Designation: D 1879 – 99

Standard Practice for Exposure of Adhesive Specimens to High-Energy Radiation[1]

This standard is issued under the fixed designation D 1879; 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 high-energy 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.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 907 Terminology of Adhesives[3]

D 1671 Test Method for Absorbed Gamma Radiation Dose in the Fricke Dosimeter[4]

D 1672 Practice for Exposure of Polymeric Materials to High-Energy Radiation[4]

D 2953 Classification System for Polymeric Materials for Service in Ionizing Radiation[5]

2.2 *ANSI Document:*

N1.1 Glossary of Terms in Nuclear Science and Technology[6]

2.3 *IEEE Documents:*[7]

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 D 907.

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] This practice is under the jurisdiction of ASTM Committee D-14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

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[2] *Annual Book of ASTM Standards,* Vol 08.01.

[3] *Annual Book of ASTM Standards,* Vol 15.06.

[4] Discontinued; see *1984 Annual Book of ASTM Standards*, Vol 12.02.

[5] Discontinued; see *1985 Annual Book of ASTM Standards,* Vol 12.02.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from Institute of Electrical and Electronics Engineers, 345 E. 47th St., New York, NY 10017.

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 high-energy 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 results in extensive changes in the nature of high polymers, which owe their unique properties to chemical linking into giant molecules of chain or net structure. These chain or net structures may be cross-linked by radiation into a rigid, three-dimensional network or in other cases, may be cleaved into smaller molecules to produce a weaker material. Both may occur at the same time. In all cases some low molecular weight fragments are produced and, if exposures are large enough, general decomposition results.

5.2 The first result of the reaction of high-energy radiation with polymers is the formation of free radicals or excited molecular fragments. The rate at which these molecular fragments are formed may be much greater than their annihilation rate, and this leads to the accumulation of 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 post-irradiation reactions it has been necessary to standardize the times and conditions of storage between irradiation and testing of specimens.

5.3 The resultant changes in the molecular structure of polymeric materials by exposure to radiation are dependent on the respective rates of recombination, crosslinking, or cleavage of the molecular fragments. These rates are affected by the mobility of the molecular fragments (which is strongly influenced by temperature) and by the concentration of the reactants.

5.4 The concentration of reactive species will vary with the rate of absorption of radiation. Either radiation intensity or dose rate is therefore specified in reporting the results of tests, even though a dose rate effect is not often observed. The effect of dose rate and specimen thickness is observed when irradiations are carried out in the presence of oxygen, where oxygen reacts with radicals produced in the irradiated material. This oxygen reaction will be diffusion controlled. The reactivity of irradiated specimens toward oxygen makes it necessary to specify whether irradiations are carried out in air. 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 energy absorbed units.

5.6 A wide variation in the stability of the various chemical structures on exposure to radiation makes it difficult to select specific exposure levels for testing. Polystyrene requires the absorption of about 50 times as much radiation energy for the formation of one crosslink as does polyethylene. At the other end of the scale, poly(methylmethacrylate) and polytetrafluoroethylene show changes in engineering properties at about $\frac{1}{20}$ the exposure required for changes in polyethylene. An aromatic ring attached to the main chain at frequent regular intervals has been found to confer marked stability toward radiation, while a quaternary carbon atom in the polymer chain leads to cleavage under radiation and a loss of strength at fairly low exposures. The exposure levels should therefore be those which will produce significant changes in a stipulated property rather than a specified fixed irradiation dose. Furthermore, the change 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 Materials that have been exposed to reactor radiation will become radioactive. For pure hydrocarbons, the amount of induced radioactivity is not large, but metallic and other inorganic adherends and fillers and small amounts of impurities may become highly radioactive and thus create a handling problem. The other common radiation sources to which polymeric materials will be exposed will not normally produce significant amounts of induced radioactivity. The obvious solution would be to expose adhesive-bonded metallic specimens in non-neutron environments only. Unfortunately it is very difficult to calculate for a given reactor spectrum the equivalent dosage in a gamma source. For exact work, where the reactor spectrum is being studied, exposure in a reactor would give the only accurate results.

5.8 Metallic adherends such as cadmium will produce large sources of secondary radiation, which will significantly add to the absorbed dose of the adhesive.

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 nonirradiated specimens of the same size and shape.

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

7. Conditioning

7.1 Condition specimens to be exposed in air in accordance with Procedure A of Practice D 618.

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