



# Standard Practice for Qualification and Acceptance of Boron Based Metallic Neutron Absorbers for Nuclear Criticality Control for Dry Cask Storage Systems and Transportation Packaging<sup>1</sup>

This standard is issued under the fixed designation C 1671; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice provides procedures for qualification and acceptance of neutron absorber materials used to provide criticality control by absorbing thermal neutrons in systems designed for nuclear fuel storage, transportation, or both.

1.2 This practice is limited to neutron absorber materials consisting of metal alloys, metal matrix composites (MMCs), and cermets, clad or unclad, containing the neutron absorber boron-10 ( $^{10}\text{B}$ ).

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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

**B 557** Test Methods for Tension Testing Wrought and Cast Aluminum- and Magnesium-Alloy Products

**B 557M** Test Methods for Tension Testing Wrought and Cast Aluminum- and Magnesium-Alloy Products [Metric]

**C 791** Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Boron Carbide

**E 8** Test Methods for Tension Testing of Metallic Materials

**E 21** Test Methods for Elevated Temperature Tension Tests of Metallic Materials

**E 456** Terminology Relating to Quality and Statistics

**E 1225** Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique

**E 1461** Test Method for Thermal Diffusivity by the Flash Method

## 3. