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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 E 265; 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 Department of Defense.

ε^1 NOTE—Ref 3 was editorially updated in April 2009.

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 E 261.

1.5 *This standard does not purport to address all of the safety problems, 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:*[2]

E 170 Terminology Relating to Radiation Measurements and Dosimetry

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides

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

E 720 Guide for Selection and Use of Neutron Sensors for Determining Neutron Spectra Employed in Radiation-Hardness Testing of Electronics

E 721 Guide for Determining Neutron Energy Spectra from Neutron Sensors for Radiation-Hardness Testing of Electronics

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

E 944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance, E 706 (IIA)

E 1018 Guide for Application of ASTM Evaluated Cross Section Data File, Matrix ~~E706(IIB)~~[2]E 706 (IIB)

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to Terminology E 170.

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 E 181 and the reaction rate, as defined in Practice E 261, 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 E 261.

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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 E10.07 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* ~~, Vol 12.02.~~volume information, refer to the standard's Document Summary page on the ASTM website.

5. Significance and Use

~~5.1Refer to Guide E844~~

5.1 Refer to Guides E 720 and E 844 for the selection, irradiation, and quality control of neutron dosimeters.

5.2 Refer to Practice E 261 for a general discussion of the determination of fast-neutron fluence and fluence rate with threshold detectors.

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 14.262 (14)[3] days **(1)** [4] and the maximum beta energy is 1710 keV **(2)**.

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 (95.02 % (9)), ^{34}S (4.21 % (8)) **(1)**, 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 Methods E 181 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.

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 95.02 ± 0.09 atom % **(1)** .

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.

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[3] The non-bolface number in parentheses after the nuclear data indicates the uncertainty in the last significant digit of the preceeding 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 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

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.

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

TABLE 2 Neutron-induced Reactions in Sulfur Giving Radioactive Products

Reaction	Cross Section		Cross Section (mb)			Product Halflife **(1)**	Maximum Energy of Product Beta (MeV) **(2)**	Average Energy of Product Beta (MeV) **(2)**	Isotopic Abundance of Target (%) **(1)**
	Library	Material ID	Thermal^A	^{235}U Thermal Fission	Fast^B ^{252}Cf Fission				
1. ^{32}S(n,p)^{32}P	GLUCS-93^C	1625	...	64.69	70.44	14.262 d (14)	1.7104	0.6949	95.02 (9)
2. ^{32}S(n,2n)^{31}S	JENDL-3^D	3161	...	7.742×10^{-6}	2.5×10^{-5}	2.572 s (13)	5.3956 (β +)	1.9975 (β +)	95.02 (9)
3. ^{33}S(n,p)^{33}P	JENDL-3^D	3162	1.6	57.46	58.77	25.34 d (12)	0.2485	0.0764	0.75 (1)
4. ^{34}S(n,P)^{34}P	JENDL-3^D	3163	...	0.8001	1.079	12.43 s (8)	5.3743	2.3108	4.21 (8)
5. ^{34}S(n,α)^{31}Si	JENDL-3^D	3163	...	3.281	4.064	157.3 m (3)	1.4908	0.59523	4.21 (8)
6. ^{34}S(n,γ)^{35}S	JENDL-3^D	3163	226	0.2749	0.2705	87.51 d (12)	0.16684	0.04863	4.21 (8)
7. ^{36}S(n,γ)^{37}S	JENDL-3^D	3164	151	0.2511	0.2509	5.05 m (2)	4.86516	0.800418	0.02 (1)

[A] The thermal cross section corresponds to neutrons with a velocity of 2200 m/s or ~~an~~ 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 **(5,6)** and the ENDF/B-VI (MAT=9861, MF=5, MT=18) ^{252} Cf spontaneous fission spectrum **(4-6)**.

[C] Cross section produced for the 1993 GLUCS library **(7)** and is similar to that in the IRDF-90 library **(8)**.

[D] The JENDL-3 **(9)** sulfur isotopes were adopted in the latest JEF 2.2 cross section **(10)** compilations. The ENDF/B-VI library **(5)** does not include the individual sulfur isotopic cross sections.