



Designation: E 262 – 9703

Standard Test Method for Determining Thermal Neutron Reaction and Fluence Rates by Radioactivation Techniques¹

This standard is issued under the fixed designation E 262; 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 The purpose of this method is to define a general procedure for determining an unknown thermal neutron-fluence rate by neutron activation techniques. It is not practicable to describe completely a technique applicable to the large number of experimental situations that require the measurement of a thermal-neutron fluence rate. Therefore, this method is presented so that the user may adapt to his particular situation the fundamental procedures of the following techniques.

1.1.1 Absolute counting technique using pure cobalt, pure gold, or cobalt-aluminum or gold-aluminum alloy.

1.1.2 Standard foil technique using pure gold, or gold-aluminum alloy, and

1.1.3 Secondary standard foil techniques using pure indium, indium-aluminum alloy, and dysprosium-aluminum alloy.

1.2 The techniques presented are limited to measurements at room temperatures. However, special problems when making thermal-neutron fluence rate measurements in high-temperature environments are discussed in 8.2. For those circumstances where the use of cadmium as a thermal shield is undesirable because of potential spectrum perturbations or of temperatures above the melting point of cadmium, the method described in Test Method E 481 can be used in some cases. Alternatively, gadolinium filters may be used instead of cadmium. For high temperature applications in which aluminum alloys are unsuitable, other alloys such as cobalt-nickel or cobalt-vanadium have been used.

1.3 Table 1 indicates the useful neutron-fluence ranges for each detector material.

1.4 *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 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods²

¹ This 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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TABLE 1 Useful Neutron Fluence Ranges of Foil Material

Foil Material	Form	≈ Useful Range (neutrons/cm ²)
Indium	pure or alloyed with aluminum	10 ³ to 10 ¹²
Gold	pure or alloyed with aluminum	10 ⁷ to 10 ¹⁴
Dysprosium	pure or alloyed with aluminum	10 ³ to 10 ¹⁰
Cobalt	pure or alloyed with aluminum	10 ¹⁴ to 10 ²⁰

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides³

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques³

E 481 Test Method for Measuring Neutron Fluence Rate by Radioactivation of Cobalt and Silver³

3. Significance and Use

3.1 This method can be extended to use any material that has the necessary nuclear and activation properties that suit the experimenter's particular situation. No attempt has been made to fully describe the myriad problems of absolute counting techniques, neutron-fluence depression, and thick-foil self-shielding. It is assumed that the experimenter will refer to existing literature on these subjects. This method does offer a referee method (the standard gold foil irradiation at National Institute of Standards and Technology (NIST) to aid the experimenter when he is in doubt of his ability to measure an absolute thermal fluence rate.

3.2 The standard foil technique uses a set of foils that are as nearly identical as possible in shape and mass. The foils are fabricated from any material that activates by an (n, γ) reaction, preferably having a cross section approximately inversely proportional to neutron speed in the thermal energy range. Some of the foils are irradiated in a known neutron field (at NIST) or other standards laboratory). The foils are counted in a fixed geometry on a stable radiation-detecting instrument. The neutron induced reaction rate of the foils is computed from the counting data, and the ratio of the known neutron fluence rate to the computed reaction rate is determined. For any given foil, neutron energy spectrum, and counting set-up, this ratio is a constant. Other foils from the identical set can now be exposed to an unknown neutron field. The magnitude of the fluence rate in the unknown field can be obtained by comparing the reaction rates as determined from the counting data from the unknown and reference field, with proper corrections to account for spectral differences between the two fields (see Section 4). One important feature of this technique is that it eliminates the need for absolute counting.

4. Theory

4.1 *1/v Cross Sections*—It is not possible using radioactivation techniques to determine the true thermal neutron fluence rate; without making some assumptions about the spectral shapes of both the thermal and epithermal components of the neutron density. For most purposes, however, the information required is only that needed to make calculations of activation and other reaction rates for various materials exposed to the neutron field. For reactions in which the cross section varies inversely as the neutron speed ($1/v$ cross sections) the reaction rates are proportional to the total neutron density and do not depend on the spectrum shape. Many radioactivation detectors have reaction cross sections in the thermal energy range which approximate to $1/v$ cross sections ($1/v$ detectors). Departures from the $1/v$ shape can be accounted for by means of correction factors.

4.2 Fluence Conventions:

4.2.1 The purpose of a fluence convention (formerly called “flux convention”) is to describe a neutron field in terms of a few parameters that can be conveniently used to calculate reaction rates. The best known fluence conventions relating to thermal neutron fields are the Westcott convention **(1)**⁴ and the Stoughton and Halperin convention **(2)**. Both make use of the concept of an equivalent 2200 m/s fluence rate, that is equal to the product of the neutron density and the standard speed, v_0 , equal to 2200 m/s which is the most probable speed of Maxwellian thermal neutrons when the characteristic temperature is 293.4°K. In the Westcott convention, it is the total neutron density (thermal plus epithermal) which is multiplied by v_0 to form the “Westcott flux”, but in the Stoughton and Halperin convention, the conventional fluence rate is the product of the Maxwellian thermal neutron density and v_0 . The latter convention is the one followed in this method:

$$\phi_0 = n_{th} v_0 \quad (1)$$

where ϕ_0 is the equivalent 2200 m/s thermal fluence rate and n_{th} represents the thermal neutron density, which is proportional to the reaction rate per atom in a $1/v$ detector exposed to thermal neutrons:

² Annual Book of ASTM Standards, Vol 14.02.

³ Annual Book of ASTM Standards, Vol 12.02.

⁴ The boldface numbers in parentheses refer to the list of references appended to this method.

$$(R_s)_0 = n_{th}\sigma_0 v_0 = \sigma_0 \phi_0 \quad (2)$$

4.2.2 $(R_s)_0$ represents only that part of the reaction rate that is induced by thermal neutrons, which have the Maxwellian spectrum shape. σ_0 is the 2200 m/s cross section. For a non-1/v detector Eq 2 needs to be replaced by:

$$(R_s)_0 = n_{th}g\sigma_0 v_0 = g\sigma_0 \phi_0 \quad (3)$$

where g is a correction factor that accounts for the departures from the ideal 1/v detector cross section in the thermal energy range. The same factor appears in the Westcott convention Ref (1), and is usually referred to as the Westcott g factor. g depends on the neutron temperature, T , and is defined as follows:

$$g = \frac{1}{v_0\sigma_0} \int_0^\infty \frac{4}{\pi^{1/2}} \left(\frac{v}{v_0}\right)^3 \left(\frac{T_0}{T}\right)^{3/2} \cdot \exp\left[-\left(\frac{v}{v_0}\right)^2 \left(\frac{T_0}{T}\right)\right] \sigma(v) dv \quad (4)$$

4.2.3 If the thermal neutron spectrum truly follows the Maxwellian distribution and if the neutron temperature is known, it is possible to calculate the true thermal neutron fluence rate by multiplying the conventional (equivalent 2200 m/s) thermal fluence rate by the factor

$$\frac{\bar{v}}{v_0} = \left(\frac{4T}{\pi T_0}\right)^{1/2} \quad (5)$$

where \bar{v} is the Maxwellian mean speed for neutron temperature T , and T_0 is the standard temperature of 293.4°K. This conversion is most often unnecessary and is usually not made because the temperature T may be unknown. Naturally, it is essential when reporting results to be absolutely clear whether the true thermal fluence rate or the equivalent 2200 m/s thermal fluence rate or the equivalent 2200 m/s total (Westcott) fluence rate is used. If the true thermal fluence rate is used, then its value must be accompanied by the associated temperature value.

4.3 *Epithermal Neutrons*—In order to determine the effects of epithermal neutrons, that are invariably present together with thermal neutrons, cadmium covered foil irradiations are made. It is important to realize that some epithermal neutrons can have energies below the effective cadmium cut-off energy, E_{cd} . The lowest energy of epithermal neutrons is usually taken to be equal to $5kT$ (where k is Boltzmann's constant) which that is equal to 0.13eV for room temperature (293°K) neutrons (1), though 4 kT has been recommended for some reactors (3). In order to correct for these, it is necessary to make some assumption about the epithermal neutron spectrum shape, and the assumption made in Refs 1 and 2 is that the epithermal neutron fluence rate per unit energy is proportional to $1/E$:

$$\phi_e(E) = \phi_e/E, \quad E \geq 5kT \quad (6)$$

where ϕ_e is an epithermal fluence parameter equal to the fluence rate per unit energy, $\phi_e(E)$, at 1 eV. This assumption is usually adequate for the purpose of correcting thermal neutron fluence rate measurements for epithermal neutrons at energies below the cadmium cut-off. To represent the epithermal fluence more correctly, however, many authors have shown that the use of a $1/E^{(1+\alpha)}$ spectrum shape is preferable, where α is an empirical parameter. Refs (4-10).

4.4 Resonance Integral:

4.4.1 The resonance integral for an ideal dilute detector is defined as follows:

$$I_0 = \int_{E_{cd}}^\infty \sigma(E) \frac{dE}{E} \quad (7)$$

4.4.2 The cadmium cut-off energy is taken to be 0.55eV for a cylindrical cadmium box of wall thickness 1 mm. (11). The data needed to correct for epithermal neutron reactions in the methods described are the values of $I_0/g\sigma_0$ for each reaction (see Table 2). These values, taken from Refs (26-28), are based on integral measurements.

4.5 Reaction Rate:

4.5.1 The reaction rate per atom, for an isotope exposed to a mixed thermal and epithermal neutron field is given by:

$$R_s = \phi_0 g \sigma_0 + \phi_e g \sigma_0 [f_1 + w' / g + I_0 / g \sigma_0] \quad (8)$$

f_1 is a function that describes the epithermal activation of a 1/v detector in the energy range $5kT$ to E_{cd} :

$$f_1 = \int_{5kT}^{E_{cd}} \left(\frac{kT_0}{E}\right)^{1/2} \frac{dE}{E} \quad (9)$$

4.5.2 For E_{cd} equal to 0.55eV and T_0 equal to 293.4°K, $f_1 = 0.468$. w' in Eq 8 is a function which accounts for departure of the cross section from the 1/v law in the energy range $5kT$ to E_{cd} :

TABLE 2 Nuclear Data from References (23), (26-28)

