Designation: E 526 – 97 (Reapproved 2002)

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

This standard is issued under the fixed designation E 526; 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 covers procedures for measuring reaction rates by the activation reactions ^{46}Ti (n, p)^{46}Sc +^{47}Ti (n, np)^{46}Sc.

NOTE 1—Since the cross section for the (n,np) reaction is relatively small for energies less than 12 MeV and is not easily distinguished from that of the (n,p) reaction, this test method will refer to the (n,p) reaction only.

1.2 The reaction is useful for measuring neutrons with energies above approximately 4.4 MeV and for irradiation times up to about 250 days (for longer irradiations, see Practice E 261).

1.3 With suitable techniques, fission-neutron fluence rates above 10^9 $cm^{-2}\cdot s^{-1}$ can be determined. However, in the presence of a high thermal-neutron fluence rate,^{46}Sc depletion should be investigated.

1.4 Detailed procedures for other fast-neutron detectors are referenced in Practice E 261.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry[2]

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides[2]

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques[2]

E 262 Test Method for Determining Thermal Neutron Reaction and Fluence Rates by Radioactivation Techniques[2]

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E 706 (IIC)[2]

E 944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance, (IIA)[2]

E 1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance, E 706 (IIIA)[2]

E 1018 Guide for Application of ASTM Evaluated Cross Section Data Files, Matrix E 706 (IIB)[2]

3. Terminology

3.1 *Definitions*:

3.1.1 Refer to Terminology E 170.

4. Summary of Test Method

4.1 High-purity titanium is irradiated in a fast-neutron field, thereby producing radioactive^{46}Sc from the^{46}Ti (n, p)^{46}Sc activation reaction.

4.2 The gamma rays emitted by the radioactive decay of^{46}Sc are counted in accordance with Methods E 181 and the reaction rate, as defined by Test Method E 261, is calculated from the decay rate and the irradiation conditions.

4.3 The neutron fluence rate above about 4.4 MeV can then be calculated from the spectral-weighted neutron activation cross section as defined by Test Method E 261.

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 Test Method E 261 for a general discussion of the determination of fast-neutron fluence rate with threshold detectors.

5.3 Titanium has good physical strength, is easily fabricated, has excellent corrosion resistance, has a melting temperature of 1675°C, and can be obtained with satisfactory purity.

5.4 ^{46}Sc has a half-life of 83.81 days.[3] The^{46}Sc decay[4] emits a 0.8893 MeV gamma 99.984 % of the time and a second gamma with an energy of 1.1205 MeV 99.987 % of the time.

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

Current edition approved June 10, 1997. Published May 1998. Originally published as E 526 – 76. Last previous edition E 526 – 92.

[2] *Annual Book of ASTM Standards*, Vol 12.02.

[3] Nuclear Wallet Cards, National Nuclear Data Center, prepared by Jagdish K. Tuli, July 1990.

[4] Evaluated Nuclear Structure Data File (ENSDF), maintained by the National Nuclear Data Center (NNDC), Brookhaven National Laboratory, on behalf of the International Network for Nuclear Structure Data Evaluation.

5.5 The isotopic content of natural titanium recommended for ^{46}Ti is 8.012 %.[5]

5.6 The radioactive products of the neutron reactions $^{47}Ti(n,p)^{47}Sc$ ($T_{1/2}$ = 3.35 d) and $^{48}Ti(n,p)^{48}Sc$ ($T_{1/2}$ = 1.82 d), might interfere with the analysis of ^{46}Sc.

5.7 Contaminant activities (for example, ^{65}Zn and ^{182}Ta) might interfere with the analysis of ^{46}Sc. See Section 7.1.2 and 7.1.3 or more details on the ^{182}Ta and ^{65}Zn interference.

5.8 ^{46}Ti and ^{46}Sc have cross sections for thermal neutrons of 0.6 and 8 barns, respectively;[6] therefore, when an irradiation exceeds a thermal-neutron fluence greater than about 2×10^{21} cm^{-2}, provisions should be made to either use a thermal-neutron shield to prevent burnup of ^{46}Sc or measure the thermal-neutron fluence rate and calculate the burnup.

5.9 Fig. 1 shows a plot of cross section versus neutron energy for the fast-neutron reactions of titanium which produce ^{46}Sc (that is, $^{Nat}Ti(n,X)^{46}Sc$). Included in the plot is the $^{46}Ti(n,p)$ reaction[7] and the $^{47}Ti(n,np)$ contribution to the ^{46}Sc production,[8] normalized (to 14.7 MeV)[9] per ^{46}Ti atom. This figure is for illustrative purposes only to indicate the range of response of the $^{46}Ti(n,p)$ reaction. Refer to Guide E 1018 for descriptions of recommended tabulated dosimetry cross sections.

