Side:*

8.3.1 Prepare a standard solution containing the desired contaminant, in a suitable solvent for that contaminant. As an alternative, a mixture of as many contaminants as desired may be prepared in a suitable solvent . The concentration of the solution, typically between 1 and 100 mg/mL, should be adjusted to reflect the worst expected contamination level in the system to be cleaned. In some cases, e.g. oils, dye penetrants, or inks, it may not be necessary to use a solvent in the preparation of the coupons to achieve the desired level of contamination.

8.3.2 Carefully dip the test coupon into the contaminant solution until it is completely immersed. Slowly withdraw the test coupon from the contaminant solution. Other methods of application include brushing or spraying. Record the number of the test coupon and the surface area (S) of each coupon that was coated. Hang the test coupon by the handling wire and allow the contaminant to dry overnight or in an oven at 40 to 50°C for 1 h. Cool to room temperature in a desiccator, and weigh. Reweigh the test coupon at intervals, 4 h typically, until a constant weight is achieved. Record the final weight (W_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.)

8.3.3 Test coupons shall be used immediately after the final weighing.

8.4 *Method C, NVR and Particulate Sample:*

8.4.1 Prepare a standard solution containing the desired NVR contaminant, in a suitable solvent for that contaminant. As an alternative, a mixture of as many NVR contaminants as desired may be prepared in a suitable solvent. The NVR contaminant concentration of the solution, typically between 1 and 100 mg/mL, should be adjusted to reflect the worst expected NVR contamination level in the system to be cleaned. Add 10, 20, or 30 mg of particulate contaminant to each 100

[4] Dusts of varying particle sizes are available from the AC Spark Plug Division of General Motors, 1300-T N. Dort Hwy., Flint, MI 48556.

mL of solution to provide a light, medium, or heavy particulate level, respectively. In some cases, that is, oils, dye penetrants, or inks, it may not be necessary to use a solvent in the preparation of the coupons to achieve the desired level of contamination.

8.4.2 Place the test coupon(s), number side down, on a horizontal surface and carefully apply the solution containing the contaminant, which is agitated or stirred to keep the particulate suspended, to the top upper surface of the coupon using a pipette, syringe, brush, or spray applicator. Hang the test coupon by the handling wire and allow the contaminant to dry overnight or in an oven at 40 to 50°C for 1 h. Cool to room temperature in a desiccator, and weigh. Reweigh the test coupon at intervals, 4 h typically, until a constant weight is achieved. Record the final weight (W_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.)

8.4.3 Test coupons shall be used immediately after final weighing.

9. Calculation

9.1 Calculate the amount of contaminant on the test coupon in mg/cm^2 using:

$$C = \frac{1000(W_2 - W_1)}{S}$$

where:

C = amount of contaminant, mg/cm^2;
W_1 = weight of the clean coupon, g;
W_2 = weight of the contaminated coupon, g; and
S = contaminated surface area of the coupon, cm^2.

10. Report

10.1 The report shall include the following:

10.1.1 The number of the test coupon,

10.1.2 Identification of alloy,

10.1.2.1 Name of Unified Numbering System (UNS) alloy designation,

10.1.2.2 Heat,

10.1.2.3 Hardness,

10.1.2.4 Surface roughness (R_a),

10.1.3 Identification of the solvent used,

10.1.4 Identification of the contaminant,

10.1.5 Method of application of the contaminant,

10.1.6 Method of drying the test coupon,

10.1.7 Weight of the uncontaminated coupon (W_1),

10.1.8 Weight of the contaminated coupon (W_2), and

10.1.9 The amount of contaminant (C) on the test coupon in mg/cm^2.

11. Keywords

11.1 cleaning agents; contaminant; contamination; non-volatile residue; oxygen systems; standard test coupon