



Standard Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Niobium¹

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1. Scope

1.1 This test method describes procedures for measuring reaction rates by the activation reaction $^{93}\text{Nb}(n,n')^{93\text{m}}\text{Nb}$.

1.2 This activation reaction is useful for monitoring neutrons with energies above approximately 0.5 MeV and for irradiation times up to about 30 years.

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 ($>10^{12}\text{cm}^{-2}\cdot\text{s}^{-1}$), the transmutation of $^{93\text{m}}\text{Nb}$ 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 $^{93\text{m}}\text{Nb}$ by reactions such as (n,2n) may occur and should be investigated.

1.4 Procedures for other fast-neutron monitors are referenced in Practice E 261.

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

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 170 Terminology Relating to Radiation Measurements and Dosimetry³

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides³

E 185 Practice for Conducting Surveillance Tests for Light-

Water Cooled Nuclear Power Reactor Vessels, E706 (IF)³

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques³

E 262 Test Method for Determining Thermal Neutron Reaction and Fluence Rates by Radioactivation Techniques³

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E706 (IIC)³

E 944 Practice for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance³

E 1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance, E706 (IIIA)³

E 1006 Practice for Analysis and Interpretation of Physics Dosimetry Results for Test Reactors, E706 (II)³

E 1018 Guide for Application of ASTM Evaluated Cross Section Data File (ENDF/A)—Cross Section and Uncertainty File, E706 (IIB)³

3. Terminology

3.1 *Definitions*—The definitions stated in Terminology E 170 are applicable to this test method.

4. Summary of Test Method

4.1 High purity niobium is irradiated in a neutron field producing radioactive $^{93\text{m}}\text{Nb}$ from the $^{93}\text{Nb}(n,n')^{93\text{m}}\text{Nb}$ reaction. The metastable state decays to the ground state by the 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 $^{93\text{m}}\text{Nb}$ are counted, and the reaction rate, as defined in Practice E 261, 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 E 261.

5. Significance and Use

5.1 Refer to Practice E 261 for a general discussion of the determination of decay rates, reaction rates, and neutron

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 12.02.

fluence rates with threshold detectors (1-29).⁴ Refer to Practice E 1006 and Guide E 1018 for the use and application of results obtained by this test method.(34-36)

5.2 The half-life of ^{93m}Nb is 5890 ± 50 days (30) and has a K X-ray emission probability of 0.1104 ± 0.0035 per decay (30). The K_α and K_β X-rays of niobium are at 16.52–16.62 and 18.6–19.07 keV, respectively. The recommended ⁹³Nb (n,n')^{93m}Nb cross section comes from the IRDF-90 cross section compendium (31) and is shown in Fig. 1.

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 ⁹³Nb(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 40 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

⁴ The boldface numbers in parentheses refer to the list of references at the end of this test method.

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 E 844 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.

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 E 181 and E 1005.

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

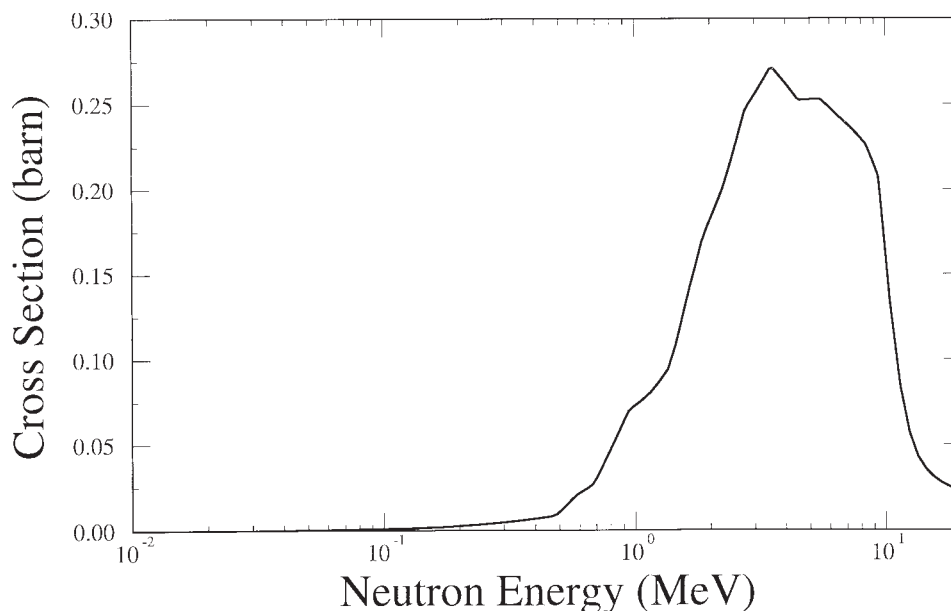


FIG. 1 IRDF-90 Cross Section Versus Energy for the ⁹³Nb(n,n')^{93m}Nb Reaction

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

7.4 *Gamma Ray Spectrometer*, using a Ge detector and a multichannel pulse-height analyzer. Refer to Test Method E 181.

