Designation: E 263 – 00

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

This standard is issued under the fixed designation E 263; 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.

1. Scope

1.1 This test method describes procedures for measuring reaction rates by the activation reaction ^{54}Fe (n,p) ^{54}Mn .

1.2 This activation reaction is useful for measuring neutrons with energies above approximately 2.2 MeV and for irradiation times up to about 3 years (for longer irradiations, see Practice E 261).

1.3 With suitable techniques, fission-neutron fluence rates above 10^8 $cm^{-2}\cdot s^{-1}$ can be determined. However, in the presence of a high thermal-neutron fluence rate (for example, $>2 \times 10^{14}$ $cm^{-2}\cdot s^{-1}$) ^{54}Mn 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:*

D 1193 Specification for Reagent Water[2]

E 170 Terminology Relating to Radiation Measurements and Dosimetry[3]

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides[3]

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques[3]

E 844 Guide for Sensor Set Design and Irradiation for Reactor Surveillance, E 706(IIC)[3]

E 944 Guide for Application of Neutron Spectrum Adjustment Methods in Reactor Surveillance, (IIA)[3]

E 1005 Test Method for Application and Analysis of Radiometric Monitors for Reactor Vessel Surveillance, E 706(IIIA)[3]

E 1018 Guide for Application of ASTM Evaluated Cross Section Data File, Matrix E 706(IIB)[3]

3. Terminology

3.1 *Definitions:*

3.1.1 Refer to ASTM Standard E 170 for definitions of terms relating to radiation measurements and neutron dosimetry.

4. Summary of Test Method

4.1 High-purity iron is irradiated in a neutron field, thereby producing radioactive ^{54}Mn from the ^{54}Fe (n,p) ^{54}Mn activation reaction.

4.2 The gamma rays emitted by the radioactive decay of ^{54}Mn are counted in accordance with Methods E 181 and the reaction rate, as defined by Practice E 261, is calculated from the decay rate and irradiation conditions.

4.3 Radioassay of the ^{54}Mn activity may be accomplished by directly counting the irradiated iron dosimeter, or by first chemically separating the ^{54}Mn activity prior to counting.

4.4 The neutron fluence rate above about 2.2 MeV can then be calculated from the spectral-weighted neutron activation cross section as defined by Practice E 261.

5. Significance and Use

5.1 Refer to Guide E 844 for guidance on 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 rate with threshold detectors.

5.3 Pure iron in the form of foil or wire is readily available and easily handled.

5.4 Fig. 1 shows a plot of cross section as a function of neutron energy for the fast-neutron reaction ^{54}Fe (n,p) ^{54}Mn . **(1)**[4] This figure is for illustrative purposes only to indicate the range of response of the ^{54}Fe (n,p) ^{54}Mn reaction. Refer to Guide E 1018 for descriptions of recommended tabulated dosimetry cross sections.

5.5 ^{54}Mn has a half-life of 312.12 days **(2)**[5] and emits a

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[1] This test method is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.05 on Nuclear Radiation Metrology.

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

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

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

[5] Reference **(3)** gives a value of 834.848 keV as the most precise value currently accepted for the ^{54}Mn decay gamma-ray energy.

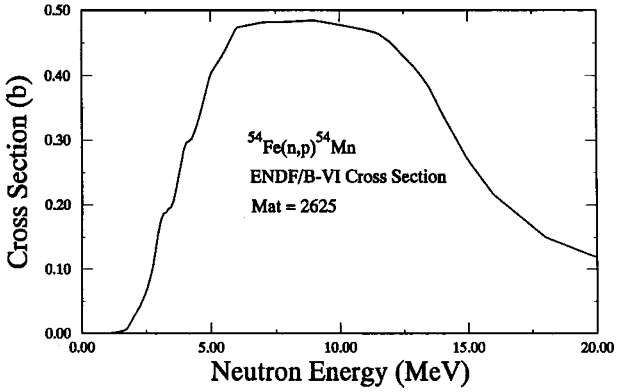

FIG. 1 ^{54}Fe (n,p) ^{54}Mn Cross Section

gamma ray with an energy of 834.8 eV. **(3)**[5]

5.6 Competing activities from thermal or fast neutrons are 2.58-h ^{56}Mn , 44.5-d ^{59}Fe , and 5.271-y ^{60}Co . (Consult reference 2 for more precise values currently accepted for the halflives.) Interference from ^{56}Mn can be eliminated by waiting 48 h before counting. Although chemical separation of ^{54}Mn from the irradiated iron is the most effective method for eliminating ^{59}Fe and ^{60}Co , direct counting of iron for ^{54}Mn is possible using high-resolution detector systems or unfolding or stripping techniques, especially if the dossimeter was covered with cadmium or boron during irradiation. Altering the isotopic composition of the iron dosimeter is another useful technique for eliminating interference from extraneous activities when direct sample counting is to be employed.