6. Apparatus

6.1 *NaI(Tl) or High Resolution Gamma-Ray Spectrometer*. Because of its high resolution, the germanium detector is useful when contaminant activities are present. See Methods E 181 and E 1005.

[5] *Isotopic Compositions of the Elements 1983*, International Union of Pure and Applied Chemistry, Vol. 56, Pergamon Press, 1984.

[6] *Chart of the Nuclides*, Knolls Atomic Power Laboratory, 14th Ed, April 1988.

[7] "International Reactor Dosimetry File (IRDF-90)," assembled by N. P. Kocherov, et al., International Atomic Energy Agency, Nuclear Data Section, IAEA–NDS–141, Rev 0, August 1990.

[8] ENDF/B-V Dosimetry Tape 531-G, Mat. No. 6427 (22-Ti-46), October 1979.

[9] J. W. Meadows, D. L. Smith, M. M. Bretscher, and S. A. Cox, "Measurement of 14.7 MeV Neutron-Activation Cross Sections for Fusion," *Annals of Nuclear Energy*, Vol 1, No. 9, 1987.

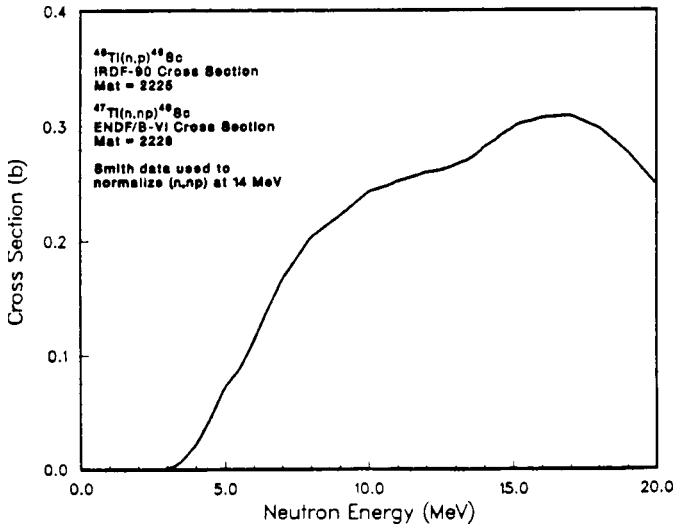

FIG. 1 $^{Nat}Ti(n,X)^{46}Sc$ Cross Section (Normalized per Ti-46 Atom)

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

6.3 *Digital Computer*, useful for data analysis (optional).

7. Materials

7.1 *Titanium Metal*—High-purity titanium metal in the form of wire or foil is available.

7.1.1 The metal should be tested for impurities by a neutron activation technique. If the measurement is to be made in a thermal-neutron environment, scandium impurity must be low because of the reaction, $^{45}Sc\ (n,\gamma)^{46}Sc$. To reduce this interference, the use of a thermal-neutron shield during irradiation would be advisable if scandium impurity is suspected. As an example, when a titanium sample containing 6 ppm scandium has been irradiated in a neutron field with equal thermal and fast-neutron fluence rates about 1 % of the ^{46}Sc in the sample is due to the reaction $^{45}Sc\ (n,\gamma)^{46}Sc$.

7.1.2 Tantalum impurities can also cause a problem. The low-energy response of the $^{181}Ta(n,\gamma)^{182}Ta$ reaction produces gamma activity that interferes with the measurement of ^{46}Sc radioactivity produced from the $^{46}Ti(n,p)^{46}Sc$ high-energy threshold reaction. The radioactive ^{182}Ta isotope has a half-life of $\tau_{1/2}$ = 114.43 d and emits a 1121.302 keV photon 34.7 % of the time. This photon is very close in energy to one of the two photons emitted by ^{46}Sc (889.3 keV and 1120.5 keV). Moreover, during the ^{46}Sc decay, the 1120.5 keV and 889.3 keV photons are emitted in true coincidence and the random coincidence between the 1121.302 keV photons from ^{182}Ta and the 889.3 keV photons from ^{46}Sc can affect the application of summing corrections when the counting is done in a close geometry and the ^{46}Sc activity is being monitoring with 889.3 keV photon.

7.1.3 Zinc contamination can lead to the production of ^{65}Zn via the $^{64}Zn(n,\gamma)^{65}Zn$ reaction. The radioactive ^{65}Zn isotope has a half-life of $\tau_{1/2}$ = 244.26 d and emits a 1115.518 keV photon 50.75 % of the time. These 1115.518 keV photons can interfere with the 1120.5 keV line from ^{46}Sc and require a multi-peak resolution. For a small contaminant level the ^{65}Zn line may be hidden in the background of the larger ^{46}Sc peak. There is no other high probability ^{65}Zn decay gamma with which to monitor or correct for the presence of zinc in the titanium sample.