Reaction	σ_0 barns	$g(T=293\text{ K})$	$\frac{I_0}{g\sigma_0}$	w'
$^{59}\text{Co}(n,\gamma)^{60}\text{Co}$	37.233 ± 0.16 %	1.0	1.98 ± .034	0
$^{197}\text{Au}(n,\gamma)^{198}\text{Au}$	98.69 ± 0.14 %	1.0051	15.7 ± 0.3	.0500
$^{115}\text{In}(n,\gamma)^{116}\text{In}$	166.413 ± 0.6 %	1.0194	15.8 ± 0.5	.2953
$^{163}\text{Dy}(n,\gamma)^{164}\text{Dy}$	2650 ± 3.8 %	0.975	0.23 ± 0.04	0

$$w' = \frac{1}{\sigma_0} \int_{5kT}^{E_{cd}} \left[\sigma(E) - g\sigma_0 \left(\frac{kT}{E} \right)^{1/2} \right] \frac{dE}{E} \quad (10)$$

Some values of w' for T equal 293.4°K are given in Table 2.

4.5.3 For a cadmium covered foil, the reaction rate is given as:

$$R_{s,Cd} = \phi_e J_0 \quad (11)$$

4.5.4 This can be used to eliminate the unknown epithermal fluence rate parameter, ϕ_e , from Eq 8. After rearrangement, one obtains an expression for the saturation activity due to thermal neutrons only:

$$\phi_0 g\sigma_0 = (R_s)_0 = R_s - R_{s,Cd} \left(1 + \frac{g\sigma_0}{I_0} f_1 + \frac{\sigma_0 w'}{I_0} \right) \quad (12)$$

4.6 *Neutron Self-Shielding:*

4.6.1 Unless extremely thin or dilute alloy materials are used, all of the measurement methods are subject to the effects of neutron self-shielding. The modified version of Eq 12 which takes into account both a thermal self-shielding factor G_{th} , and an epithermal self shielding factor G_{res} is:

$$\begin{aligned} \phi_0 g\sigma_0 &= \frac{(R_s)_0}{G_{th}} \\ &= \frac{1}{G_{th}} \left[R_s - R_{s,Cd} \left(1 + \frac{g\sigma_0}{G_{res} I_0} f_1 + \frac{\sigma_0 w'}{G_{res} I_0} \right) \right] \end{aligned} \quad (13)$$

4.6.2 Values of the self-shielding factors G_{th} and G_{res} for gold and cobalt foils and wires and for indium foils are given in Tables 3-7. In the literature, values for the resonance self-shielding factor are given in two ways, and those must not be confused. G_{res} used here, is a factor by which multiplies the resonance integral as defined in Eq 7. G'_{res} is a self-shielding factor that multiplies the reduced resonance integral from which the $1/v$ part of the cross section has been subtracted. The necessary conversion factor; that has been applied where needed in Tables 3-7 is:

$$G_{res} = G'_{res} + (1 - G'_{res}) 0.429 \frac{g\sigma_0}{I_0} \quad (14)$$

4.7 *Fluence Depression Factors* —Thermal fluence depression is an additional perturbation that occurs when an absorber is surrounded by a moderator. Because the effects are sensitive to the details of individual situations, it is not possible to provide correction factors here. References (12-20) describe these effects. The problem is avoided when foils are exposed in cavities of vary large volume compared to the detector volume. In other cases, a rough guide is that the external perturbation effect is usually less than the thermal self-shielding effect, and much less when the hydrogenous moderator is absent.

5. Apparatus

5.1 *Radiation-Detection Instruments :*

5.1.1 The radiation detectors that may be used in neutron activation techniques are described in the Standard Methods, E 181. In addition, or as an alternative, a calibration high-pressure ionization chamber may be used. Details for its construction and calibration may be found in Ref (21).

5.2 *Precision Punch:*

5.2.1 A precision punch is required to fabricate a set of identical foils for the standard foil technique. The punch must cut foils that have smooth edges. Since finding such a punch commercially available is difficult, it is recommended that the punch be custom made. It is possible to have several dies made to fit one punch so that a variety of foil sizes can be obtained. Normally, foil diameters are 12.7 mm (0.500 in.) or less. The precision punch is one of the most important items in the standard foil technique particularly if the counting technique includes β or soft-photon events.

5.3 *Aluminum and Cadmium Boxes :*

5.3.1 One set of foils must be irradiated in cadmium boxes or covers to determine that part of the neutron activation resulting from absorption of epithermal neutrons. The cadmium box must be constructed so that the entire foil is surrounded by 1 mm (0.040 in.) of cadmium. This can be accomplished by using a circular cup-shaped design as shown in Fig. 1. To eliminate

**TABLE 3 Resonance Self-Shielding Data for Cobalt Foils
(Reference (30))**

Foil Thickness		G'_{res} (132eV)	G_{res}
(in.)	(cm)		
0.0004	0.001018	0.8264	0.864
0.0010	0.02254	0.7000	0.765
0.0025	0.00635	0.5470	0.645
0.0050	0.0127	0.4395	0.561
0.0075	0.01905	0.3831	0.517
0.010	0.0254	0.3476	0.489
0.015	0.0381	0.3028	0.454
0.020	0.0508	0.2744	0.432

TABLE 4 Thermal and Resonance Self-Shielding Data for Cobalt Wires (Reference (31))

