



Designation: E 262 – 03

# Standard Test Method for Determining Thermal Neutron Reaction and Fluence Rates by Radioactivation Techniques<sup>1</sup>

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## 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.*

<sup>1</sup> 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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## 2. Referenced Documents

### 2.1 ASTM Standards:

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>2</sup>

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides<sup>3</sup>

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques<sup>3</sup>

E 481 Test Method for Measuring Neutron Fluence Rate by Radioactivation of Cobalt and Silver<sup>3</sup>

## 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 ( $n, \gamma$ ) 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

<sup>2</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>3</sup> Annual Book of ASTM Standards, Vol 12.02.

**TABLE 1 Useful Neutron Fluence Ranges of Foil Material**

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

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)**<sup>4</sup> 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  $\phi_0$  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:

$$(R_s)_0 = n_{th}\sigma_0v_0 = \sigma_0\phi_0 \quad (2)$$

**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.  $\sigma_0$  is the 2200 m/s cross section. For a non-1/v detector Eq 2 needs to be replaced by:

$$(R_s)_0 = n_{th}g\sigma_0v_0 = g\sigma_0\phi_0 \quad (3)$$

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) 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  $\phi_e$  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  $\alpha$  is an empirical parameter. Refs **(4-10)**.

##### 4.4 Resonance Integral:

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

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  $E_{cd}$ :

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

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:

$$R_{s,Cd} = \phi_e I_0 \quad (11)$$

4.5.4 This can be used to eliminate the unknown epithermal fluence rate parameter,  $\phi_e$ , 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:

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

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

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

Reaction	$\sigma_0$ barns	$g$ ( $T = 293$ 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

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  $\beta$  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

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

Foil Thickness		$G'_{res}$ (132 eV)	$G_{res}$
(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	$0.95 \pm 0.04$	$0.99 \pm 0.01$	0.96
0.001	0.00254	100	$0.81 \pm 0.03$	$0.99 \pm 0.02$	0.85
0.005	0.01270	100	$0.52 \pm 0.02$	$0.97 \pm 0.01$	0.62
0.010	0.0254	100	$0.42 \pm 0.02$	$0.94 \pm 0.01$	0.55
0.015	0.0381	100	$0.38 \pm 0.01$	$0.92 \pm 0.02$	0.51
0.020	0.0508	100	$0.34 \pm 0.01$	$0.90 \pm 0.02$	0.48
0.025	0.0635	100	$0.32 \pm 0.01$	$0.88 \pm 0.03$	0.47

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

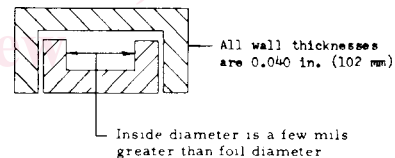
Foil Thickness (cm)	$\lambda$ (barn)	$G_{res}$ (theory)	$G_{res}$ (experiment)	$(G_{theo}-G_{exp})/G_{exp}$ (%)
$2 \times 10^{-6}$	1556.83	0.9936	...	...
$4 \times 10^{-6}$	1550.04	0.9893	...	...
$8 \times 10^{-6}$	1577.91	0.9815	...	...
$2 \times 10^{-5}$	1507.41	0.9621	0.9644	-0.24
$4 \times 10^{-5}$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

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

Natural indium foil thickness (mg/cm <sup>2</sup> )	$G_{res}$	$G_{th}$	$G_{res}/G_{th}$
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

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

Wire Diameter		Average (cm)	$G_{res}$
Nominal (10 <sup>-3</sup> in.)	Average (10 <sup>-3</sup> 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


**FIG. 1 Side View of Cadmium Box Cross Section**

in Fig. 1. To eliminate 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<sup>-2</sup>. 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<sup>-2</sup>. The neutron reaction involved is  $^{59}\text{Co}(n, \gamma)^{60}\text{Co}$ .  $^{60}\text{Co}$  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).  $^{60m}\text{Co}$  is also formed in the reaction, but this isomeric state decays to  $^{60}\text{Co}$  by means of a single 0.059 MeV gamma ray having a half-life of 10.467 min (22).

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: