



Standard Test Method for Determination of Effective Boron-10 Areal Density in Aluminum Neutron Absorbers using Neutron Attenuation Measurements¹

This standard is issued under the fixed designation E2971; 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 This test method is intended for quantitative determination of effective boron-10 (^{10}B) areal density (mass per area of ^{10}B , usually measured in grams- $^{10}\text{B}/\text{cm}^2$) in aluminum neutron absorbers. The attenuation of a thermal neutron beam transmitted through an aluminum neutron absorber is compared to attenuation values for calibration standards allowing determination of the effective ^{10}B areal density. This test is typically performed in a laboratory setting. This method is valid only under the following conditions:

1.1.1 The absorber contains ^{10}B in an aluminum or aluminum alloy matrix.

1.1.2 The primary neutron absorber is ^{10}B .

1.1.3 The test specimen has uniform thickness.

1.1.4 The test specimen has a testing surface area at least twice that of the thermal neutron beam's surface cross-sectional area.

1.1.5 The calibration standards of uniform composition span the range of areal densities being measured.

1.1.6 The areal density is between 0.001 and 0.080 grams of ^{10}B per cm^2 .

1.1.7 The thermalized neutron beam is derived from a fission reactor, sub-critical assembly, accelerator or neutron generator.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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.*

¹ This test method is under the jurisdiction of ASTM Committee E07 on Nondestructive Testing and is the direct responsibility of Subcommittee E07.05 on Radiology (Neutron) Method.

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2. Referenced Documents

2.1 *ASTM Standards*²

C1671 Practice for Qualification and Acceptance of Boron Based Metallic Neutron Absorbers for Nuclear Criticality Control for Dry Cask Storage Systems and Transportation Packaging

E1316 Terminology for Nondestructive Examinations

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E1316.

4. Summary of Test Method

4.1 In this test method, aluminum neutron absorbers are placed in a thermal neutron beam and the number of neutrons transmitted through the material in a known period of time is counted. The neutron count can be converted to ^{10}B areal density by performing the same test on a series of appropriate calibration standards and comparing the results.

4.2 This test method uses a beam of neutrons with the neutron energy spectrum thermalized by an appropriate moderator. Other methods such as neutron diffraction may be used to generate a thermal neutron beam.

4.3 A beam of thermal neutrons shall be derived from a fission reactor, sub-critical assembly, accelerator or neutron generator.

5. Significance and Use

5.1 The typical use of this test method is determination of ^{10}B areal density in aluminum neutron absorber materials used to control criticality in systems such as: spent nuclear fuel dry storage canisters, transfer/transport nuclear fuel containers, spent nuclear fuel pools, and fresh nuclear fuel transport containers.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard

5.2 Areal density measurements are also used in the investigation of the uniformity in ^{10}B spatial distribution.

5.3 The expected users of this standard include designers, suppliers, neutron absorber users, testing labs, and consultants in the field of nuclear criticality analysis.

5.4 Another known method used to determine areal density of ^{10}B in aluminum neutron absorbers is an analytical chemical method as mentioned in Practice C1671. However, the analytical chemical method does not measure the “effective” ^{10}B areal density as measured by neutron attenuation.

6. Interferences

6.1 Counts not associated with attenuation by the sample shall be accounted for by measuring and incorporating background readings. Background reading will vary depending on the set up of the electronics of the system and the presence/absence of high energy photons.

6.2 Measured count rates approaching the background count rate may limit the abilities of a system to accurately measure highly attenuating samples.

6.3 Coincidence loss may occur in the ^{10}B detector(s) when the neutron count rate is too high.

7. Apparatus

7.1 The essential features required for areal density measurement are the following:

7.1.1 Source of thermal neutrons of an appropriate intensity to obtain the desired counting statistics in a reasonable time period while not saturating the detector. If the counting rate is too high, pulses can pile up, causing counts to be lost. The detector time constant in most modern counting circuits is sufficiently small to accommodate up to 2×10^6 CPM. However, checks should be made to ensure that the system resolving time is not excessive.

7.1.2 A neutron beam intensity monitor for correction of neutron intensity fluctuations.

7.1.3 A collimator long enough to result in a thermal neutron beam with a minimal beam divergence that will reduce scattering contributions and ^{10}B measurement variability with sample thickness. The collimator may be evacuated, filled with air, or an inert gas.

7.1.4 A physical support, preferably adjustable, to mount the standard and the test specimens in the neutron beam.

7.1.5 A neutron detector, usually a boron tri-fluoride (BF_3) filled detector tube. In BF_3 detectors, the pulse amplitudes from neutrons are much larger than the pulses produced by gamma radiation. The pulse height discriminator is normally readily able to bias out the gamma pulses.

7.1.6 Electronic circuitry to count the number of neutrons detected by the neutron detector(s). The electronics generally consist of a pre-amplifier, amplifier, pulse-height discriminator, counting circuits and an appropriate timer

7.1.7 A thermal neutron beam with a cross-sectional area between 0.75 cm^2 and 6.0 cm^2 . The diameter of the beam should not exceed the active area of the neutron detector.

8. Hazards

8.1 This test method does not address radiation safety. It is the responsibility of the user of this test method to establish appropriate safety procedures, if necessary.

9. Calibration and Standardization

9.1 A series of standards with uniform, homogenous, and accurately known ^{10}B areal densities is necessary for quantitative interpretation of the counting data acquired in the attenuation measurements. If the standards are not chemically homogenous, the user of this standard must demonstrate that the uniformity of the sample's ^{10}B is sufficient to meet the intention of this standard. These standards shall include ^{10}B areal densities spanning the range of areal densities expected in the test specimens. Calibration standards must have a testing surface area at least twice that of the thermal neutron beam's cross-sectional area

9.2 The number of standards used shall take into consideration the magnitude and range of the sample's target areal density and required accuracy of the measurement. A minimum of three standards shall be used. The facility, calibration standards, and the test samples' areal densities should be considered when determining the spacing of the calibration areal densities. For example, when using a poly-energetic beam, the optimal spacing of the calibration standard's areal densities will not be uniform.

9.3 Aluminum shim plate(s) may be required with the standards to simulate the aluminum in the test specimen. Because the absorption and scattering cross-sections of aluminum are very small, exact replication of the aluminum in the test specimens is not critical. Scattering plays a very minor role in neutron attenuation measurements. The standards shall be shimmed to ensure an equivalent or larger scattering contribution than the test specimen.

9.4 If the material used for calibration standards contains neutron absorbing or scattering nuclides not present in the test specimens, or vice versa, the effect of these nuclides on the accuracy of the measurements shall be addressed.

10. Procedure

10.1 The following procedure describes the method used to measure the calibration standards as well as the samples. Calibration, background, and beam intensity shall be measured each time a set of samples are undergoing investigation, so the measurement of these values is also described as part of the procedure. This particular approach measures all values as counts per measurement period.

10.2 Prepare the neutron source for use. Verify that calibration standards and test specimens are available and ready for use.

10.3 Measure the counting rate for the direct beam (db) with any holders in place.

10.4 Measure the background counting rate (bkg) with a strong absorber at the sample position sufficient to attenuate the neutrons responsible for the measurement.

10.5 Position a calibration standard at the exposure location ensuring that its thinnest dimension is perpendicular to the beam line and the beam will not extend past any edges of the calibration standard.

10.6 Use the apparatus to establish the count rate through the calibration standard ensuring an exposure of sufficient duration to obtain a minimum number of counts. The minimum number of counts shall be established to ensure an acceptable level of uncertainty in calculated ^{10}B areal densities.

10.7 Repeat steps 10.5 and 10.6 with all other selected calibration standards.

10.8 Record the values obtained from the measured calibration standards.

10.9 Position a sample at the exposure location ensuring that the thinnest dimension of the sample is perpendicular to the beam line and the beam will not extend past any edges of the sample.

10.10 Use the apparatus to establish the count rate through the sample ensuring an exposure of sufficient duration to obtain a minimum number of counts. The minimum number of counts shall be established to ensure an acceptable level of uncertainty in calculated ^{10}B areal densities.

11. Calculation or Interpretation of Results

11.1 The effective ^{10}B areal density of a sample is determined from the measurements detailed in the procedure in Section 10. After correcting the measured counts of the sample and calibration standards, the effective ^{10}B areal density is determined by mathematical or graphical methods (on the basis of the logarithmic attenuation of neutrons) to establish the effective ^{10}B areal density of the samples from the known ^{10}B areal densities of the calibration standards.

11.2 Count Rate

11.2.1 The raw count rate for each data point must be corrected for fluctuations in neutron intensity and corrected for background radiation detections. The corrected count rate is calculated by:

$$C_c(i) = \frac{\frac{C_{raw}(i)}{t_{raw}(i)} \times \frac{C_{power}(db)}{t_{power}(db)}}{\frac{C_{power}(i)}{t_{power}(i)}}} - \frac{\frac{C_{raw}(bkg)}{t_{raw}(bkg)} \times \frac{C_{power}(db)}{t_{power}(db)}}{\frac{C_{power}(bkg)}{t_{power}(bkg)}}} \quad (1)$$

where:

i	= a sample or calibration standard reference identifier
$C_c(i)$	= corrected counts per second for the test part i
$C_{raw}(i)$	= raw counts from the test part i
$t_{raw}(i)$	= count time from the test part i
$C_{power}(i)$	= power counts from the test part i
$t_{power}(i)$	= power count time from the test part i
$C_{raw}(bkg)$	= raw counts from the background calibration
$t_{raw}(bkg)$	= count time from the background calibration
$C_{power}(bkg)$	= power counts from the background calibration
$t_{power}(bkg)$	= power count time from the background calibration
$C_{power}(db)$	= power counts from the direct beam
$t_{power}(db)$	= power count time from the direct beam

NOTE 1—Eq 1 normalizes the count rates with the power counts from the direct beam measurement. Normalizing with any consistent calibration power count is valid.

