Designation: ~~E526 – 17ε1~~ E526 – 22

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

This standard is issued under the fixed designation E526; 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.

~~ε1 NOTE—Editorial changes, such as removing extra spacing, correcting notation and a variable, were made in November 2017.~~

## 1. Scope

1.1 This test method covers procedures for measuring reaction rates by the activation ~~reactions~~reaction $^{nat}Ti(n,X)^{46}$~~Ti(n,p)~~Sc. The "X" designation represents any combination of light particles associated with the production of the residual $^{46}$Sc ~~+~~ product. Within the applicable neutron energy range for fission reactor applications, this reaction is a properly normalized combination of three different reaction channels: $^{46}Ti(n,p)^{46}Sc$; $^{47}Ti(n, np)^{46}$~~Sc +~~Sc; and $^{47}Ti(n,d)^{46}Sc$.



NOTE 1—The $^{47}Ti(n,np)^{46}Sc$ reaction, ENDF-6 format file/reaction identifier MF=3, MT=28, is distinguished from the $^{47}Ti(n,d)^{46}Sc$ reaction, ENDF-6 format file/reaction identifier MF=3/MT=104, even though it leads to the same residual product (**1**).[2] The combined reaction, in the IRDFF-II library, has the file/reaction identifier MF=10/MT=5.

NOTE 2—The cross section for the combined $^{47}$~~Ti(n,np+d)~~Ti(n,np:d) reaction is relatively small for energies less than 12 MeV ~~and~~ and, in fission reactor spectra, the production of the residual $^{46}$Sc is not easily distinguished from that ~~of~~ due to the $^{46}$Ti(n,p) reaction. ~~This test method will apply to the composite $^{nat}Ti(n,X)^{46}Sc$ reaction that is typically used for dosimetry purposes.~~



1.2 The reaction is useful for measuring neutrons with energies above approximately 4.4 MeV and for irradiation times, under uniform power, up to about 250 days (for longer irradiations, or for varying power levels, see Practice E261).

1.3 With suitable techniques, fission-neutron fluence rates above $10^9$ $cm^{-2}·s^{-1}$ 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 E261.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *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.*

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[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.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*[3]

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
E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra 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
E1018 Guide for Application of ASTM Evaluated Cross Section Data File

## 3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminologies E170 and E456.

## 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 reaction as well as the $^{47}$Ti(n,np:d)$^{46}$Sc activation ~~reaction.~~reactions.

4.2 The gamma rays emitted by the radioactive decay of $^{46}$Sc are counted in accordance with Test Methods E181 and the reaction rate, as defined by ~~Test Method~~ Practice E261, 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~~ Practice E261.

## 5. Significance and Use



5.1 Refer to Guide E844 for the selection, irradiation, and quality control of neutron dosimeters.



5.2 Refer to ~~Test Method~~ Practice E261 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 ~~1668°C,~~1668 °C, and can be obtained with satisfactory purity.

5.4 $^{46}$Sc has a half-life of 83.787 (16)[4] days (~~1~~2). The $^{46}$Sc decay emits a 0.889271 (2) MeV gamma 99.98374 (35) % of the time and a second gamma with an energy of 1.120537 (3) MeV 99.97 (2) % of the time.

5.5 The ~~isotopic content of natural titanium recommended for~~ recommended "representative isotopic abundances" for natural titanium $^{46}$~~Ti is 8.25 %.~~ (~~2~~3) are:

8.25 (3) % $^{46}$Ti
7.44 (2) % $^{47}$Ti
73.72 (2) % $^{48}$Ti
5.41 (2) % $^{49}$Ti
5.18 (2) % $^{50}$Ti

[3] 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.

[4] The value of uncertainty, in parentheses, refers to the corresponding last digits, thus 14.958(2) corresponds to 14.958 ± 0.002.

5.6 The radioactive products of the neutron reactions $^{47}$Ti(n,p)$^{47}$Sc ($\tau_{1/2}$= 3.3485 (9) d) (~~1~~2) and $^{48}$Ti(n,p)$^{48}$Sc ($\tau_{1/2}$ = 43.67 h), (~~2~~3) 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 7.1.2 and 7.1.3 for more details on the $^{182}$Ta and $^{65}$Zn interference.

5.8 $^{46}$Ti and $^{46}$Sc have cross sections for thermal neutrons of 0.59 ± 0.18 and 8.0 ± 1.0 barns, respectively (~~3~~4); therefore, when an irradiation exceeds a thermal-neutron fluence greater than about $2 \times 10^{21}$ cm$^{-2}$, provisions should be made to either use a thermal-neutron shield to prevent burn-up of $^{46}$Sc or measure the thermal-neutron fluence rate and calculate the burn-up.

5.9 Fig. 1 shows a plot of the ~~Russian~~International Reactor Dosimetry ~~File (RRDF-2002)~~ and Fusion File, IRDFF-II cross section (~~4~~5) versus neutron energy for the fast-neutron reactions of titanium which produce $^{46}$Sc ~~[that~~(that is, $^{nat}$Ti(n,X)$^{46}$~~Sc]. This cross section is identical, for energies up to 20 MeV, to what is found in the latest International Atomic Energy Agency (IAEA) International Reactor Dosimetry and Fusion File, IRDFF-1.05~~ Sc). ~~(5).~~Included in the plot is the $^{46}$Ti(n,p) reaction and the $^{47}$~~Ti(n,np) contribution~~Ti(n,np:d) contributions to the $^{46}$Sc production, normalized per ~~46~~nat Ti atom with the individual isotopic contributions weighted using the natural abundances (~~2~~3). This figure is for illustrative purposes only and should be used to indicate the range of response of the $^{nat}$~~Ti(n,p)~~ Ti(n,X)$^{46}$Sc reaction. Refer to Guide E1018 for descriptions of recommended tabulated dosimetry cross sections. Fig. 2 compares the cross section for the $^{46}$~~Ti(N,p)~~Ti(n,p)~~47~~46Sc reaction to the current experimental database (**6, 7**).Fig. 3 compares the cross section for the $^{47}$~~Ti(N, np+d)~~ Ti(n,np:d) reaction to the current experimental database (**6, 7**).

## 6. Apparatus

6.1 *NaI(Tl) or High Resolution Gamma-Ray* ~~*Spectrometer.*~~ *Spectrometer*—Because of its high resolution, the germanium detector is useful when contaminant activities are present. See Test Methods E181 and E1005.

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

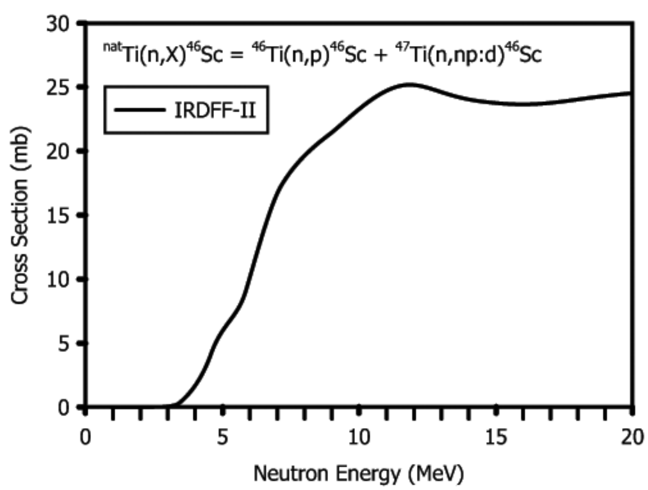


**FIG. 1 SAND-II 640-Group Histogram Representation of the ~~Nat~~nat Ti(n,X)$^{46}$Sc Cross Section (Normalized per ~~Ti-46~~ Elemental Ti Atom Using Natural Abundance ~~Data)~~Data), Represented By the Sum of the $^{nat}$Ti(n,p)$^{46}$Sc, $^{nat}$Ti(n,np)$^{46}$Sc, and $^{nat}$Ti(n,d)$^{46}$Sc Cross Section Components**

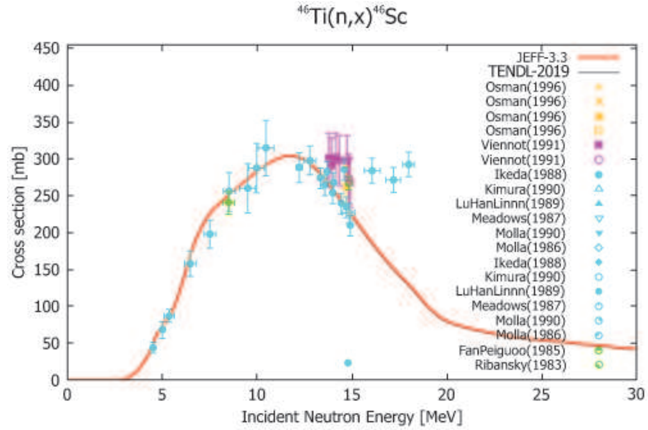


**FIG. 2 $^{46}$Ti(n,p)$^{46}$Sc Cross ~~Section,~~ Section (Normalized per Isotopic $^{46}$Ti Atom), from ~~RRDF-2002/IRDFF-1.05,~~IRDFF-II, with EXFOR Experimental Data**

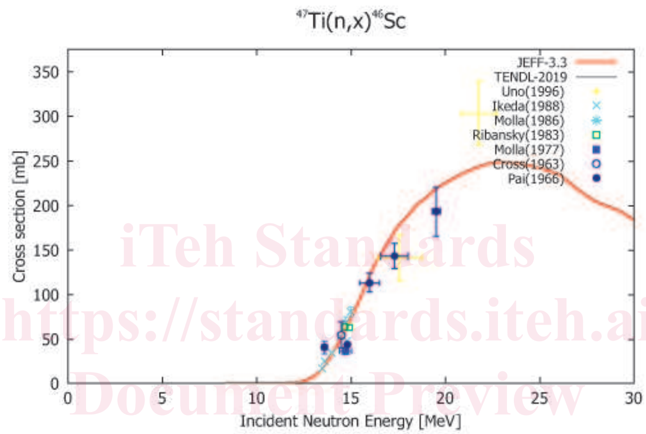


**FIG. 3 $^{47}$~~Ti(n,np+d)~~Ti(n,np:d)$^{46}$Sc Cross ~~Section,~~ Section (Normalized per Isotopic $^{47}$Ti Atom), from ~~RRDF-2002/IRDFF-1.05,~~IRDFF-II, with EXFOR Experimental Data**



thermal-neutron environment, scandium impurity must be low because of the reaction, $^{45}$Sc(n,γ)$^{46}$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,γ)$^{46}$Sc.

7.1.2 Tantalum impurities can also cause a problem. The low-energy response of the $^{181}$Ta(n,γ)$^{182}$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 $\tau_{1/2}$ = 114.61 (13) d and emits a 1121.290 (3) keV photon 35.17 (33) % of the time (~~1~~2). This photon is very close in energy to one of the two photons emitted by $^{46}$Sc (889.271 (2) keV and 1120.537 (3) keV). Moreover, during the $^{46}$Sc decay, the 1120.537 keV and 889.271 keV photons are emitted in true coincidence and the random coincidence between the ~~1121.395~~1121.290 keV photons from $^{182}$Ta and the 889.271 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 889.271 keV photon.

7.1.3 Zinc contamination can lead to the production of $^{65}$Zn via the $^{64}$Zn(n,γ)$^{65}$Zn reaction. The radioactive $^{65}$Zn isotope has a half-life of $\tau_{1/2}$ = 244.01 (9) d and emits a 1115.539 (2) keV photon 50.22 (11) ~~%~~% of the time. These 1115.539 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,α)$^{60}$Co reaction, are the two ~~highest-energy~~ highest-threshold 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 E844).

## 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 E844.)

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 43.67 (9) -h $^{48}$Sc (**~~2~~3**) to decay so that there is no interference from the gamma rays emitted by $^{48}$Sc, that is, the ~~0.175361, 0.983526, 1.037522, and 1.312120-MeV~~ 0.175361 (5), 0.983526 (12), 1.037522 (12), and 1.312120 (12)-MeV gamma rays (**~~2~~8**). If the ~~0.159373-MeV~~ 0.159373 (12)-MeV gamma ray emitted by ~~3.3485-day~~ 3.3485 (9) day $^{47}$Sc interferes with counting conditions, a longer decay time may be necessary. The 5.76-min (**~~2~~8**) $^{51}$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 Test Methods E181 and E1005).



8.7 Disintegrations of $^{46}$Sc nuclei produces 0.8893-MeV and 1.120537-MeV gamma rays with probabilities per decay of 0.9998374 (25) and 0.9997 (2), respectively. 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 Test Methods E181).



## 9. Calculation

9.1 Calculate the saturation activity, $A_s$, as follows:

$$A_s = A/\left[\left(1 - \exp^{-[\lambda t_i]}\right)\left(\exp^{-[\lambda t_w]}\right)\right] \tag{1}$$

where:

$A$ = $^{46}$Sc disintegrations per second measured by counting,
$\lambda$ = decay constant for $^{46}$Sc = 9.574918 × $10^{-8}$ $s^{-1}$,
$t_i$ = irradiation duration, s, and
$t_w$ = elapsed time between the end of irradiation and counting, s.

NOTE 3—The equation for $A_s$ is valid if the reactor is operated at essentially constant power and if corrections for other reactions (for example, impurities, burnout, etc.) are negligible. Refer to ~~Test Method~~ Practice E261 for more generalized treatments.

9.2 Calculate the reaction rate, $R$, as follows:

$$R = A_s/N_o \tag{2}$$

where:

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