Wire diameter		Cobalt content (mass %)	$G'_{res}(132 \text{ eV})$	G_{th}	G_{res}
(in.)	(cm)				
0.050	0.127	0.104	1.00	1.00	1.00
0.050	0.127	0.976	0.95 ± 0.04	0.99 ± 0.01	0.96
0.001	0.00254	100	0.81 ± 0.03	0.99 ± 0.02	0.85
0.005	0.01270	100	0.52 ± 0.02	0.97 ± 0.01	0.62
0.010	0.0254	100	0.42 ± 0.02	0.94 ± 0.01	0.55
0.015	0.0381	100	0.38 ± 0.01	0.92 ± 0.02	0.51
0.020	0.0508	100	0.34 ± 0.01	0.90 ± 0.02	0.48
0.025	0.0635	100	0.32 ± 0.01	0.88 ± 0.03	0.47

**TABLE 5 Resonance Self-Shielding Data for Gold Foils
(References 32 and 33)**

Foil Thickness (cm)	l (barn)	G_{res} (theory)	G_{res} (experiment)	$(G_{theo}-G_{exp})/G_{exp}$ (%)
2×10^{-6}	1556.83	0.9936
4×10^{-6}	1550.04	0.9893
8×10^{-6}	1577.91	0.9815
2×10^{-5}	1507.41	0.9621	0.9644	-0.24
4×10^{-5}	1465.83	0.9355	0.9340	+0.16
8×10^{-5}	1398.77	0.8927	0.8852	+0.85
2×10^{-4}	1252.38	0.7993	0.7852	+1.80
4×10^{-4}	1088.91	0.6950	0.6836	+1.66
8×10^{-4}	890.482	0.5683	0.5612	+1.27
2×10^{-3}	628.570	0.4012	0.3952	+1.51
4×10^{-3}	468.493	0.2990	0.3020	-0.99
8×10^{-3}	347.671	0.2219	0.2219	-0.0036
2×10^{-2}	234.983	0.1450	0.1505	-0.35

**TABLE 6 Resonance Self-Shielding Data for Gold Wires
(Reference 34)**

Wire Diameter		Average (cm)	G_{res}
Nominal (10^{-3} in.)	Average (10^{-3} in.)		
0.5	0.505	0.00128	0.703
1.0	0.98	0.00249	0.552
2.0	1.98	0.00503	0.410
4.0	4.05	0.01029	0.302
6.0	6.02	0.01529	0.258
8.0	7.98	0.02027	0.228
10.0	10.01	0.02542	0.208

positioning errors, aluminum boxes identical to the cadmium boxes should be used for the “bare” or total neutron activation measurements. Small-bore cadmium tubing having 1 mm walls is commercially available for use with wires.

6. Materials and Manufacture

6.1 The four materials required for the techniques in this method are cobalt, gold, indium, and dysprosium. These metals are available commercially in very pure form (at least 99.9 %) and can be obtained in either foil or wire form. Cobalt, gold, indium, and dysprosium are also available as an alloy with aluminum, for example NIST Standard Reference Material 953. The alloy dilutions are useful for extending the range of measurement of higher neutron fluences; in the case of indium, the alloy has the additional advantage of mechanical strength. Pure indium is so soft that it must be handled with extreme care to prevent distortions in the precision punched foils. The use of alloys result in uncertainties and nonuniformity of alloy concentrations, but reduces the self-shielding corrections and their uncertainties.

7. Procedure

7.1 Cobalt Method:

7.1.1 Pure cobalt wire, 0.127 mm (0.005 in.) in diameter will conveniently monitor thermal neutron fluences in the range of 10^{14} to 10^{18} cm^{-2} . Cobalt-aluminum alloy wire of the same diameter (0.50 % by weight of cobalt or less) can be used for higher fluences. Burn-up of the target material needs to be considered at fluences above 10^{20} cm^{-2} . The neutron reaction involved is $^{59}\text{Co}(n, \gamma) ^{60}\text{Co}$. ^{60}Co emits two gamma rays per disintegration in cascade with energies of 1.17 and 1.33 MeV having a half-life of 1925.5 days (22). ^{60m}Co is also formed in the reaction, but this isomeric state decays to ^{60}Co by means of a single 0.059 MeV gamma ray having a half-life of 10.467 min (22).

TABLE 7 Self-Shielding Data for Indium Foils (Reference 33)

Natural indium foil thickness (mg/cm ²)	G_{res}	G_{th}	G_{res}/G_{th}
0.05	0.988	1.000	0.988
0.1	0.977	1.000	0.977
0.2	0.959	0.999	0.960
0.5	0.920	0.998	0.922
1.0	0.868	0.997	0.870
2.0	0.796	0.993	0.801
5.0	0.649	0.987	0.658
10	0.519	0.976	0.531
20	0.400	0.956	0.417
30	0.334	0.939	0.357
40	0.294	0.924	0.319
60	0.243	0.897	0.271
100	0.192	0.850	0.226
150	0.156	0.800	0.195
200	0.134	0.759	0.177
250	0.120	0.720	0.167

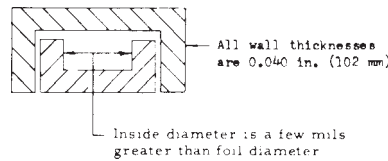


FIG. 1 Side View of Cadmium Box Cross Section

7.1.2 The equivalent 2200 m/s thermal fluence rate in which a thin sample of cobalt has been irradiated may be calculated as follows:

$$\phi_0 = \frac{R_s}{g\sigma_0} \tag{15}$$

where:

R_s = reaction rate per target atom,

σ_0 = 2200 m/s cross section.

7.1.3 The reaction rate is given by

$$R_s = \frac{C \exp(\lambda t_w)}{(\epsilon N_0 (1 - \exp(-\lambda t_i)))} \tag{16}$$

where:

C = net counting rate of ⁶⁰Co in the sample at the time of measurement, corrected for background radiations,

λ = decay constant of $4.170 \times 10^{-9} \text{ s}^{-1}$ corresponding to the half-life of ⁶⁰Co of 1925.5 days,

N_0 = original number of atoms of nuclide to be activated (given by the product of the weight in grams of ⁵⁹Co in the sample and Avogadro's number divided by the atomic weight, 58.9332, in g),

ϵ = efficiency of the detector for ⁶⁰Co radiation in the given geometry,

t_i = duration of the exposure, and

t_w = elapsed time from the end of the exposure period to the time of counting.

7.1.4 When the exposure time is small compared to the 1925.5-day half-life of ⁶⁰Co, as is usually the case, we may write

$$1 - \exp(-\lambda t_i) \approx \lambda t_i \tag{17}$$

Eq 15 becomes

$$\phi_0 = C \exp \lambda t_w / \lambda t_i N_0 \sigma_0 \epsilon \tag{18}$$

7.1.5 The fluence over the irradiation period is

$$\Phi = \phi_0 t_i = C \exp \lambda t_w / \lambda N_0 \sigma_0 \epsilon \tag{19}$$

7.1.6 If the cobalt sample has been activated in a neutron spectrum that is not totally thermalized, then the reaction rate must be corrected for epithermal neutron activation. This is done by irradiating a similar cobalt sample shielded by cadmium (1 mm (0.040-in.) thick) and using Eq 13 which yields,

$$\Phi = \frac{1}{G_{th}} \left(C_B - C_{cd} \left(1 + \frac{g\sigma_{01}}{G_{res}I_0} + \frac{\sigma_0 w'}{G_{res}I_0} \right) \right) \cdot \exp(\lambda t_w) / \lambda N_0 g \sigma_0 \epsilon \tag{20}$$

where C_B and C_{cd} are the ^{60}Co counting rates in the bare and cadmium-covered samples, respectively. In practice, the 0.127-mm cobalt wire cannot be considered a thin sample. The self-shielding effects of the wire are accounted for by the G_{th} and G_{res} factors in Eq 20 (see also Tables 4 and 5). If the cobalt-aluminum alloy (0.50 % by weight of cobalt or less) is being used, no self-shielding correction factors are needed.

7.1.7 There are two methods for obtaining the absolute disintegration rate of the ^{60}Co in the sample. The first method uses a high-pressure ionization chamber, a heavily shielded well-type counter that almost completely surrounds the sample being counted with an ionization volume, thereby allowing for essentially $4\text{-}\pi$ geometry to detect the radiation. A voltage placed across the collecting electrodes generates a current proportional to the number of ions produced, which in turn is proportional to the sample source strength. Measure the current, expressed as the voltage drop across precision resistors, with a potentiometer. Calibrate the chamber for ^{60}Co with a ^{60}Co gamma source having a certified activity which is traceable to a National Standard. A calibration constant S , expressed as disintegrations per second per volt, is thereby obtained. Accordingly, the disintegration rate of the cobalt wire sample is the product of S multiplied by the voltage reading obtained.

7.1.8 A second method for determining the disintegration rate in the cobalt sample as described in Method E 181, makes use of high resolution gamma detectors for interference-free counting of ^{60}Co . The detection efficiency for the ^{60}Co radiation must be measured using a certified ^{60}Co source, or a multi-gamma-ray reference standard which includes ^{60}Co , having a ^{60}Co activity that is traceable to a National Standard as described in Section 12.5 of Test Methods E 181. The shape, positioning, and encapsulation of both the standard source and the activation monitor sample must be carefully controlled to ensure the same detection efficiency in each case.

7.2 Absolute Counting Technique Using Gold:

7.2.1 Pure gold foil, 0.051 mm (0.002 in.) thick and 12.7 mm (0.5 in.) in diameter, or similar gold-aluminum alloy foils containing 0.5 % by weight of gold, can be used for neutron fluences in the range 10^7 to 10^{14} cm^{-2} . The reaction involved is $^{197}\text{Au}(n,\gamma)^{198}\text{Au}$. The product nucleus, which has a half-life of 2.6943 days (22) can be counted by means of a calibrated gamma spectrometer or by $4\pi\beta\text{-}\gamma$ coincidence counting. Other details are as described for the cobalt method.

7.3 Standard Foil Technique for Gold:

7.3.1 When a standard gold foil has been activated by neutrons, the following relationship is true:

$$\phi_0 = K(R_s)_0 \quad (21)$$

where K is a constant for any given size of foil, and counting set-up. The constant K includes the counter efficiency, the macroscopic nuclear properties of the foil material, and the geometry of the foil and detector. The object of the standard foil technique is to determine K for a particular experimental set-up.