11.3 B_{10} Areal Density Determination

11.3.1 The ^{10}B areal density is determined based on interpolation from the calibration standard and test samples' corrected count rates. This interpolation needs to take into account the exponential attenuation of neutrons. The mathematical method to determine a test sample's areal density, as described below, uses the two calibration standards that bound the test sample's count rate. This is intended to reduce bias from beam hardening (a gradual increase in the energy spectrum of the neutron beam as it passes through the absorber in broad energy spectrum beams) and the associated change in neutron attenuation that results from this change in the neutron energy spectrum. Alternative mathematical or graphical interpolation methods using two or more calibration points may also be acceptable provided they have been properly validated.

11.3.2 Interpolating between two calibration standards, a sample's ^{10}B content can be determined as follows:

$$N_{AD}(i) = \left[\frac{\ln \frac{C_c(\text{calib high})}{C_c(i)}}{\ln \frac{C_c(\text{calib high})}{C_c(\text{calib low})}} \right] \times (N_{AD(\text{low})} - N_{AD(\text{high})}) + N_{AD(\text{high})} \quad (2)$$

where,

$C_c(\text{calib high})$	= corrected counts per second for the calibration part with ^{10}B areal density greater than $C_c(i)$
$C_c(\text{calib low})$	= corrected counts per second for the calibration part with ^{10}B areal density less than $C_c(i)$
$N_{AD(i)}$	= nominal areal density of test part i
$N_{AD(\text{high})}$	= nominal areal density of calibration part chosen as $C_c(\text{calib high})$
$N_{AD(\text{low})}$	= nominal areal density of calibration part chosen as $C_c(\text{calib low})$

12. Report

12.1 Report the following information:

12.1.1 The ^{10}B areal density calculated with the associated uncertainty,

12.1.2 The number and ^{10}B areal density of the calibration standards used,

12.1.3 The testing facility and apparatus, and

12.1.4 The calculation method used.

13. Precision and Bias

13.1 *Precision*—The repeatability standard deviation from a single operator has been determined to be 0.00012 g/cm² (0.4 %) and the 95 % repeatability limit is 0.00034 g/cm² (1.2 %). These values are representative of the repeatability; variations in setup, detailed measurement procedure and sample composition may affect the repeatability. The reproducibility of this test method is not provided at this time because only a single laboratory provides testing to Test Method E2971 at time of publication. The reproducibility of this test method

will be determined in the future if additional laboratories provide testing to Test Method E2971.

NOTE 2—Data for repeatability determination was collected from a single laboratory. Three samples of the same material type were measured 27 times over a period spanning 33 months. The sample with the largest relative standard deviation was utilized to prepare the repeatability statement. The effective boron-10 areal density of this sample was measured to be on average 0.02862 g/cm² with the range of 0.00042 g/cm² across all of the measurements for this sample. The two other samples had measured effective boron-10 areal density of 0.02011 ± 0.00007 g/cm² and 0.02364 ± 0.00009 g/cm² (±1σ).

13.2 The precision is influenced by the counting uncertainty and the uncertainty in the known areal density of the calibration standards.

13.3 Care should be exercised to assure that no other strong attenuators are present in the test specimen or reference standards. Strongly attenuating impurities in the test specimen may be interpreted as ¹⁰B and distort the ¹⁰B areal density.

13.4 Beam hardening of the thermal neutron beam can result in somewhat non-exponential attenuation of neutrons. To

reduce bias, reduced spacing between the areal densities in calibration standards for poly energetic neutron beams may be required.

13.5 As ¹⁰B areal densities approach the limits of a facility's measurement capabilities, the impact of detector saturation, collimation, counting background, and thermalization of the neutrons may become prohibitive without system modifications or additional corrections in the analysis.

13.6 The neutron energy spectrum may vary over time with non-diffraction derived neutron beams. Periodic re-measurement of calibration standards can correct for gradual changes in the neutron energy spectrum while increased neutron moderation will reduce this bias.

13.7 *Bias*—No bias was observed in the measured data collected for determining repeatability.

14. Keywords

14.1 areal density; boron; criticality control; neutron absorber; neutron attenuation test; poison material

SUMMARY OF CHANGES

Committee E07 has identified the location of selected changes to this standard since the last issue (E2971-14) that may impact the use of this standard.

(1) Wording changes with removal of the use of “coincidence loss” in Section 7 as several types of loss at high counting rates can occur, coincidence loss not necessarily being the most significant.

(2) Updated precision statement in Section 13 to reflect the results of a recent interlaboratory study.

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