Designation: E1297 – 18

Standard Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Niobium[1]

This standard is issued under the fixed designation E1297; 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 This test method describes procedures for measuring reaction rates by the activation reaction ^{93}Nb(n,n')^{93m}Nb.

1.2 This activation reaction is useful for monitoring neutrons with energies above approximately 0.5 MeV and for irradiation times up to about 48 years (three half-lives), provided that the analysis methods described in Practice E261 are followed.

1.3 With suitable techniques, fast-neutron reaction rates for neutrons with energy distribution similar to fission neutrons can be determined in fast-neutron fluences above about 10^{16} cm^{-2}. In the presence of high thermal-neutron fluence rates ($>10^{12} cm^{-2} \cdot s^{-1}$), the transmutation of ^{93m}Nb due to neutron capture should be investigated. In the presence of high-energy neutron spectra such as are associated with fusion and spallation sources, the transmutation of ^{93m}Nb by reactions such as (n,2n) may occur and should be investigated.

1.4 Procedures for other fast-neutron monitors are referenced in Practice E261.

1.5 Fast-neutron fluence rates can be determined from the reaction rates provided that the appropriate cross section information is available to meet the accuracy requirements.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *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]

D1193 Specification for Reagent Water

E170 Terminology Relating to Radiation Measurements and Dosimetry

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E181 Test Methods for Detector Calibration and Analysis of Radionuclides

E185 Practice for Design of Surveillance Programs for Light-Water Moderated Nuclear Power Reactor Vessels

E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques

E262 Test Method for Determining Thermal Neutron Reaction Rates and Thermal Neutron Fluence Rates by Radioactivation Techniques

E456 Terminology Relating to Quality and Statistics

E844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance

E944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance

E1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance

E1006 Practice for Analysis and Interpretation of Physics Dosimetry Results from Test Reactor Experiments

E1018 Guide for Application of ASTM Evaluated Cross Section Data File

3. Terminology

3.1 *Definitions*—The definitions stated in Terminology E170 and E456 are applicable to this test method.

4. Summary of Test Method

4.1 High purity niobium is irradiated in a neutron field producing radioactive ^{93m}Nb from the ^{93}Nb(*n,n'*)^{93m}Nb reaction. The metastable state decays to the ground state by the

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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.

virtual emission of 30 keV gamma rays that are all internally converted giving rise to the actual emission of orbital electrons followed by X rays.

4.2 Sources of the irradiated niobium are prepared for X ray or liquid scintillation counting.

4.3 The X rays emitted as a result of the decay of ^{93m}Nb are counted, and the reaction rate, as defined in Practice E261, is calculated from the decay rate and irradiation conditions.

4.4 The neutron fluence rate may then be calculated from the appropriate spectral-weighted neutron activation cross section as defined by Practice E261.

5. Significance and Use

5.1 Refer to Practice E261 for a general discussion of the determination of decay rates, reaction rates, and neutron fluence rates with threshold detectors (**1-29**).[3] Refer to Practice E1006, Practice E185 and Guide E1018 for the use and application of results obtained by this test method.(**30-32**)

5.2 The half-life of ^{93m}Nb is 16.1 (2)[4] years[5](**34**) and has a K X-ray emission probability of 0.11442 ± 3.356 % per decay (**35**). The K_α and K_β X-rays of niobium are at 16.521–16.615 and 18.607–18.9852 keV, respectively (**35**). The recommended ^{93}Nb(n,n')^{93m}Nb cross section comes from the International Reactor Dosimetry and Fusion File (IRDFF version 1.05, cross section compendium (**36**), and is shown in Fig. 1. This nuclear data evaluation is part of the Russian Reactor Dosimetry File (RRDF), cross section evaluations (**37**). The nuclear decay data referenced here are not taken from the latest dosimetry recommended database (**33**) but are selected to be consistent with the nuclear data used in the recommended IRDFF evaluation.

5.3 Chemical dissolution of the irradiated niobium to produce very low mass-per-unit area sources is an effective way to obtain consistent results. The direct counting of foils or wires can produce satisfactory results provided appropriate methods and interpretations are employed. It is possible to use liquid scintillation methods to measure the niobium activity provided the radioactive material can be kept uniformly in solution and appropriate corrections can be made for interfering activities.