7.3.2 Most neutron-fluence-rate measurements are made in a field of neutrons where all energies are present, not just thermal energies. Therefore, the experiment requires measuring the amount of activation on a standard foil due to epithermal neutrons. This is accomplished by irradiating the foil in a cadmium cover. For a thickness of 1 mm (0.040 in.) of cadmium, essentially all neutrons having energies less than about 0.5 eV will be absorbed by the cadmium. (A neutron having a velocity of 2200 m/s has an energy of 0.025 eV.)

7.3.3 Thus the general procedures for determining K include two types of standard foil irradiations. For one set of irradiations, the samples are bare or aluminum covered; the second set of irradiations are cadmium covered. After the counting data have been converted to saturation activities K can then be determined from the following relationship:

$$K = \frac{\phi_0}{(R_s)_0} \quad (22)$$

and

$$(R_s)_0 = R_s - R_{s,Cd} \left(1 + \frac{g\sigma_{g1}}{G_{res}I_0} + \frac{\sigma_0 w'}{G_{res}I_0} \right) \quad (23)$$

7.3.4 Gold is composed of only one isotope, ^{197}Au . When this isotope captures a neutron, it becomes radioactive ^{198}Au , emitting beta particles and gamma rays. The half-life for this decay is 2.6943 days. Thus, gold is a convenient material for standard foils, especially if the foils are exposed at one site and transported to another site for counting. The relatively long half-life also allows great latitude in exposure times, thus increasing the range of fluence rates over which a set of standard gold foils is useful. Gold foils that are 12.7 mm (0.500 in.) in diameter, and 0.051 mm (0.002 in.) thick can be used to measure neutron-fluence from 10^7 to 10^{14} cm^{-2} .

7.3.5 Make a set of standard gold foils by punching them on a precision punch from the stock material of pure gold sheet. Weigh the foils on an analytical balance, and select only those foils whose weights differ by less than 0.5 %. Then lightly scribe numbers on the foils with a sharp stylus. Afterwards, irradiate a minimum of six foils in a known thermal neutron field; three in aluminum covers and three in cadmium covers. ~~It is recommended that the~~ The facilities at the NIST may be used for the standard foil irradiations. Both thermal beams, from the thermal column of NIST reactor, and a nearly isotropic thermal field in a cavity in that thermal column are available. Calibrated neutron beams of up to 2.5-cm diameter and with fluence rates of 5×10^7 $\text{cm}^{-2}\cdot\text{s}^{-1}$ can be requested. Free-field neutron fluences in these beams are established relative to an absolute ^{235}U fission chamber with an estimated uncertainty of ± 1.5 % to ± 3 % (1σ) depending on the scattering in the experiment. Fluence rates of higher intensities, up to 10^{11} $\text{cm}^{-2}\text{ s}^{-1}$ at uncertainties approaching ± 2 % (1σ), are available in the thermal column cavity.

7.3.6 Upon receipt of the irradiated gold foils, count them on the counting set-ups that are to be calibrated; calculate the bare and cadmium-covered saturation activities; and determine the constant K as shown in Eq 22 and 23. An unknown thermal neutron fluence rate can now be measured by irradiating both bare and cadmium-covered gold foils from the standard set. A radioisotope having a long half-life (^{137}Cs or ^{60}Co) is recommended as a daily performance standard check for the counting set-ups to guarantee long-range stability.

7.3.7 For high fluence irradiations the second-order reaction $^{197}\text{Au}(n,\gamma)^{198}\text{Au}(n,\gamma)^{199}\text{Au}$ may be important. As discussed in E 261, the high 25,000 barn cross section of ^{198}Au leads to its removal by capture during irradiation: at a fluence rate of $10^{14}\text{ cm}^{-2}\text{ s}^{-1}$, the removal rate $A = \sigma\Phi$ is nearly twice the decay rate λ .

7.4 Secondary Standard Foil Technique for Indium:

7.4.1 Elemental indium is comprised of two stable isotopes, ^{113}In (4.29 %) and ^{115}In (95.71 %) (23). When ^{115}In captures a neutron, it becomes radioactive $^{116\text{m}}\text{In}$ and decays in a complex manner by beta emission. The half-life for this decay is 54.29 min (23). ^{113}In also activates upon neutron capture, but the resulting ^{114}In decays with a 49.51-day half-life (23). For the relatively short irradiation time necessary to activate ^{115}In , there is essentially no ^{114}In produced. Thus, indium is a convenient material for standard foils as the set can be reused on a daily basis if necessary due to the short half-life. Also, a few hours of irradiation time will essentially saturate the 54.4529-min activity. These properties of indium, coupled with its good thermal cross section, allow pure indium foils to measure a minimum neutron fluence rate of $1\text{ cm}^{-2}\cdot\text{s}^{-1}$. By diluting indium as an alloy of aluminum and also reducing the foil size to about 6.35 mm (0.250 in.) in diameter, it is possible to measure neutron fluence rates in the range of $10^{10}\text{ cm}^{-2}\cdot\text{s}^{-1}$. Indium foils must be calibrated directly in an on-site standard field; otherwise, the experimenter should use gold foils for the standard pile calibration, and then intercalibrate the set of indium foils in whatever neutron field is available at his own laboratory.

