



Standard Test Method for Measuring Reaction Rates by Analysis of Molybdenum-99 Radioactivity From Fission Dosimeters¹

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1. Scope

1.1 This test method covers the measurement of reaction rates by determining the amount of the fission product ⁹⁹Mo produced by the non-threshold reactions ²⁴¹Am(n,f), ²³⁵U(n,f), and ²³⁹Pu(n,f) and by the threshold reactions ²³⁸U(n,f), ²³⁷Np(n,f), and ²³²Th(n,f).

1.2 These reactions produce many fission products, among which is ⁹⁹Mo, having a half-life of 65.94 h. Because of unresolved or interfering gamma rays when gamma-ray spectrometry is accomplished, the competing activity from other fission products requires that a chemical separation be employed to isolate the ⁹⁹Mo. ⁹⁹Mo emits gamma rays of several energies, which are easily detected and identified by a gamma-ray spectrometer.

1.3 With suitable techniques, neutron fluence rates can be measured in the range 10⁷ to approximately 10¹⁴ n·cm⁻²·s⁻¹.

1.4 The measurement of time-integrated reaction rates with fission dosimeters by ⁹⁹Mo analysis is limited by the half-life of ⁹⁹Mo to irradiation times up to about 300 h.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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. Referenced Documents

2.1 ASTM Standards:

C 697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets²

D 1193 Specification for Reagent Water³

E 170 Terminology Relating to Radiation Measurements and Dosimetry⁴

¹ This test method is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.05 on Nuclear Radiation Metrology.

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² Annual Book of ASTM Standards, Vol 12.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 12.02.

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides⁴

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques⁴

E 704 Test Method for Measuring Reaction Rates by Radioactivation of Uranium-238⁴

E 705 Test Method for Measuring Reaction Rates by Radioactivation of Neptunium-237⁴

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E706(IIC)⁴

E 944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance (IIA)⁴

E 1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance, E706(IIIA)⁴

E 1018 Guide for Application of ASTM Evaluated Cross Section Data File, Matrix E 706 (IIB)⁴

2.2 Other Document:

NCRP Handbook of Radioactivity Measurements Procedures, NCRP Report No. 58.⁵

3. Terminology

3.1 Definitions:

3.1.1 Refer to Terminology E 170.

4. Summary of Test Method

4.1 The fission product ⁹⁹Mo is separated from the irradiated fission dosimeter by a solvent extraction technique. The activity of the ⁹⁹Mo is determined by counting the 740 or 778 keV, or both, gamma-energy peaks. This information combined with the fission yield of ⁹⁹Mo gives the reaction (fission) rate. When the proper cross section is used with the reaction rate, the equivalent fission rate can be determined.

5. Significance and Use

5.1 Refer to Guide E 844 for the selection, irradiation, and quality control of neutron dosimeters.

5.2 Refer to Practice E 261 for a general discussion of the measurement of neutron fluence rate and fluence. The neutron spectrum must be known in order to measure neutron fluence rates with a single detector. Also it is noted that cross sections

⁵ Available from National Council on Radiation Protection and Measurements (NCRP), 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814.

TABLE 1 Recommended Cumulative Fission Yields for ⁹⁹Mo Production

Fission Dosimeter	Thermal or Fast Neutron Field	Fission Yield, (%) ^{A,B}
²³⁵ U	T	6.18839 ± 1.4 %
	F	5.91653 ± 1.4 %
²³⁸ U	F	6.18839 ± 1.4 %
²³⁹ Pu	T	6.19863 ± 1.4 %
	F	5.95472 ± 2 %
²³⁷ Np	F	6.11547 ± 4 %
²³² Th	F	2.95528 ± 2 %
²⁴¹ Am	T	6.63629 ± 2.8 %
	F	5.40483 ± 6 %

^A These ENDF/B-VI values are considered the best available data. The uncertainties are expressed as a percentage of the fission yield.

