Designation: E265 – 15 (Reapproved 2020)

Standard Test Method for Measuring Reaction Rates and Fast-Neutron Fluences by Radioactivation of Sulfur-32[1]

This standard is issued under the fixed designation E265; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method describes procedures for measuring reaction rates and fast-neutron fluences by the activation reaction $^{32}S(n,p)^{32}P$.

1.2 This activation reaction is useful for measuring neutrons with energies above approximately 3 MeV.

1.3 With suitable techniques, fission-neutron fluences from about 5×10^8 to 10^{16} n/cm^2 can be measured.

1.4 Detailed procedures for other fast-neutron detectors are described in Practice E261.

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

1.6 *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]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E181 Test Methods for Detector Calibration and Analysis of Radionuclides

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

E720 Guide for Selection and Use of Neutron Sensors for Determining Neutron Spectra Employed in Radiation-Hardness Testing of Electronics

E721 Guide for Determining Neutron Energy Spectra from Neutron Sensors for Radiation-Hardness Testing of Electronics

E844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance

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

E1018 Guide for Application of ASTM Evaluated Cross Section Data File

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology E170.

4. Summary of Test Method

4.1 Elemental sulfur or a sulfur-bearing compound is irradiated in a neutron field, producing radioactive ^{32}P by means of the $^{32}S(n,p)^{32}P$ activation reaction.

4.2 The beta particles emitted by the radioactive decay of ^{32}P are counted by techniques described in Methods E181 and the reaction rate, as defined in Practice E261, is calculated from the decay rate and irradiation conditions.

4.3 The neutron fluence above 3 MeV can then be calculated from the spectral-averaged neutron activation cross section, $\bar{\sigma}$, as defined in Practice E261.

5. Significance and Use

5.1 Refer to Guides E720 and E844 for the selection, irradiation, and quality control of neutron dosimeters.

5.2 Refer to Practice E261 for a general discussion of the determination of fast-neutron fluence and fluence rate with threshold detectors.

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[1] This test method is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.07 on Radiation Dosimetry for Radiation Effects on Materials and Devices.

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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.

5.3 The activation reaction produces ^{32}P, which decays by the emission of a single beta particle in 100 % of the decays, and which emits no gamma rays. The half life of ^{32}P is 14.284 (36)[3] days (**1**) [4] and the maximum beta energy is 1710.66 (21) keV (**1**).

5.4 Elemental sulfur is readily available in pure form and any trace contaminants present do not produce significant amounts of radioactivity. Natural sulfur, however, is composed of ^{32}S (94.99 % (26)), ^{34}S (4.25 % (24)) (**2**), and trace amounts of other sulfur isotopes. The presence of these other isotopes leads to several competing reactions that can interfere with the counting of the 1710-keV beta particle. This interference can usually be eliminated by the use of appropriate techniques, as discussed in Section 8.

6. Apparatus

6.1 Since only beta particles of ^{32}P are counted, proportional counters or scintillation detectors can be used. Because of the high resolving time associated with Geiger-Mueller counters, their use is not recommended. They can be used only with relatively low counting rates, and then only if reliable corrections for coincidence losses are applied.

6.2 Refer to Test Methods E181 for preparation of apparatus and counting procedures.

7. Materials and Manufacture

7.1 Commercially available sublimed flowers of sulfur are inexpensive and sufficiently pure for normal usage. Sulfur can be used directly as a powder or pressed into pellets. Sulfur pellets are normally made at least 3 mm thick in order to obtain maximum counting sensitivity independent of small variations in pellet mass. A 0.8 g/cm^2 pellet can be considered infinitely thick for the most energetic beta particle from ^{32}P (see Table 1). Due to the relatively long half-life of ^{32}P, it may not be practical to use a pellet more than once. A period of at least one year is recommended between uses. However, see 8.2 regarding long-lived interfering reaction products.