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.⁵ 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 D 1193.

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

8.4 *Nitric Acid*—Concentrated (16*M*) nitric acid (HNO₃).

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 pre-cut 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 over-exposure 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 ⁹⁵Nb, or both, to decay to an insignificant level. Check the samples for activity from contamination by other materials or reactions (see Test Method E 262) 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 a preweighed 50 mL polyethylene or TFE-fluorocarbon (non-wettable) beaker and adding enough concentrated hydrofluoric acid to cover the sample (usually about 1 to 10 mL of HF). Add concentrated nitric acid dropwise to start dissolution; as dissolution slows, add additional drops to maintain a controlled slow rate of dissolution until the entire sample dissolves. After dissolution is complete, bring the final volume of solution to the desired amount by adding distilled water. Weigh the solution in the beaker if mass aliquoting is used. A preweighed polyethylene pycnometer (weighing bottle) is recommended for mass aliquoting. The ratio of the niobium mass to the solution mass determines the concentration of niobium in the solution. When transferring the solution from one container to another, ensure that all of the solution is transferred by using multiple rinses. Use accurately calibrated pipets if volumetric aliquoting is performed.

⁵ "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."

9.7.2 Deposit the desired amount of the solution on a filter paper disk surrounded by a TFE-fluorocarbon ring to produce a counting source. Deposit the solution on the paper drop-by-drop so the paper does not become saturated but is uniformly wetted. The deposits are allowed to dry by evaporation of the solvent. Gently remove the paper disk from the TFE-fluorocarbon ring. Seal the sources between thin layers of plastic or plastic tape to contain the niobium. Mount the sources on the source holder for the counting geometry being used. Determine and record the amount of niobium in each source.

9.7.3 Count the prepared sources using an X-ray spectrometer with either an Si(Li) or a Ge detector with a beryllium window and designed to count X rays in the energy range between about 5 and 50 keV. Refer to Test Methods E 181 and Practice E 261. It is assumed that the persons doing the counting are knowledgeable in the operation and use of the counting apparatus and the handling and counting of X-ray sources.

9.7.4 The ^{93m}Nb activity can be determined from the K X-ray emission rates by direct comparison to a certified standard of ^{93m}Nb prepared and counted under the same conditions. Alternatively, the detection efficiency for the spectrometer system may be determined from other standardized K X-ray sources and applied to the counting of the unknown sources as described in Test Methods E 181 and Ref (8).

9.7.5 If the prepared sources contain significant and variable amounts of niobium relative to each other or to the standard source, correction for self-attenuation may be necessary. These corrections can be determined as described in Test Methods E 181. The accuracy of these corrections will depend on the uniformity of the distribution of the niobium in the sources. If possible, sources with less than 1 mg/cm² of niobium should be prepared for counting to minimize the self-attenuation of the sources.

9.8 Comparison Counting:

9.8.1 If direct counting of metal niobium foils or wires is being done by comparison with appropriate standard sources of ^{93m}Nb in the same form and geometry as the unknown sources, or by comparison with certified standards and with appropriate corrections made for geometry differences, all of the factors that may influence the results must be appropriately controlled. In this approach, the ^{93m}Nb emission rate may be compared to that of a fluence standard to produce a fluence relative to that of the standard and the neutron field in which it was irradiated. Fluence standards in the same form as the foils or wires are produced in standardized neutron fields. See Practice E 261 for more information.

9.8.2 If foils are counted and compared to standard ^{93m}Nb sources of different form and geometry, corrections for self attenuation and geometry are necessary. These corrections may be determined as described in Test Methods E 181. The geometry corrections are minimized if the source-to-detector spacing is large. The geometry correction to account for a disk source related to a point source will be less than 2 % if the ratio of the disk radius to the source-to-detector distance is less than 0.2 and the ratio of the detector radius to the source-to-detector distance is less than 0.4. The X-ray attenuation correction for a

0.1 mm thick, 0.63 cm diameter niobium metal foil is about a factor of 1.9. Counting methods are described in 9.7.3.