^B England, T. R., and Rider, B. F., *ENDF-349 Evaluation and Compilation of Fission Product Yields*, Los Alamos National Laboratory, Los Alamos, NM, report LA-UR-94-3106, ENDF-349, October 1994.

are continuously being reevaluated. The latest recommended fission cross sections and details on how they can be obtained are discussed in Guide E 1018.

5.3 The reaction rate of a detector nuclide of known cross section, when combined with information about the neutron spectrum, permits the determination of the magnitude of the fluence rate impinging on the detector. Furthermore, if results from other detectors are available, the neutron spectrum can be defined more accurately. The techniques for fluence rate and fluence determinations are explained in Practice E 261.

5.4 ⁹⁹Mo is a radioactive nuclide formed as a result of fission. Its half-life is 65.94 h.⁶ Recommended fission yields for ⁹⁹Mo production are given in Table 1.

5.5 Because of the short half-life and substantial fission yield,⁹⁹Mo is useful for short irradiation times or moderate neutron fields. Its decay constant and yield are known more accurately than those of many fission products, so it is sometimes used as a standard or basic reaction with which other measurements can be normalized.

6. Apparatus

6.1 *Nal—(Tl), or High Resolution, Gamma-Ray Scintillation Spectrometer*, using a single- or a multichannel pulse height analyzer. See Test Methods E 181.

6.2 *Balance*, providing the accuracy and precision required by the experiment.

6.3 *Separatory Funnels*, 125-mL capacity.

6.4 *Centrifuge*, clinical-type, accommodating 50-mL centrifuge tubes.

6.5 *Filter Chimney*—The filter chimney must hold a nominal 25.4-mm filter-paper disk rigidly in place during filtration. It is commercially available from many laboratory supply houses. A diagram of such an apparatus is shown in Fig. 1.

6.6 *Drying Oven*.

6.7 *Glass Filter Crucibles*, fine-porosity.

6.8 *Filter Papers*, open, rapid filtering, and fine texture retentive.

7. Reagents and Materials

7.1 *Purity of Fission Dosimeters*—High purity uranium,

⁶ *Nuclear Wallet Cards*, compiled by J. K. Tuli, National Nuclear Data Center, July 1990.

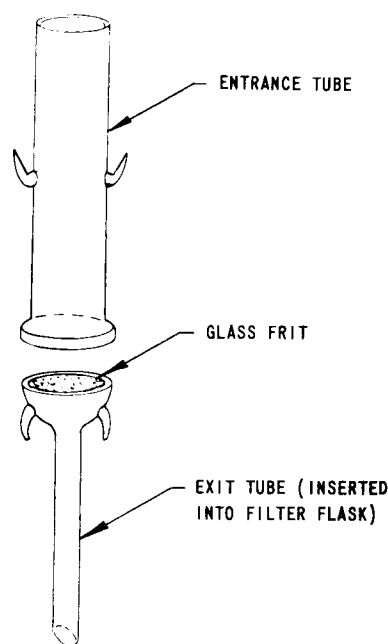


FIG. 1 Filter Chimney

plutonium, neptunium, and thorium in the form of alloy wire, foil, or oxide powder are available.

7.1.1 *Target material* shall be furnished with a certificate of analysis indicating any impurity concentrations.

7.1.2 *Fission dosimeters* shall be encapsulated in hermetically sealed containers to avoid loss of materials and for health-hazard requirements. (Plutonium should be doubly sealed.)

7.1.3 *In thermal reactors threshold reaction dosimeters* (For example, ²³⁸U, ²³⁷Np, ²³²Th) shall be shielded from thermal neutrons with elemental, or compounds of, cadmium, gadolinium, or boron to prevent fission production from trace quantities (>40 ppm) of ²³⁵U, and ²³⁹Pu and to suppress buildup of interfering fissionable nuclides, for example, ²³⁹Pu in the ²³⁸U dosimeter, ²³⁸Np and ²³⁸Pu in the ²³⁷Np dosimeter, and ²³³U in the ²³²Th dosimeter (see Guide E 844).

7.2 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁷ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.3 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7.4 *Ammonium Acetate Solution (5 %)*—Dissolve 40 g of ammonium acetate in 760 mL of water.

⁷ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."