TABLE 1 Sulfur Counting Rate Versus Mass for a Pellet of 25.4-mm Diameter

Sample Mass, g	Relative Counting Rate
0.4	0.46
0.6	0.58
0.8	0.66
1.0	0.73
1.2	0.78
1.4	0.82
1.6	0.86
1.8	0.89
2.0	0.91
2.2	0.93
2.4	0.94
2.6	0.95
2.8	0.96
3.0	0.97
3.2	0.98
3.4	0.99
3.6	0.99
3.8	1.0
4.0	1.0

7.2 Where temperatures approaching the melting point of sulfur are encountered (113°C), sulfur-bearing compounds such as ammonium sulfate $(NH_4)_2SO_4$, lithium sulfate Li_2SO_4, or magnesium sulfate $MgSO_4$ can be used. These are suitable for temperatures up to 250, 850, and 1000°C, respectively. The reduced sensitivity of these compounds offers no disadvantage since high temperatures are usually associated with a high-neutron fluence rate. The sulfur content by weight of $(NH_4)_2SO_4$ is 24 %, of Li_2SO_4 is 29.2 %, and of $MgSO_4$ is 26.6 %.

7.3 The isotopic abundance of ^{32}S in natural sulfur is 94.99 ± 0.26 atom % (**2**,**3**).

8. Sample Preparation and Irradiation

8.1 Place sulfur in pellet or powdered form in a uniform fast-neutron flux for a predetermined period of time. Record the beginning and end of the irradiation period.

8.2 Table 2 lists competing reaction products that must be eliminated from the counting. Those resulting from thermal-neutron capture, that is, ^{33}P, ^{35}S, and ^{37}S, can be reduced by the irradiation of the sulfur inside 1 mm-thick cadmium shields. This should be done whenever possible in thermal-neutron environments. Those reaction products having relatively short half-lives, that is, ^{31}S, ^{34}P, ^{31}Si, and ^{37}S, can be eliminated by a waiting period before the counting is started. A delay of 24 h is sufficient for the longest lived of these, although shorter delays are possible depending on the degree of thermalization of the neutron field. Finally, those with relatively low beta particle energies, that is, ^{33}P and ^{35}S, can be eliminated by the inclusion of a 70-mg/cm^2 aluminum absorber in front of the detector. For particularly long decay times, an absorber must be used because the ^{35}S becomes dominant. Note that the use of an internal (windowless) detector maximizes the interference in counting from ^{35}S.

8.3 Irradiated sulfur can be counted directly, or may be burned to increase the efficiency of the counting system. Dilution may be used to reduce counting system efficiency for measurements of high neutron fluences.

8.4 Burning the sulfur leaves a residue of ^{32} P that can be counted without absorption of the beta particles in the sulfur pellet. Place the sulfur in an aluminum planchet on a hot plate until the sulfur melts and turns to a dark amber color. At this point the liquid gives off sulfur fumes. Ignite the fumes by bringing a flame close to the dish, and allow the sulfur to burn out completely. In order to reduce the sputtering that can lead to variations in the amount of ^{32}P remaining on the planchet, the hot plate must be only as hot as necessary to melt the sulfur. In addition, air flow to the burning sulfur must be controlled, such as by the placement of a chimney around the sulfur. Count the residue remaining on the dish for beta activity.

NOTE 1—The fumes given off by the burning sulfur are toxic. Burning should be done under a ventilating hood.

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[3] The non-boldface number in parentheses after the nuclear data indicates the uncertainty in the last significant digit of the preceding number. For example, 8.1 s (5) means 8.1 ± 0.5 seconds.

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

TABLE 2 Neutron-induced Reactions in Sulfur Giving Radioactive Products

Reaction	Cross Section		Cross Section (mb)			Product Halflife (1,2,3)	Maximum Energy of Product Beta (MeV) (1,4)	Average Energy of Product Beta (MeV) (1,4)	Isotopic Abundance of Target (%) (2)
	Library(5)	Material ID	Thermal^A	^{235}U Thermal Fission	Fast^B ^{252}Cf Fission				
1. $^{32}S(n,p)^{32}P$	RRDF-2008	1625	...	68.2	74.10	14.284 d (36)	1.71066 (21)	0.6955 (3)	94.99 (26)
2. $^{32}S(n,2n)^{31}S$	JENDL-4.0	1625	...	7.760×10^{-6}	2.5×10^{-5}	2.572 s (13)	5.3956 (β +)	1.9975 (β +)	94.99 (26)
3. $^{33}S(n,p)^{33}P$	JENDL-4.0	1628	2 ± 1	57.46	58.72	25.383 d (40)	0.2485 (11)	0.0764 (5)	0.75 (2)
4. $^{34}S(n,p)^{34}P$	JENDL-4.0	1631	...	0.8001	1.080	12.43 s (8)	5.374 (5)	2.30 (9)	4.25 (24)
5. $^{34}S(n,\alpha)^{31}Si$	JENDL-4.0	1631	...	3.281	4.067	157.3 m (3)	1.4905 (4)	0.595231	4.25 (24)
6. $^{34}S(n,\gamma)^{35}S$	JENDL-4.0	1631	256 ± 9	0.2753	0.2710	87.37 d (4)	0.16714 (8)	0.04863	4.25 (24)
7. $^{36}S(n,\gamma)^{37}S$	JENDL-4.0	1637	236 ± 6	0.2511	0.2508	5.05 m (2)	4.86530 (25)	0.800 (16)	0.01 (1)

[A] The thermal cross section corresponds to neutrons with a velocity of 2200 m/s or energy of 0.0253 eV.

[B] The fast cross section corresponds to the spectrum-averaged cross section from the ENDF/B-VI (MAT=9228, MF=5, MT=18) ^{235}U thermal fission spectrum (6,7) and the ENDF/B-VI (MAT=9861, MF=5, MT=18) ^{252} Cf spontaneous fission spectrum (6-8).

8.5 An alternative to burning is sublimation of the sulfur under a heat lamp. Removal of the sulfur is very gradual, and there is no loss of ^{32}P from sputtering.

8.6 Counting of dilute samples is useful for measuring high neutron fluences, although it is applicable to virtually all irradiation conditions. Use lithium sulfate, reagent grade or better, as the target material because of its high melting point (860°C), good solubility in water, and minimum production of undesirable activation products. Prepare a dry powder by spreading about 10 g of Li_2SO_4 in a weighing bottle and place in a drying oven for 24 h at 150°C. Place the dried Li_2SO_4 in a dessicator for cooling and storage. Prepare a phosphorus carrier solution by dissolving 21.3 g of $(NH_4)_2HPO_4$ in water to make 1 L of solution. Prepare a Li_2SO_4 sample for irradiation by placing about 150 mg of material in an air-tight aluminum capsule or other suitable container. Following the irradiation, accurately weigh a sample of about 100 mg and dissolve in 5 mL of phosphorus carrier solution to minimize adsorption of ^{32}P on the glass container. A drop of concentrated HCl may be used to speed solution of the sample. Place the solution in a volumetric flask and add additional phosphorus carrier solution to bring the total volume to 100 mL. Prepare a sample for counting by pipetting 0.050 mL of the ^{32}P solution onto a standard planchet and evaporating in air to dryness. Counting procedures and calculations are the same as in other methods with the exception that an aliquot factor of 2000 must be introduced for the 0.050-mL sample removed from the 100-mL flask.

9. Calibration

9.1 Calibration is achieved by irradiation of sulfur in a fast-neutron field of known spectrum and intensity, and measuring the resulting ^{32}P activity to determine a counting system's efficiency. This calibration is specific for a given detector system, counting geometry, and sulfur pellet size and mass or sample preparation. It is, however, valid for subsequent use in measuring activities in any arbitrary spectrum, and therefore, may be used with activation data from other foils in determining neutron energy spectra as described in Practice E721 and Practice E944.

9.2 ^{235}U fission and ^{252}Cf spontaneous fission neutron sources of known source strength have been used for direct free-field calibrations (9).

9.3 Once a sulfur counting system is calibrated, it must be monitored to ensure that the calibration remains valid. There are several isotopes that can be used as reference standards for this monitoring. One is ^{234}Pa, having a maximum beta energy of about 2000 keV, comparable to the 1710-keV beta from ^{32}P. It is obtained as a daughter of ^{238}U, that can be dispersed as a powder in plastic granules and formed to the shape of a standard pellet. The concentration of ^{238}U can be varied to obtain the desired counting rate. Uranium alpha particles can be prevented from reaching the detector by use of a 7-mg/cm ^{2} absorber. Another useful isotope is ^{210}Bi that produces beta particles having a maximum energy of 1161 keV. It is obtained as a daughter of ^{210}Pb, and sources are commercially available.

10. Activity and Fluence by Detector Efficiency Method

10.1 Using a sulfur sample irradiated in a calibration neutron field, determine the efficiency, ε, for the detector system:

$$\varepsilon = \frac{C\, f_\tau \exp[\lambda t_d]\, \lambda\, t_i}{N\, \bar{\sigma}_s\, \Phi\, (1-\exp[-\lambda t_c])\,(1-\exp[-\lambda t_i])} \tag{1}$$

where:

C = counts recorded in detector, less background,
f_τ = correction for coincidence losses, if needed,
λ = ^{32}P decay constant, = 5.625×10^{-7} s^{-1},
t_d = decay time, s,
t_c = count time, s,
t_i = duration of irradiation, s,
N = number of ^{32}S atoms in pellet,
$\bar{\sigma}_s$ = spectrum-averaged cross section for ^{32}S in the calibration neutron field, $cm^2 = 10^{24}$ b, and
Φ = neutron fluence, n/cm^2.

10.1.1 Fig. 1 shows a plot of sulfur cross section as a function of energy. Fig. 2 shows a plot of the uncertainty in the

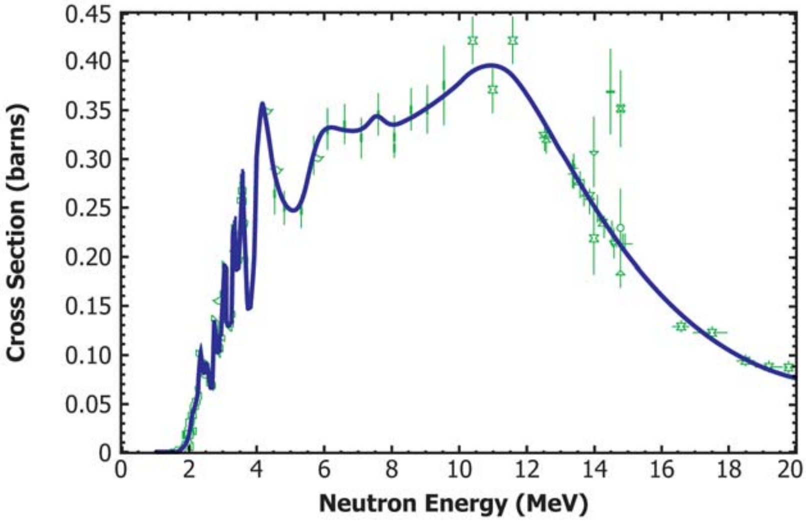

FIG. 1 ^{32}S(n,p)^{32}P Cross Section with EXFOR Experimental Data (3) Reaction

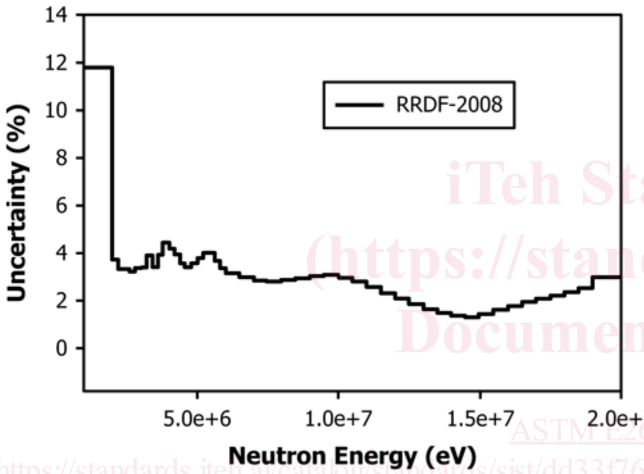

FIG. 2 Energy-dependence of Uncertainty (%) for the ^{32}S(n,p)^{32}P Cross Section

sulfur cross section as a function of energy. (See Guide E1018 for the recommended cross section source.) The spectrum-averaged cross section for ^{252}Cf fission neutrons is about 74.10 mb, and for ^{235}U fission is about 68.2 mb. (See Table 2 and Refs (**4**,**6**,**7**,**8**,**10**,**11**,**12**,**13**,**14**,**15**).)

10.1.2 The correction for coincidence losses, f_τ, is a function of the particular counting system, and may be already accounted for by the system electronics if "live time" is used (see Methods E181). Coincidence loss corrections can be large, especially when Geiger-Mueller counters are used.

NOTE 2—Because of β self-absorption in counting thick pellets intact, detection efficiency is not sensitive to small variations in pellet mass but is rather a function of the pellet dimensions. The detection efficiency should be determined for each different pellet size that is to be used. The value of *N* in Eq 1 can be taken to be the arithmetic mean over a number of pellets. Whether or not this is adequate depends on the uniformity of the pellets and the desired measurement uncertainties.

NOTE 3—When the calibration is performed using a point source such as ^{252}Cf, a correction should be made for fluence gradients and neutron scattering in the sulfur pellet. An estimate of the gradient effect is complicated by the fact that the detector is thick with respect to the range of the beta particles. If the calibration fluence is averaged over the front face of the pellet, the gradient correction is typically about [16/ *r*(cm)] % for a 2.54-cm diameter pellet, where *r* is the distance from the calibration source to the front face of the pellet. Effects of neutron scattering in the detector are typically a few tenths of a percent and can generally be assumed to be the same in both calibration and measurement fields (**12**).

10.2 Count the beta particles from the sulfur pellet or prepared sample in a calibrated detector system, and determine the ^{32}P activity:

$$A_O = \frac{Cf_\tau \exp[\lambda t_d]\,\lambda}{\varepsilon(1-\exp[-\lambda t_c])} \qquad (2)$$

where:

A_O = pellet activity adjusted to the end of the irradiation, and

ε = detector system efficiency previously determined as described in 10.1.

10.3 The saturation activity, A_s, as defined in Practice E261, is related to A_O by the following equation:

$$A_s = \frac{A_O}{(1-\exp[-\lambda t_i])} \qquad (3)$$

10.4 The reaction rate, R_s, is determined from the ^{32}P activity as follows:

$$R_s = A_s/N \qquad (4)$$

10.5 Determination of the neutron fluence requires knowledge of the spectrum average cross section for the specific neutron field. The neutron fluence is determined from the ^{32}P activity:

$$\Phi = \frac{A_O t_i}{N\bar{\sigma}_x\ (1-\exp[-\lambda t_i])} = \frac{A_s t_i}{N\bar{\sigma}_x} \qquad (5)$$

where:

$\bar{\sigma}_x$ = spectrum averaged cross section for ^{32}S(n,p) in the neutron field being measured, cm^2 = 10^{24} b.

or for a pulse rather than an extended irradiation:

$$\Phi = \frac{A_O}{N\bar{\sigma}_x\,\lambda} \qquad (6)$$