5.7 The vapor pressures of manganese and iron are such that manganese diffusion losses from iron can become significant at temperatures above about 700°C. Therefore, precautions must be taken to avoid the diffusion loss of ^{54}Mn from iron dosimeters at high temperature. Encapsulation in quartz, vanadium, or other materials will contain the manganese for temperatures up to about 900°C.

5.8 Sections 6, 7 & 8 that follow were specifically written to describe the method of chemical separation and subsequent counting of the ^{54}Mn activity. When one elects to count the iron dosimeters directly those portions of Sections 6, 7 & 8 that pertain to radiochemical separation should be disregarded.

NOTE 1—The following portions of this test method apply also to direct sample-counting methods: 6.1-6.3, 7.4, 7.9, 7.10, 8.1-8.5, 8.18, 8.19, and 9-12.

6. Apparatus (Note 1)

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.

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

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

6.4 *Cylinder*, borosilicate glass, about 25-mL capacity, equipped with stopcock and funnel. This apparatus is illustrated in Fig. 2.

6.5 *Beakers*, borosilicate glass, 50 mL; *volumetric flasks*, 25 and 50 mL, and *volumetric pipets*, 1 mL.

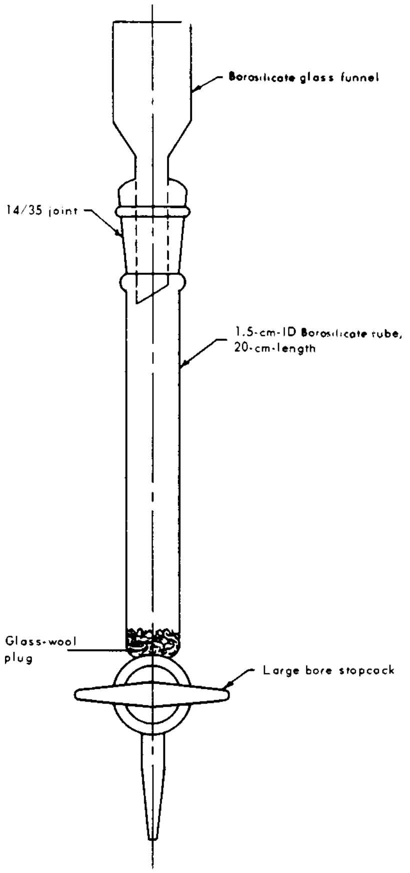

FIG. 2 Ion-Exchange Separation Apparatus

7. Reagents and Materials (Note 1)

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Commerce on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] 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.

7.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

7.3 *Anion Exchange Resin*, strongly basic type, 100 to 200 mesh size.

7.4 *Iron Foil or Wire*, high purity.

7.5 *Hydrochloric Acid (sp gr 1.19, 1190 kg/m^3)*—Concentrated hydrochloric acid (HCl).

7.6 *Hydrochloric Acid (1 + 3)*—Mix 1 volume of concentrated HCl (sp gr 1.19) with 3 volumes of water.

7.7 *Manganese Carrier Solution (10 mg MnCl$_2$/cm^3).*

7.8 *Nitric Acid (sp gr 1.42, 1420 kg/m^3)*—Concentrated nitric acid (HNO$_3$).

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[6] “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 “Analar Standards for Laboratory Chemicals,” BDH Ltd., Poole, Dorset, U.K., and the “United States Pharmacopeia.”

7.9 *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 capsule is to be opened remotely. (See Guide E 844.)

7.10 The purity of the iron is important in that no impurities should be present which produce long-lived radionuclides that interfere with the ^{54}Mn determination. This condition includes species that will accompany ^{54}Mn through the separation scheme and that have gamma rays, of energy 0.6 MeV or higher. The presence of impurities may be determined either by emission spectroscopy or by activation analysis.

8. Procedure (Note 1)

8.1 Decide on the size and shape of the iron sample to be irradiated. Consider convenience in handling and available irradiation space when making this selection, but it is more important to ensure that sufficient ^{54}Mn activity will be produced to permit accurate radioassay. A preliminary calculation of the expected production of ^{55}Mn , using the activation equation described in Section 9, will aid in selecting the mass of iron required.

8.2 Determine a suitable irradiation time.

8.3 Weigh the iron sample. The chemical manipulations described below function best with a wire weight of 10 to 20 mg.

NOTE 2—It is necessary to avoid a high iron concentration in the solutions that are to be used for separation so that the efficiency of the ion-exchange resin will not be seriously lowered. For the column described herein the amount of iron let onto the resin should not exceed 1 mg.

8.4 Irradiate the samples 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 the irradiation, and the relative position of the monitors in the irradiation facility.

8.5 A waiting period of 2 days is recommended between termination of the exposure and the start of counting. This allows 2.58-h ^{56}Mn , produced by fast-neutron reaction with ^{56}Fe and also by thermal-neutron activation of impurity manganese, to decay below levels at which it may cause error in the ^{54}Mn assay. Check the samples for activity from cross contamination by other monitors or material irradiated in the vicinity and for any foreign substance adhering to the sample. Clean, if necessary, and reweigh. If direct-counting techniques are used, disregard the remaining procedures to step 8.18.