7.4.2 There is one additional correction, however, that must be made. Indium has a tremendous resonance in its neutron absorption cross section curve at about 1.44 eV. This resonance is sufficiently close to the cadmium cut-off energy ($\approx 0.5\text{ eV}$) that a correction must be made to all cadmium-covered indium measurements. The difficulty is that the cadmium not only absorbs the thermal neutrons effectively, but also begins to absorb many of the neutrons that should be captured by the lower wing of the tremendous indium resonance. Experimentally, this effect is seen as a rapid decrease in the cadmium-covered saturation activity of the indium foil as the thickness of cadmium increases. Thus, increase all cadmium-covered indium data by a correction factor greater than 1.00. Many experimenters have measured this correction, and there is good evidence that it is also a function of the size and thickness of the indium foil. It is recommended then, that each experimenter empirically determine this correction for his own particular foils by irradiating an indium foil in cadmium covers ranging in thickness from 0.25 to 1.00 mm (0.010 to 0.040 in.). For a thin or dilute foil in a 1 mm thick cadmium box the measured activity should be divided by the transmission factor of 0.93 reported in Ref (25).

7.5 Secondary Standard Foil Technique for Dysprosium:

7.5.1 Dysprosium is comprised of the following seven isotopes; (22):

Isotope Mass Number	Abundance, %
156	0.06
158	0.10
160	2.34
161	18.9
162	25.5
163	24.9
164	28.2

7.5.2 For many years the only pure form of this rare earth element commercially available was its oxide, Dy_2O_3 . In recent years, the pure metal has been processed and also an alloy with aluminum, containing approximately 5 % by weight of dysprosium is commercially available. The total absorption cross section for all the isotopes (about 940 barns) is due almost entirely to the last isotope, ^{164}Dy . Upon neutron capture, $(n, \gamma)^{164}\text{Dy}$ becomes $^{165\text{m}}\text{Dy}$ (half-life of 1.257 min) or ^{165}Dy (half-life of 2.334h, (23)). The isomeric state, $^{165\text{m}}\text{Dy}$ decays into ^{165}Dy . The activation cross sections for the two reactions are 1610 and 1040 barns, respectively, see Ref (24). Although the cross-section-versus-energy curve shows several large resonances in the eV-region, dysprosium has considerably less cadmium-covered activation than the more common neutron detector materials. It is this property, along with the large cross section and convenient half-life, that makes dysprosium an attractive choice for a thermal neutron detector. It is particularly useful in experiments where the physical space limitations will not allow the rather bulky cadmium-covered measurements to be made, such as inside the more tightly lattice plate-type or pin-type reactor fuel elements. The very low $I_0/g\sigma_0$ value for dysprosium means that the corrections for epithermal neutrons can be neglected. It also follows that self-shielding factors are not needed for relative thermal fluence rate measurements using dysprosium. Variation of the g factor with temperature must be accounted for when the neutron temperature in the unknown field differs from that in the standard field used.

7.5.3 As in the case of indium, the relatively short half-life of ^{165}Dy requires that the foils be counted at the same location where they were irradiated. Thus dysprosium-aluminum foils are standardized with previously calibrated standard gold foils. When the dysprosium-aluminum foils have been irradiated in a neutron spectrum containing a large fraction of high-energy neutrons (greater than 1 MeV), the experimenter must be aware of the 9.458-min ^{27}Mg and 0.62356-day ^{24}Na activities produced in the aluminum from the (n, p) and (n, α) reactions, respectively. If these activities are present, eliminate the ^{27}Mg by allowing the foils to decay for 1 to 2 h before counting, and determine the ^{24}Na contribution by recounting the foils after 24 to 36 h. Dysprosium foils are

usually beta counted since the total gamma activity is only about 1 % of the beta activity. Due to nonuniformity in the dysprosium-aluminum alloy, individually calibrate each foil, because the weight of the foil, even though precision punched, does not necessarily represent the relative dysprosium content. Make these calibrations conveniently by irradiating a set of dysprosium-aluminum foils on the rim (at equal radii) of a rotating disk in a thermal-neutron field. The observed saturation activities of the foils will be in the same ratio as their individual dysprosium content, since each foil will have received an identical exposure. A set of dysprosium-aluminum foils will remain calibrated over long periods of time owing to the alloy having good mechanical strength and not corroding or oxidizing under reasonable exposure conditions.

8. Discussion of Problems

8.1 Long-Term Fluence Monitoring :

8.1.1 As indicated in the cobalt method, low-concentration cobalt-aluminum-alloy wire is suitable for monitoring long-term thermal fluence. When the thermal-neutron fluence rate being monitored exceeds $10^{14} \text{ cm}^{-2}\cdot\text{s}^{-1}$ and the exposure times exceed a few weeks, the experimenter must be aware of possible burn-up of the target ^{59}Co nuclei and of the ^{60}Co being formed in the monitor, and burnout of the ^{113}Cd , if a cadmium sleeve is being used. A correction of the burnup of ^{59}Co and ^{60}Co can be made by solving the following equation (5) for ϕ :

$$A = N_0 \sigma_1 \phi \lambda [\exp - (\phi \sigma_1 t_i) - \exp - (\phi \sigma_2 + \lambda) t_i] / [\phi (\sigma_2 - \sigma_1) + \lambda] \exp \lambda t_w \quad (24)$$

where:

- A = measured ^{60}Co activity,
- N_0 = original number of ^{59}Co atoms (see 7.1.3),
- σ_1 = ^{59}Co cross section = 37.233 barns,
- σ_2 = ^{60}Co cross section = 2 barns,
- λ = ^{60}Co decay constant = 4.16647×10^{-9} ,
- t_i = exposure time, s, and
- t_w = elapsed time after the end of the exposure.

