Designation: D1879 − 06 (Reapproved 2023)

Standard Practice for Exposure of Adhesive Specimens to Ionizing Radiation[1]

This standard is issued under the fixed designation D1879; 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 purpose of this practice is to define conditions for the exposure of polymeric adhesives in bonded specimens to ionizing radiation prior to determination of radiation-induced changes in physical or chemical properties. This recommended practice specifically covers the following kinds of radiation: gamma or X-ray radiation, electron or beta radiation, neutrons, and mixtures of these such as reactor radiation.

1.2 This practice specifies only the conditions of irradiation but does not cover the preparation of test specimens, testing conditions, or the evaluation of test. These are covered in the various ASTM methods or specifications for specific materials.

1.3 This practice covers procedures for the following five types of exposure:

Procedure A—Exposure at ambient conditions.
Procedure B—Exposure at controlled temperature.
Procedure C—Exposure in a medium other than air.
Procedure D—Exposure under load.
Procedure E—Exposure combining two or more of the variables listed in Procedures A to D.

NOTE 1—The problems of measuring the properties of materials during irradiation involve shielding and remote control facilities and are, therefore, not considered in this practice.

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

1.5.1 *Electrical Hazard:* **Warning**—The users of this practice must be aware that there are inherent dangers associated with the use of electrical instrumentation and that this practice cannot and will not substitute for a practical knowledge of the instrument used for a particular procedure.

1.5.2 *Radio Frequency:* **Warning**—Persons with pacemakers may be affected by the radio frequency.

1.6 *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]

D618 Practice for Conditioning Plastics for Testing
D907 Terminology of Adhesives
D1672 Practice for Exposure of Polymeric Materials to High-Energy Radiation (Withdrawn 1984)[3]
D2953 Classification System for Polymeric Materials for Service in Ionizing Radiation (Withdrawn 1984)[3]
E170 Terminology Relating to Radiation Measurements and Dosimetry
E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques
E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation
E720 Guide for Selection and Use of Neutron Sensors for Determining Neutron Spectra Employed in Radiation-Hardness Testing of Electronics
E2005 Guide for Benchmark Testing of Reactor Dosimetry in Standard and Reference Neutron Fields

2.2 *ISO/ASTM Standards:* [2]

ISO/ASTM 51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing
ISO/ASTM 51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 300 keV and 25 MeV
ISO/ASTM 51702 Practice for Dosimetry in Gamma Irradiation Facilities for Radiation Processing
ISO/ASTM 51818 Practice for Dosimetry in an Electron

[1] This practice is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

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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.

Beam Facility for Radiation Processing at Energies Between 80 and 300 keV

2.3 *ANSI Document:*

N1.1 Glossary of Terms in Nuclear Science and Technology[4]

2.3 *IEEE Documents:*[5]

278 Classifying Electrical Insulating Materials Exposed to Neutron and Gamma Radiation

323 Qualifying Class 1E Equipment for Nuclear Power Generating Stations

3. Terminology

3.1 Many terms in this practice are defined in Terminology D907 and in Terminology E170.

3.2 *gray, n*—the unit of absorbed dose when the energy per unit mass imparted to matter by radiation is one joule per kilogram.

3.3 *rad, n*—the unit of absorbed dose when the energy per unit mass imparted to matter by radiation is 100 ergs per gram.

NOTE 2—To convert from rad to gray (Gy), multiply by 1.00×10^{-2}. 1 rad = 0.01 gray and 1 megarad (MR) = 10 kilograys (kGy).

4. Significance and Use

4.1 The procedures outlined in this practice are designed to standardize the exposure of adhesive-bonded specimens for the purpose of studying the effects of ionizing radiation, but have been made flexible enough so that a large variety of conditions may be met within the scope of this one irradiation method. Because of this flexibility in the procedures, it is important that the experimenter have some idea of the kind of changes that will occur, and of the conditions that will affect these changes.

5. Effects of Irradiation

5.1 Exposure to radiation can result in changes in monomers, oligomers and high polymers, which owe some of their properties to chemical links formed within molecular structures. These structures may be cross-linked by radiation into insoluble, three-dimensional networks, may be cleaved into smaller molecules, or unaffected by radiation exposure. Crosslinking and cleavage or scission may occur at the same time.

5.2 One effect of the reaction of ionizing radiation with polymers is the formation of free radicals, atoms containing unpaired electrons. In some instances, the rate at which free radicals are formed may be much greater than their rate of extinction. In a few instances, this can lead to trapped reactive species within the irradiated material and to the possibility of continuing reactions for days or weeks after the specimen has been removed from the radiation field. Because of these limited post-irradiation reactions it has been found necessary to standardize the times and conditions of storage between irradiation and testing of specimens.

5.3 The resultant changes in the morphology of polymeric materials caused by exposure to radiation can be dependent on the respective rates of recombination, crosslinking, or cleavage of the material segments. These rates are affected by the mobility of the excited atoms (free radicals or ionized) which in turn is influenced by temperature and by the concentration of the excited or ionized atoms.

5.4 The concentration of reactive species will vary with the rate of absorption of radiation. Both radiation exposure or dose and dose-rate should be specified in reporting the results of tests. The effect of dose, dose-rate and specimen thickness can sometimes be observed when irradiations are carried out in air, that is in the presence of oxygen, wherein oxygen reacts with radicals produced in the irradiated material. This oxygen reaction is diffusion controlled. The reactivity of some irradiated specimens toward oxygen makes it necessary to specify whether irradiations are carried out in air or in an inert atmosphere. The accessibility to an air supply undepleted in oxygen should be assured if possible.

5.5 The localized concentration of reactive species during irradiation will vary, depending on the type of radiation employed. The proton and carbon recoils from neutron bombardment produce densely ionized tracks in the specimen compared to the diffuse ionization in the wake of protons or electrons. The effect of different types of radiation may therefore be different. It is required that the type of radiation to which the specimen has been exposed be reported as well as the irradiation dose in terms of energy absorbed units, that is, grays or kiloGrays (kGy).

5.6 Various chemical structures respond differently on exposure to radiation. The exposure levels for testing should be based upon the end-use of the bonded assembly and upon consideration of the chemical structure of the adhesive material. Aromatic materials, such as polystyrene (PS), polycarbonates (PC) and polyethylene terephthalate (PET), tend to be unaffected, in terms of physical properties, by modest radiation exposure. Materials with an abstractable hydrogen, such as polyethylene (PE), will crosslink, with the radiation response being very dependent on the specific morphology of a given grade and its additives. Materials with tetra-substituted carbon atoms, such as polymethylmethacrylate (PMMA), polytetrafluorothylene (PTFE) and polyvinylidene chloride (PVdC), will exhibit scissioning and generally a weakening of physical properties. The exposure levels or cumulative dose should be those which will produce measurable changes in a stipulated property rather than a specified fixed irradiation dose. Such changes in property may progress at different rates, with some materials changing rapidly once a change has been initiated, while others may change quite slowly. It is necessary therefore to irradiate to several fixed levels of property change in order to establish the rate of change (see 13.2).

5.7 Some materials that have been exposed to reactor radiation in terms of neutron flux may become radioactive. These can be metallic and other inorganic adherends and fillers. For exact work, where the reactor spectrum is being studied, exposure in a reactor would give the only accurate results.

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

[5] Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., P.O. Box 1331, Piscataway, NJ 08854-1331, http://www.ieee.org.

6. Test Specimens

6.1 Wherever possible, use the type of specimens in accordance with the ASTM test methods for the specific properties to be measured.

6.2 Where it is not possible to utilize standard test specimens, make irradiated and non-irradiated specimens of the same size and shape.

6.3 Since organic adherends may be sensitive to radiation, they should be tested independently of the adhesive assembly under the same conditions, using irradiated and non-irradiated adherend specimens.

7. Conditioning

7.1 Condition specimens to be exposed in air in accordance with Procedure A of Practice D618.

7.2 Condition specimens to be exposed in a gas other than air at the temperature of exposure in an appropriate container at a pressure of 10 Pa (10^{-3} mm Hg) or less for at least 8 h followed by three flushes with the gas to be present during exposure. After flushing, fill the container with the exposure gas and seal it.