5.4 The measured reaction rates can be used to correlate neutron exposures, provide comparison with calculated reaction rates, and determine neutron fluences. Reaction rates can be determined with greater accuracy than fluence rates because of the current uncertainty in the cross section versus energy shape.

5.5 The ^{93}Nb(n,n')^{93m}Nb reaction has the desirable properties of monitoring neutron exposures related to neutron damage of nuclear facility structural components. It has an energy response range corresponding to the damage function of steel and has a half-life sufficiently long to allow its use in very long exposures (up to about 48 years). Monitoring long exposures is useful in determining the long-term integrity of nuclear facility components.

6. Interferences

6.1 Pure niobium in the forms of foil and wire is available and easily handled as a metal. When thin niobium is irradiated, it may become brittle and fragile, thus requiring careful handling or encapsulation to prevent damage or loss of the niobium. Refer to Guide E844 for the selection, irradiation, and quality control of neutron dosimeters.

6.2 There are some distinct advantages and limitations to three measurement techniques identified in 5.3. It is the responsibility of the user to evaluate these and determine the optimum technique for the situation.

6.2.1 Low mass source X-ray spectrometry advantages include sufficient energy resolution to eliminate other X-ray emissions, stable long life sources, reduced interference fluorescence due to other radionuclides, small and precise background corrections, and minimal X-ray source self-absorption corrections. Limitations are low counting efficiency, complex source preparation, and use of hazardous chemicals.

6.2.2 Direct X-ray spectrometry of metal (foil or wire) sources has the advantages of simple source preparation, stable long life sources, sufficient energy resolution to eliminate other X-ray emissions, small and precise background corrections, and no use of hazardous chemicals. Limitations are low counting efficiency, large X-ray source self-absorption corrections, larger corrections for interference fluorescence due to the other radionuclides, and source geometry control.

6.2.3 Liquid scintillation counting advantages include very high detection efficiency, reproducible source preparation, and no source self absorption corrections. Limitations include specialized calibration techniques to reduce interference from other radionuclides, limited source stability, use of hazardous chemicals, and disposal of hazardous chemical waste.

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

[4] The value of uncertainty, in parenthesis, refers to the corresponding last digits, thus 16.1(2) corresponds to 16.1 ± 0.2, which corresponds to 16.1 ± 1.24 %.

[5] One year is defined to be 365.242198 days – 31556926 seconds in the source documents referenced (**33**).

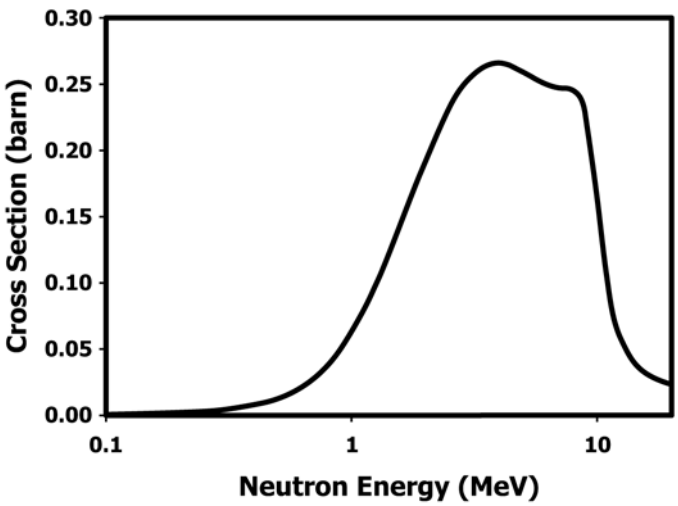

FIG. 1 RRDF/IRDFF-1.05 Cross Section Versus Energy for the ^{93}Nb(n,n')^{93m}Nb Reaction

7. Apparatus

7.1 *X-ray Spectrometer,* using a Si(Li) detector or a Ge detector and a multichannel pulse-height analyzer. For more information, refer to Test Methods E181 and E1005.

7.2 *Precision Balance,* able to achieve the required accuracy.

7.3 *Beakers,* 50 mL polyethylene; pycnometer (weighing bottle), 50 mL polyethylene; volumetric pipets, 10 µL to 5 mL.

