Standard Practices for
Ground Laboratory Atomic Oxygen Interaction Evaluation of Materials for Space Applications[1]

This standard is issued under the fixed designation E2089; 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.

1. Scope

1.1 The intent of these practices is to define atomic oxygen exposure procedures that are intended to minimize variability in results within any specific atomic oxygen exposure facility as well as contribute to the understanding of the differences in the response of materials when tested in different facilities.

1.2 These practices are not intended to specify any particular type of atomic oxygen exposure facility but simply specify procedures that can be applied to a wide variety of facilities.

1.3 The values stated in SI units are to be regarded as the 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.*

2. Terminology

2.1 *Definitions:*

2.1.1 *atomic oxygen erosion yield*—the volume of a material that is eroded by atomic oxygen per incident oxygen atom reported in cm^3/atom.

2.1.2 *atomic oxygen fluence*—the arrival of atomic oxygen to a surface reported in atoms/cm^2

2.1.3 *atomic oxygen flux*—the arrival rate of atomic oxygen to a surface reported in $atoms \cdot cm^{-2} \cdot s^{-1}$.

2.1.4 *effective atomic oxygen fluence*—the total arrival of atomic oxygen to a surface reported in atoms/cm^2, which would cause the observed amount of erosion if the sample was exposed in low Earth orbit.

2.1.5 *effective atomic oxygen flux*—the arrival rate of atomic oxygen to a surface reported in $atoms \cdot cm^{-2} \cdot s^{-1}$, which would cause the observed amount of erosion if the sample was exposed in low Earth orbit.

2.1.6 *witness materials or samples*—materials or samples used to measure the effective atomic oxygen flux or fluence.

2.2 *Symbols:*

A_k = exposed area of the witness sample, cm^2
A_s = exposed area of the test sample, cm^2
E_k = in-space erosion yield of the witness material, cm^3/atom
E_s = erosion yield of the test material, cm^3/atom
f_k = effective flux, atoms/cm^2/s
F_k = effective fluence, total atoms/cm^2
ΔM_k = mass loss of the witness coupon, g

3. Significance and Use

3.1 These practices enable the following information to be available:

3.1.1 Material atomic oxygen erosion characteristics.

3.1.2 An atomic oxygen erosion comparison of four well-characterized polymers.

3.2 The resulting data are useful to:

3.2.1 Compare the atomic oxygen durability of spacecraft materials exposed to the low Earth orbital environment.

3.2.2 Compare the atomic oxygen erosion behavior between various ground laboratory facilities.

3.2.3 Compare the atomic oxygen erosion behavior between ground laboratory facilities and in-space exposure.

3.2.4 Screen materials being considered for low Earth orbital spacecraft application. However, caution should be exercised in attempting to predict in-space behavior based on ground laboratory testing because of differences in exposure environment and synergistic effects.

4. Test Specimen

4.1 In addition to the material to be evaluated for atomic oxygen interaction, the following four standard witness materials should be exposed in the same facility using the same operating conditions and duration exposure within a factor of 3, as the test material: Kapton(R)[2] H or HN polyimide, tetrafluoroethylene (TFE)-fluorocarbon fluorinated ethylene

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[1] These practices are under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and are the direct responsibility of Subcommittee E21.04 on Space Simulation Test Methods.

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[2] Kapton(R) and DuPont (TM) are trademarks or registered trademarks of E. I. DuPont de Nemours and Company.

propylene (FEP), low-density polyethylene (PE), and pyrolytic graphite (PG). The atomic oxygen effective flux (in atoms·cm^{-2}·s^{-1}) and effective fluence (in atoms/cm^2) for Kapton H or HN polyimide should be reported along with the mass or thickness loss relative to Kapton H or HN polyimide for the test material, TFE-fluorocarbon FEP, PE, and PG. For atomic oxygen interaction testing at effective fluences beyond 2×10^{21} atoms/cm^2, Kapton H polyimide has been recommended in the past, however E. I. du Pont de Nemours and Company (DuPont (TM[2])) has discontinued its manufacture. Kapton H polyimide is the preferred replacement, but Kapton HN polyimide contains atomic oxygen-resistant inorganic particles which begin to protect the underlying polyimide, thus resulting in an atomic oxygen erosion yield in low Earth orbit (2.81×10^{-24} cm^3/atom) that is slightly less than that of Kapton H (3.00×10^{-24} cm^3/atom)) (**1**)[3].

4.2 It is not necessary to test the four standard witness samples for each material exposure if previous data exists at the same exposure conditions and if the fluence for the test sample is within a factor of 3 of the standard witness exposure. When possible, the recommended standard witness polymer materials should be 0.05 mm thick and of a diameter greater than 5 mm. It is recommended that the pyrolytic graphite witness sample be 2 mm thick and of a diameter greater than 5 mm. High-fluence tests, which may erode through the full thickness of the standard polymer witness, can use the recommended thickness sample materials by stacking several layers of the polymer on top of each other.

5. Procedure

5.1 *Sample Preparation:*

5.1.1 *Cleaning:*

5.1.1.1 The samples to be evaluated for atomic oxygen interactions should be chemically representative of materials that would be used in space. Thus, the surface chemistry of the samples should not be altered by exposure to chemicals or cleaning solutions which would not be representatively used on the functional materials to be used in space.

5.1.1.2 Wiping samples or washing them may significantly alter surface chemistry and atomic oxygen protection characteristics of materials, and is therefore not recommended. However, if the typical use in space will require preflight solvent cleaning, then perform such cleaning to simulate actual surface conditions expected.

5.2 *Handling*—The atomic oxygen durability of materials with protective coatings may be significantly altered as a result of mechanical damage associated with handling. In addition, unprotected materials can become contaminated by handling, resulting in anomalous consequences of atomic oxygen exposure. It is recommended that samples be handled such as to minimize abrasion, contamination and flexure. The use of soft fluoropolymer tweezers is recommended for handling polymeric films with protective coatings. For samples too heavy to be safely held with tweezers, use clean vinyl, latex, or other gloves which will not allow finger oils to soak through and which are lint-free to carefully handle the samples.

5.3 *Exposure Area Control:*

5.3.1 *Masking*—Frequently it is desirable to limit the exposure of atomic oxygen to one side of a material or a limited area on one side of the material. This can be done by wrapping metal foil (such as aluminum foil) around the sample, covering an area with a sacrificial polymer (such as a polyimide), salt-spraying to produce sites of atomic oxygen protection, or by using glass to cover areas not to be exposed. It is recommended that the protective covering be in intimate contact with the material to prevent partial exposure of the masked areas. When using metal foil within the RF or microwave excitation region of an atomic oxygen source, it is likely that electromagnetic interactions could take place between the metal and the plasma that could cause anomalous atomic oxygen fluxes or shielding from charged species, or both. It is important to expose the four standard witness coupons in this configuration before any other testing to determine the effects of the masking on the atomic oxygen flux.

5.3.2 *Cladding*—Samples which are coated with protective coatings on one side can be clad together by means of adhesives to allow the protective coating to be exposed on both sides of the sample. The use of thin polyester adhesives (or other non-silicone adhesive) is recommended to perform such cladding. The use of silicone adhesives should be avoided because of potential silicone contamination of the sample. Although cladding allows samples to be tested with the protective coatings on both faces, edge exposure of the samples and their adhesive does occur and should be accounted for in calculating erosion characteristics of the desired surfaces.

5.4 *Dehydration and Outgassing (for Samples Undergoing Weight Measurement)*—Because most nonmetals and nonceramic materials contain significant fractional quantities of water or other volatiles, or both, it is recommended that these types of materials be vacuum-dehydrated before weighing to eliminate errors in weight because of moisture loss. Dehydrate samples of a thickness less than or equal to 0.127 mm (5 mils) in a vacuum of a pressure less than 200 millitorr for a duration of 48 h before sample weighing to ensure that the samples retain negligible absorbed water. Dehydrate and weigh thicker samples periodically until weight loss indicates that no further water is being lost. Dehydrate multiple samples in the same vacuum chamber provided they do not cross-contaminate each other, and that they are not of sufficient quantity so as to inhibit uniform dehydration of all the samples.

5.5 *Weighing*—Because hydration occurs quickly after removal of samples from vacuum, weighing the samples should occur within five minutes of removal from vacuum dehydration chambers. Reduction of uncertainty associated with moisture uptake can be minimized by weighing the samples at measured intervals following removal from vacuum and back extrapolating to the mass at time of removal from vacuum. Weigh samples using a balance whose sensitivity is capable of measuring the mass loss of the atomic oxygen fluence witness samples. For 2.54-cm-diameter by 0.127-mm-thick Kapton H or HN polyimide fluence witness samples, a balance sensitivity

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[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

of 1 mg is acceptable for effective fluences of at least 10^{19} atoms/cm^2. Weigh the samples at room temperature (20 to 25°C). If the temperature is outside this range, measure and record at the time of weighing.

5.6 *Effective Fluence Prediction:*

5.6.1 *Fluence Witness Samples:*

5.6.1.1 If the test sample is a material that does not have any protective coating, then use polyimide Kapton H or HN samples to determine the effective atomic oxygen fluence. If the test sample has an atomic oxygen protective coating, then test an unprotected sample of the substrate material as well. The unprotected sample can also be used to determine the effective atomic oxygen fluence provided that in-space erosion yield data is available. If such in-space data is not available, then use a sample of polyimide Kapton H or HN should be used for determination of effective atomic oxygen fluence assuming an in-space erosion yield of 3.0×10^{-24} cm^3/atom or 2.81×10^{-24} cm^3/atom respectively.

5.6.1.2 It is recommended that where physically possible, the atomic oxygen fluence witness material be exposed to atomic oxygen simultaneously with the test samples to enable calculation of the effective atomic oxygen fluence. If chamber geometry prevents this, expose a fluence witness coupon just prior to or immediately after the test sample. If high-fluence exposure is necessary, quite often polymeric sheets are too thin to survive long exposures. Therefore, thick coupons of polyimide or graphite are suggested to be used for high-fluence weight or thickness loss measurements. The atomic oxygen erosion yield of pyrolytic graphite relative to polyimide Kapton H or HN is different in some ground laboratory facilities than in space. Therefore, it is necessary to convert the mass loss or thickness loss of the pyrolytic graphite to the equivalent loss of polyimide Kapton H. This can be accomplished by simultaneous or sequential exposure of pyrolytic graphite and the Kapton, and will enable the effective fluence to be calculated in terms of Kapton effective fluence, which is the accepted standard.

5.6.1.3 It is recommended that, periodically, samples of Kapton H or HN, TFE-fluorocarbon FEP, polyethylene, and pyrolytic graphite be exposed to atomic oxygen in the test chamber to verify operational consistency and to allow comparisons to be made between this test facility, space, and other ground-based systems. Report this data along with any test data so that test results can be compared more easily.

5.6.2 *Test, Standard Witness, and Fluence Witness Sample Position and Orientation*—Facilities typically experience some spatial flux variation depending on how the atomic oxygen is formed. Minimization of errors in effective atomic oxygen fluence will be achieved if witness samples are placed as close as possible to the same location as the test sample, and that the exposed surfaces of the test sample and witness sample are identical in size and orientation. The use of witness samples of the same size, position, and orientation as the test samples is recommended.

5.6.3 *Inspection and Validation of Standard Witness and Fluence Witness Sample Erosion*—Visibly inspect and compare witness samples with previously exposed witness samples that have demonstrated acceptable performance to validate that contamination of the surface of the sample has not occurred. Contamination can look like oil spots on the surface, a protective thin film, or other optical deviation from a normally diffuse reflecting exposed surface. Compare the effective flux for the witness sample with that from tests previously known to be acceptable which were performed in the same facility to ensure that neither contamination nor anomalous operation has occurred.

5.6.4 *Erosion Measurement*—Measurement of atomic oxygen erosion of test samples and witness samples generally can be accomplished by weight loss or thickness loss measurements.

5.6.4.1 *Weight Loss*—Weigh witness samples within five minutes of removal from the vacuum chamber. Remove only one sample at a time for weighing. The rest should remain under vacuum to minimize rehydration mass increases. When witness samples are of the same chemistry as the substrate of protected samples, it is important to weigh both samples as close as possible to the same time interval after removal from vacuum.

5.6.4.2 *Thickness Loss*—Witness coupon material loss can also be measured using various surface profiling techniques if the exposure area is too small for accurate weight measurements to be taken. Profiling can be accomplished by stylus profiling, scanning atomic force microscopy, or other recession measurement techniques. Take care when exposing samples to atomic oxygen which will be subsequently used for profiling measurements that a portion of the original surface is kept intact and that a clear step exists between the original surface and the atomic oxygen exposed portion. This requires that a thin (<0.2 mm thick) removable mask be used that is in intimate contact with the surface during the atomic oxygen exposure to prevent scattering of atomic oxygen from the edges of the mask, which would provide an artificial increase in fluence on the sample.

6. Calculation

6.1 *Atomic Oxygen Kapton Effective Fluence Determination:*

6.1.1 Measurement of absolute fluence in ground laboratory facilities is typically difficult to perform. In addition, such measurements do not reliably predict in-space durability because of differences in erosion yields in the ground laboratory facility compared to in-space. There is likely to be a substantial dependence of erosion yield upon energy, fluence, and degree to which the arriving oxygen atoms or ions have isotropic or paraxial arrival, which is also material-dependent.

6.1.2 To assist in the prediction of in-space performance based on ground laboratory atomic oxygen testing, it is desirable, to measure an effective fluence for the material being tested using a comparison of mass or thickness loss.

6.1.3 If in-space atomic oxygen erosion data is not available for the material being tested, then an effective fluence can be reported based on witness sample erosion of a material of known in-space erosion yield (such as polyimide Kapton H or HN). The witness sample material used must be reported along with the result. Calculate effective flux and effective fluence using the following equations:

$$\text{Effective flux} = f_k = \frac{\Delta M_k}{A_k \rho_k E_k t} \tag{1}$$

$$\text{Effective fluence} = F_k = f_k t = \frac{\Delta M_k}{A_k \rho_k E_k} \tag{2}$$

where:

ΔM_k = mass change in witness, g;
A_k = exposed area of witness, cm^2;
ρ_k = density of witness, g/cm^3;
E_k = erosion yield of the fluence witness material in space, cm^3/atom; and
t = duration of exposure, s.

6.2 *Erosion Yield Calculation*

6.2.1 Based on mass loss, the erosion yield of a material is typically given in units of cubic centimetres per incident oxygen atom. Calculate the erosion yield as follows:

$$E_S = \frac{\Delta M_S}{A_S \rho_S F_k} \tag{3}$$

where:

ΔM_S = mass loss of the sample, g;
A_S = surface area of the sample exposed to atomic oxygen, cm^2;
ρ_S = density of the sample, g/cm^3; and
F_k = effective fluence, atoms/cm^2 or actual fluence if measured in space.

Because the fluence is typically based on the effective fluence of a witness coupon, calculate the erosion yield as follows:

$$E_S = \frac{\Delta M_S A_k \rho_k E_k}{\Delta M_k A_S \rho_S} \tag{4}$$

where:

A_k = area of the witness coupon, cm^3;
ρ_k = density of the witness coupon, g/cm^3;
E_k = in-space erosion yield of the witness coupon, cm^3/atom; and
ΔM_k = mass loss of the witness coupon, g.

6.2.2 Based on thickness loss, calculate the erosion yield as follows:

$$E_S = \frac{\Delta x_S}{F_k} \tag{5}$$

where:

Δx_S = thickness loss of the sample, cm and
F_k = effective fluence, atom/cm^2 or actual fluence if measured in space.

If the effective fluence is also measured by thickness loss, then calculate as follows:

$$E_S = \frac{\Delta x_S E_k}{\Delta x_k} \tag{6}$$

where:

Δx_k = thickness loss of the fluence witness coupon, cm and
E_k = in-space erosion yield of the fluence witness coupon, cm^3/atom.

7. Report

7.1 *Comparative Erosion Data Table*—Report results of atomic oxygen interactions with materials including comparative erosion data on the four standard witness materials listed in Table 1. Note mass loss data taken at temperatures other than room temperature (20 to 25°C) with the sample temperature reported as an additional column in Table 1.

7.2 *Test Sample Data*—Report materials used, effective fluence, thickness loss or mass loss, assumed in-space erosion yield for effective fluence measurement, and test sample exposure and data for an unprotected sample of the same material (where applicable) as in Table 2.

7.3 *Description of the Atomic Oxygen Exposure Facility and the Sample Exposure Environment*—(Brief description of atomic oxygen exposure facility including physical processes and conditions used to produce atomic oxygen and, where possible, quantification of the temperature of the samples during exposure; the types, energy, and flux of species exposed to the samples; and information on the atomic oxygen facility, sample preparation, sample exposure, and sample mounting, grounding or applied potential, sample surface orientation with respect to atomic oxygen arrival direction, and measurement equipment that could have impact on results and their accuracy or the interpretation of the results.)

8. Precision and Bias

8.1 It is not possible to specify the precision of the procedure in Practice E2089 for measuring the effects of atomic oxygen interaction with materials because the atomic oxygen exposure systems used are not prescribed.

TABLE 1 Comparative Erosion Data

Standard Witness Materials	Kapton H or HN Effective Flux, atoms/cm^2	Kapton H or HN Effective Fluence, atoms/cm^2	Thickness or Mass Loss Relative to Kapton HN	Measured Erosion Yield	Assumed In-Space Erosion Yield, cm^3/atom **(2, 1)**
Kapton H					3.00×10^{-24}
Kapton HN			1		2.81×10^{-24}
P E					3.97×10^{-24}
F E P					2.00×10^{-25}
P G					4.05×10^{-25}

TABLE 2 Test Sample Data

Materials Used for:	Effective Fluence, atoms/cm^2	Thickness Loss, CM or Mass Loss/ Area,^{A} g/cm^2	Assumed In-space Erosion Yield, cm^3/atom
Effective fluence measurement			
Test sample			
Unprotected test sample			

^{A}When applicable.

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