7.3 Condition specimens to be exposed in a vacuum at the temperature of exposure in an appropriate container at a pressure of 10 Pa (10^{-3} mm Hg) or less for at least 48 h. Then seal the container from the vacuum system. Where increase in pressure due to outgassing may be undesirable or where the outgassing products themselves may be undesirable, the vacuum in the container may be maintained by pumping continuously during the irradiation.

7.4 Condition specimens to be exposed in a liquid medium in accordance with 7.1 before placing in the liquid medium. Immerse the specimens completely in the liquid during the entire period of irradiation.

7.5 Depending upon the type and energy of radiation, inorganic adherends may have a shielding effect on the adhesive bond. Because of this position the specimens so that the shielding effect is uniform over all the adhesive layer.

8. Procedure A—Exposure at Ambient Conditions

8.1 After conditioning in accordance with 7.1, expose the specimens on suitable racks or in containers such that free access to air is assured on all sides.

8.2 When the radiation source requires that the specimens be enclosed in a container, package the specimens in the Standard Laboratory Atmosphere (7.1).

NOTE 3—It is likely that the composition of the atmosphere in the container will be changed by radiation-induced reactions. Therefore, it should be clearly stated in the report that the irradiation was made in a closed container.

8.3 If irradiation is performed using a beam-emitting accelerator, convey the specimens in some manner such that they traverse the radiation beam; hold the ratio of exposure time to non-exposure time constant throughout this procedure. In the absence of a conveyor type system, place the specimens in a fixed position in the beam where it is known that this irradiation dose will be uniform throughout the area and thickness of the specimen. Expose the specimens to only one total dose. For each new total dose, expose additional properly conditioned specimens. Exposure in nuclear reactors or other sources having uniform radiation fields will not require traversing the radiation field.

8.4 After the required period of time, remove the specimens from the field and condition prior to test in the Standard Laboratory Atmosphere (7.1), for no less than 16 and no more than 32 h, unless it is necessary to store the specimens for longer periods of time because of radioactivity or other reasons. Report the time and condition of such storage.

8.5 Condition non-irradiated control specimens in accordance with 7.1 prior to test in the Standard Laboratory Atmosphere.

9. Procedure B—Exposure at Controlled Temperatures

9.1 Follow the procedure outlined in 8.1 and 8.2.

9.2 Irradiate the specimens as described in 8.3 at the desired temperature. Place a dummy specimen containing a grounded thermocouple in the radiation field at the same conditions as the test specimens to determine the temperature. If the temperature varies by more than ±5 °C, it should be reported.

9.3 Condition the specimens as outlined in 8.4.

9.4 After conditioning in accordance with 7.1, expose non-irradiated control specimens to the same temperature employed in 9.2 for the same period of time as the irradiated specimens.

9.5 After treatment, condition the control specimens along with the irradiated specimens in accordance with 7.1 prior to test.

10. Procedure C—Exposure in Medium Other than Air

10.1 After conditioning in accordance with 7.2, 7.3, or 7.4, irradiate the specimens as described in 8.3.

10.2 After removal from the medium, condition the specimens according to the procedure outlined in 8.4.

10.3 The non-irradiated control specimens that have been conditioned in accordance with 7.2, 7.3, or 7.4 shall remain in the selected medium for the same period of time as the irradiated specimens.

10.4 After treatment, condition the control specimens along with the irradiated specimens in accordance with 8.4 prior to test.

11. Procedure D—Exposure Under Load

11.1 After conditioning in accordance with 7.1, arrange the specimens on a suitable fixture such that they may be subjected to a load during irradiation and have maximum access to air.

11.2 Follow the packaging and irradiating procedures outlined in 8.1 or 8.2 and 8.3.

11.3 After removal from the radiation field release the load and condition the specimens prior to test in the Standard Laboratory Atmosphere in accordance with 8.4.

11.4 After conditioning in accordance with 7.1, load non-irradiated control specimens in the same manner and for the