7.1.4 Impurity problems in titanium are a particular concern for applications to reactor pressure vessel surveillance dosimetry because the $^{46}Ti(n,p)^{46}Sc$, along with the $^{63}Cu(n,\alpha)^{60}Co$ reaction, are the two highest-energy dosimetry reactions used to detect spectrum differences in reactor neutron environments. Incorrect radioactivity measurements of these two reactions can alter the high-energy end of the derived spectrum, and result in the incorrect prediction of neutron irradiation damage.

7.2 *Encapsulating Materials*—Brass, stainless steel, copper, aluminum, quartz, or vanadium have been used as primary encapsulating materials. The container should be constructed in such a manner that it will not create significant flux perturbation and that it may be opened easily, especially if the monitors must be removed remotely (see Guide E 844).

8. Procedure

8.1 Decide on the size and shape of the titanium sample to be irradiated, taking into consideration the size and shape of

the irradiation space. The mass and exposure time are parameters that can be varied to obtain a desired disintegration rate for a given neutron-fluence rate level. (See Guide E 844.)

8.2 Weigh the sample.

8.3 Irradiate the sample for the predetermined time period. Record the power level and any changes in power during the irradiation, the time at the beginning and end of each power level and the relative position of the monitors in the irradiation facility.

8.4 If the counting procedure available requires that the activity be pure ^{46}Sc, a waiting period of about 20 days is recommended between termination of the exposure and analyzing the samples for ^{46}Sc content. This allows the 44-h ^{48}Sc to decay so that there is no interference from the gamma rays emitted by ^{48}Sc, that is, the 0.175, 0.983, 1.037, and 1.312-MeV gamma rays. If the 0.159-MeV gamma ray emitted by 3.35-day ^{47}Sc interferes with counting conditions, a longer decay time may be necessary. The 5.75-min ^{51}Ti will usually have decayed by count time. However, gamma-ray spectra may be taken with germanium detectors soon after irradiation, if count rates are not excessive.

8.5 Check the sample for activity from cross-contamination by other irradiated materials. Clean, if necessary, and reweigh.

8.6 Analyze the sample for ^{46}Sc content in disintegrations per second using the gamma-ray spectrometer (see Methods E 181 and E 1005).

8.7 Disintegrations of ^{46}Sc nuclei produces 0.8893-MeV and 1.1205-MeV gamma rays with probabilities per decay of 0.99984 and 0.99997, respectively.[4] When analyzing either peak in the gamma-ray system, a correction for coincidence summing may be required if the sample is placed close to the detector (10 cm or less) (see Methods E 181).

9. Calculation

9.1 Calculate the saturation activity, A_s, as follows:

$$A_s = A/[(1 - \exp -[\lambda t_i])\,(\exp -[\lambda t_w])] \quad (1)$$

where:

A = ^{46}Sc disintegrations per second measured by counting,
λ = decay constant for ^{46}Sc = 9.570×10^{-8} s^{-1},
t_i = irradiation duration, s,
t_w = elapsed time between the end of irradiation and counting, s.

NOTE 2—The equation for A_s is valid if the reactor operated at essentially constant power and if corrections for other reactions (for example, impurities, burnout, etc.) are negligible. Refer to Test Method E 261 for more generalized treatments.

9.2 Calculate the reaction rate, R_s, as follows:

$$R_s = A_s/N_o \quad (2)$$

where:

A_s = saturation activity, and
N_o = number of ^{46}Ti atoms.

9.3 Refer to Test Method E 261 and Practice E 944 for a discussion of fast-neutron fluence rate and fluence.

10. Report

10.1 Test Method E 261 describes how data should be reported.

11. Precision and Bias

NOTE 3—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.[10,11] 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.

11.1 General practice indicates that disintegration rates can be determined with a bias of ±3 % (1S %) and with a precision of ±1 % (1S %). For a fission spectrum, the typical uncertainty in the spectrum-averaged cross section[12] is 2.4 %.

12. Keywords

12.1 activation reaction; cross section; dosimetry; nuclear metrology; pressure vessel surveillance; reaction rate; titanium

[10] B. N. Taylor, C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, National Institute of Standards and Technology, Gaithersburg, MD, 1994.

[11] *Guide in the Expression of Uncertainty in Measurement*, International Organization for Standardization, 1993, ISBN 92-67-10188-9.

[12] P. J. Griffin, "Comparison of Uncertainty Metrics for Calculated Dosimetry Activities," American Nuclear Society Proceedings of the 1996 Topical Meeting Radiation Protection and Shielding, held in No. Falmouth, Massachusetts, April 21–25, 1996, Vol. 1, pp. 27–35.