8.6 After irradiation, dissolve the sample in 10 mL of concentrated hydrochloric acid to which 2 drops of nitric acid have been added. The solution may be heated gently to hasten dissolution.

8.7 After dissolution is complete, transfer the solution with washing to a 25-mL volumetric flask. Wash only with concentrated hydrochloric acid and use this also in diluting to the calibration mark on the volumetric flask.

8.8 Prepare a slurry of anion exchange resin with distilled or deionized water and pour it into the ion exchange column apparatus (see Fig. 2) to a height of 100 mm. Place a glass-wool plug above the resin and keep the column under liquid at all times.

8.9 Prepare the ion exchange column for use by passing concentrated hydrochloric acid through until it completely displaces the water used to form the resin slurry.

8.10 Transfer an aliquot of the sample solution by volumetric pipet to the empty funnel above the column. This aliquot should be of sufficient volume so that accurate counting data can be obtained.

8.11 Run the sample onto the column.

8.12 Immediately pour a few millilitres from a premeasured 50-mL volume of hydrochloric acid (1+3) into the funnel to wash any remaining sample solution onto the column.

8.13 Place a 50-mL volumetric flask, to which 1 mL of $MnCl_2$ carrier solution has been added, under the tip of the column and open the column stopcock.

8.14 Add the remaining hydrochloric acid (1+3) to the funnel and adjust the stopcock to obtain a flow rate of about 1 drop in 5 to 10 s. This will allow elution of a 50-mL volume in about 2 h.

8.15 Elute from the column until the solution reaches the calibration mark on the volumetric flask.

NOTE 3—To prepare the ion exchange resin for further separations, run about 50 mL of distilled or deionized water through the column. This will remove iron and cobalt from the resin. Regenerate the column as before by passing concentrated hydrochloric acid through until the acid completely displaces the water.

NOTE 4—The ^{54}Mn recovery should be checked by passing a known ^{54}Mn spike solution and iron carrier through the column.

8.16 Stopper the flask and invert several times to mix the contents thoroughly.

8.17 Remove an accurately measured aliquot from the volumetric flask for radioassay. A 1-mL sample is convenient if the counting is to be done with a well-type scintillation detector. If assay is to be made using a solid crystal, the aliquot can be deposited into a cup planchet and dried under a heat lamp.

8.18 Analyze the samples for ^{54}Mn content in disintegrations per second using the gamma ray spectrometer (see Methods E 181 and E 1005).

8.19 Disintegration of an ^{54}Mn nucleus produces one gamma ray with a probability per decay of 0.9998 **(4)**[7].

9. Calculation

9.1 Calculate the saturation activity A_s, as follows:

$$A_s = \frac{A \exp[\lambda t_w]}{(1 - \exp[-\lambda t_i])} \tag{1}$$

where:

A = ^{54}Mn disintegrations per second measured by counting, s^{-1},

λ = decay constant for ^{54}Mn $= 2.570 \times 10^{-8}$, s^{-1},

t_i = irradiation duration, s, and

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[7] Reference 4 gives a value of 0.99976 as the most precise value currently accepted for the emission probability of the 834.8-keV gamma-ray emitted from the nuclear decay of ^{54}Mn .

t_w = elapsed time between the end of irradiation and counting, s.

NOTE 5—The equation for A_s is valid if the reactor is operated at constant power and if corrections for other reactions (for example, impurities, burnout, etc.) are negligible. Refer to Practice E 261 for more generalized treatments.

9.2 Calculate the reaction rate, R_s, as follows:

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

where:

A_s = saturation activity, and

N_o = number of ^{54}Fe atoms.

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

10. Report

10.1 Practice E 261 describes how data should be reported.

11. Precision and Bias

11.1 General practice indicates that ^{54}Mn decay rate can be determined with a bias of ± 3 % (1σ) and with a precision of ± 1 % (1σ). Measurement of ^{54}Mn activity produced from the ^{54}Fe (n,p) ^{54}Mn reaction in a ^{235}U thermal fission standard neutron field can be accomplished with an uncertainty is 2.86 % **(5)**, where the uncertainty component attributed to knowledge of the cross section is 2.12 % **(6)**. Measurement of ^{54}Mn activity produced from the ^{54}Fe (n,p) ^{54}Mn reaction in a ^{252}Cf spontaneous fission standard neutron field can be accomplished with an uncertainty of 1.34 % **(7)**.

NOTE 6—Measurement uncertainty expressed in this standard is described by a statement of precision and bias. Another acceptable approach is to use Type A and Type B uncertainty components **(8,9)**. The Type A/B uncertainty specification is now used in the International Organization for Standardization (ISO) standards and this approach can be expected to play a more prominent role in future uncertainty analyses.

12. Keywords

12.1 fast neutron detector; iron activation; reaction rate; ^{54}Fe (n,p)

REFERENCES

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(**4**) “Nuclear Data Sheets for A=54,” W. Gongqing, Z. Jiabi, Z. Jingen, Nuclear Data Sheets, Vol 50 No. 2, p. 255, Academic Press, 1987.

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