9.9 When the fluorescence effect of ^{182}Ta or other activity is not negligible in a niobium neutron monitor source, correction must be made. This correction is determined for the source geometry and niobium quantity being used by experimentally measuring the K X-ray fluorescence rate-per-unit ^{182}Ta or other activity as a function of niobium mass. The amount of ^{182}Ta or other activity in the sample is measured by gamma-ray spectrometry (see Test Methods E 181), and the correction is determined from the results. If the ^{182}Ta activity is less than 2 % of the ^{93m}Nb activity in a 0.1 mm thick niobium foil, the correction of ^{182}Ta induced fluorescence will be less than about 5 %. The correction is less for lighter foils and greater for heavier foils.

9.10 Liquid Scintillation Counting:

9.10.1 If liquid scintillation counting of the irradiated niobium is being used to determine the ^{93m}Nb activity, the niobium must be dissolved (see 9.7.1) and an accurate amount of the niobium placed in the liquid scintillant. Place an accurate and reproducible amount of liquid scintillant, chelating agent, and niobium solution in a standard scintillation vial to form a “cocktail” counting source. In this approach, the ^{93m}Nb activity is determined by comparing the liquid scintillation counting rates of an unknown source and a known source or by using the detection efficiency of the counter. Make corrections for the chemical interference changes in the process of light production (quenching).

9.10.2 Prepare the counting source by placing the xylene-based emulsion scintillant⁶ along with 10 volume % of chelating agent (di-2-ethylhexyl phosphoric acid, HDEHP) into a standard liquid scintillation counting vial. Add the dissolved niobium solution to the mixture and mix vigorously. This solution will remain in a stable form for about one week. Keep the amounts of chemicals in all sources (blank, unknown, and known) as constant as possible to minimize quenching differences.

9.10.3 Count the sources as soon as possible following preparation and after temperature equilibrium is established since they are stable only for about one week. Count the sources, blanks, and standards in the same manner and correct them for instrument background. Differences in the quenching can be monitored using the “external source” technique.

9.10.4 If interfering radionuclides are present in the source, the responses to these must be determined and corrections made. Counting in optimized energy windows can be used in conjunction with the responses of the interfering radionuclides to obtain the ^{93m}Nb activity. It is assumed that the persons doing the counting are knowledgeable in the operation and use of the counting apparatus, the handling and counting of liquid scintillation sources, and the reduction of liquid scintillation counting data.

10. Calculation

10.1 Calculate the reaction rate, R_s , as follows, assuming the

⁶ Insta Gel, available from Packard Instrument Co., One State St., Meriden, CT 06450, has been found satisfactory for this purpose.

irradiation was at a constant neutron level and there were no interruptions:

$$R_s = A/N_o[1 - \exp(-\lambda t_i) (\exp - \lambda t_w)]$$

where:

- A = number of ^{93m}Nb disintegrations, s^{-1} , measured during the counting period and corrected for all interferences,
 N_o = number of ^{93}Nb atoms in the sample,
 λ = disintegration constant for $^{93m}\text{Nb} = 1.362 \times 10^{-9} \text{ s}^{-1}$,
 t_i = irradiation time duration, s, and
 t_w = elapsed time between the end of irradiation and counting, s.

NOTE 1—If the irradiation is not at a constant neutron level or if the irradiation duration approaches the recommended maximum, corrections will be necessary. Refer to Practice E 261 for procedures to make these corrections.

10.2 Refer to Practice E 261 and Practice E 944 for procedures to determine fast-neutron fluence rate and fluence.

11. Report

11.1 Practice E 261 describes how data should be reported.

12. Precision and Bias

NOTE 2—Measurement uncertainty is described by a precision and bias statement in this standard. Another acceptable approach is to use Type A and B uncertainty components, (31 and 32). This Type A/B uncertainty specification is now used in International Organization for Standardization (ISO) standards and this approach can be expected to play a more prominent role in future uncertainty analyses.

12.1 Disintegration rates of ^{93m}Nb from niobium neutron dosimeters can be determined with a bias of $\pm 2\%$ (1S %) and with a precision of $\pm 1\%$ (1S %) by use of a ^{93m}Nb reference standard. By using a detector calibrated for counting efficiency and the K X-ray emission probability, the disintegration rate can be determined with a bias of $\pm 3\%$ (1S %) and with a precision of $\pm 1\%$ (1S %).

12.2 The $^{93}\text{Nb}(n,n')^{93m}\text{Nb}$ reaction rate can be determined with approximately the same bias and precision as the disintegration rate if the irradiation history is well known.

13. Keywords

13.1 activation; activation reaction; cross section; dosimetry; fast neutron monitor; neutron dosimeter; neutron metrology; niobium; pressure vessel surveillance; reaction rate; reaction rate monitor

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