7.4 *Gamma Ray Spectrometer,* using a Ge detector and a multichannel pulse-height analyzer. Refer to Test Method E181.

7.5 *Liquid Scintillation Counter.*

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] 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.

8.2 *Purity of Water*—Unless otherwise indicated, any water used shall be understood to mean reagent water as defined by Type I of Specification D1193.

8.3 *Hydrofluoric Acid*—Concentrated (32*M*) hydrofluoric acid (HF).

8.4 *Nitric Acid*—Concentrated (16*M*) nitric acid (HNO_3).

8.5 *Niobium Metal*—The purity of the niobium is important in that no impurities (such as tantalum) should be present to produce long-lived radionuclides that interfere with the ^{93m}Nb activity determination. To avoid problems from tantalum, the niobium should have the lowest tantalum content possible. Niobium metal in the form of foil and wire with tantalum content of about 5 ppm (parts per million) or less is obtainable and can be used under most conditions. The niobium material should be tested for interfering radioactivity by neutron activation techniques.

8.6 *Encapsulation Material*—The encapsulation material (such as quartz, stainless steel, aluminum, etc.) should be selected to prevent corrosion of the niobium during irradiation and to be compatible with the irradiation environment and post-irradiation handling. If thermal and epithermal neutron filters or shrouds are used, these materials (such as cadmium, tantalum, gadolinium, etc.) must also be compatible with the encapsulation and irradiation environment.

8.7 *Analytical Paper*—Analytical grade filter paper of uniform thickness (about 0.076 cm) and density (about 8 mg cm^{-2}). The paper can be cut or obtained precut to the desired size (usually between 0.5 and 1.5 cm diameter) that is compatible with the activity concentration of the solution and the counting conditions. The paper should be able to absorb as much liquid as is necessary and not decompose from the acid. TFE-fluorocarbon rings with an inside diameter matching the outside diameter of the filter paper disks so they fit together with light contact.

8.8 *Support and Cover Materials*—Thin plastic film and plastic tape materials are useful to support and cover the filter paper sources. They should be strong enough to contain the sources and thin enough to minimize attenuation of the X rays.

8.9 *Source Holder*—A source holder must be used to accurately and reproducibly position the sources for the counting geometry to be used. The source holder should be constructed of low density materials such as aluminum or plastic.

8.10 *Liquid Scintillation Materials*—Vials, emulsion scintillant (xylene-based), chelating agent (di-2-ethylhexyl phosphoric acid).

9. Procedure

9.1 Determine the size and shape of the niobium sample being irradiated. Consider the convenience in handling and available irradiation space. Ensure that sufficient ^{93m}Nb activity will be produced to permit accurate radioassay. Typically, samples of 0.2 to 20 mg of niobium may be used, but a preliminary calculation of the expected production of ^{93m}Nb will aid in selecting the appropriate mass for the irradiation.

9.2 Accurately weigh the niobium sample being irradiated.

9.3 Encapsulate the niobium sample so that it can be retrieved and identified following the irradiation. Record the sample identification, sample weight, and exact details of the encapsulation. Shroud the niobium with neutron filter material if necessary. If the thermal-to-fast neutron fluence rate ratio is high (greater than 5) or the tantalum impurity is high (greater than 10 ppm), use neutron filter materials, if possible.

9.4 Irradiate the niobium samples. Keep an accurate record of the irradiation history including neutron level versus time, starting and ending time of the irradiation, and the periods when the neutron level is zero. Record the spatial position of the sample in the irradiation facility.

9.5 After the irradiation, retrieve and identify the irradiated sample. Take necessary precautions to avoid personnel overexposure to radiation and the spread of radioactive contamination.

9.6 A waiting time between the end of irradiation and the start of counting may be necessary to allow ^{92m}Nb or ^{95}Nb, or both, to decay to an insignificant level. Check the samples for activity from contamination by other materials or reactions (see Test Method E262) and for any material adhering to the sample. Check the weight of the sample. If necessary, clean and reweigh the sample.

9.7 *X-Ray Source Preparation and Counting:*

9.7.1 If the metal is being dissolved and reduced to a low mass-per-unit area source, dissolve the sample by placing it in

[6] "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."