8.1.2 The method of dealing with long-term irradiations in which variations in fluence-rate occur is discussed in Method E 261.

8.2 High-Temperature Measurements :

8.2.1 As stated in the scope of this method, the methods being reported are essentially limited to room temperature environments. At higher temperatures, two basic problems arise. First, the most probable velocity for the Maxwell-Boltzman distribution of neutrons in thermal equilibrium with their moderator shifts upward from the 2200 m/s value for 20°C. In his interpretation of fluence rate, the experimenter must make the correct choice of cross sections, be aware of the departure of the cross sections from the 1/v-law, and remember that the effective cadmium cut-off energy is a function of temperature for a given thickness of cadmium. Second, pure cadmium has a relatively low melting point (321°C) and gives considerable trouble for temperatures above 100°C. It has been observed that above 100°C, cadmium metal tends to diffuse rapidly into metals in contact with it. Thus, standard gold or indium foils are immediately ruined when cadmium covered at these temperatures by receiving a readily observed layer of cadmium impregnated on their surfaces. Two possible compounds of cadmium for high-temperature experiments are cadmium oxide, and cadmium silicate. The melting points for these compounds are above 900°C. Equivalent cadmium metal thicknesses, stability under intense gamma and neutron bombardment, and fabrication properties would have to be determined before these compounds could be reliably used. Gadolinium filters, which have a lower cut-off energy than cadmium, have been successfully used (27). For high temperatures, Co-Ni or Co-V alloy detectors should be selected instead of the Co-Al alloy referred to in 8.1.1.

8.2.2 An alternate method for eliminating the use of cadmium at elevated temperatures is presented in Test Method E 481. The method uses one monitor (cobalt) with nearly a 1/v absorption cross-section curve and a second monitor (silver) with a large resonance peak so that its resonance integral is large compared to the thermal cross section. The method relies on the assumption that the epithermal part of the spectrum follows a 1/E distribution. In this method, the absolute activities of both cobalt and silver monitors are determined. This differs from the method described in 7.1 wherein only the one absolute activity of cobalt is determined. The advantages of Test Method E 481 are the elimination of three difficulties associated with the use of cadmium: (1) the perturbation of the neutron field by the cadmium, (2) the inexact cadmium cut-off energy, and (3) the low-melting temperature of cadmium. Studies indicate that the accuracy of the two-reaction method can be comparable to the cadmium-ratio method. Also, the long half-lives of the two monitors, cobalt and silver, permit the determination of fluence for long-term monitoring.

9. Precision and Bias

NOTE 1—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 (36, 37). 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.

9.1 Absolute Counting Using Cobalt :

9.1.1 The estimated systematic uncertainties in the determination of thermal-neutron fluence rate by the 0.127-mm (0.005-in.) cobalt wire method are listed below.

Source of Uncertainty	Uncertainty, %
⁶⁰ Co half-life (1925.5 days)	0.03
⁵⁹ Co cross section (37.233 barns)	0.16
NIST-calibrated ⁶⁰ Co source	+1.2 ^A
Self-shielding factor	±1.0
Gamma-ray detector calibration	±1.0 ^B

^AThe value may vary from one batch of NIST-calibrated ⁶⁰Co sources to another. The total uncertainty given on the NIST certificate should be used, if different from this value.

^BThis uncertainty is in addition to the contribution, calculated above, to the total systematic uncertainty from the uncertainty in the ⁶⁰Co half-life, and may vary from one detector to another.

The square root of the sum of the squares of the preceding errors yields a total estimated systematic uncertainty, that is, approximately ±1.9 %.

When cobalt-aluminum alloy is used there is no self shielding factor uncertainty, but instead there is an uncertainty in alloy composition.

9.2 Gold Indium and Dysprosium Foils :

9.2.1 *Standard Neutron Field*—The uncertainty in the calibrated neutron fluence rate in a standard field will vary depending upon which standard neutron field is used. In general, these uncertainties are in the range of from 5 to 10 %. However, the NIST estimates an uncertainty of only ±1.5 to ±3 % (1 σ).

9.2.2 *Counting Uncertainties*—The counting rates from foils exposed in a standard pile are usually quite small, thus, counting statistics are poor. However, in the case of gold foils, the long half-life allows long counting times; and with care, the counting uncertainties can be kept to 2 % or less.

9.2.3 *Experimental Uncertainty*—Determine the experimental uncertainties that occur while using the calibrated foils to measure an unknown neutron fluence rate by observing the precision in repeating the experiment. Normal statistical methods of calculating the standard deviation will yield the best estimate of these uncertainties. Under normal experimental conditions, these uncertainties are usually 2 % or less as defined in Recommended Practice E 177.

9.2.4 *Fluence Perturbation Uncertainties* —Uncertainties resulting from fluence perturbations due to the foils measuring the fluence rate are usually quite small. Even though the actual perturbations are large, it is assumed that the foil causes the same perturbation in the thermal neutron component of the standard field as it does in the unknown field. This feature is another major advantage of the standard foil technique. However, in extreme cases where the moderator materials are vastly different or if the neutron spectra to be measured is greatly different from the standard pile, uncertainties will arise unless the fluence perturbation corrections are carefully applied.

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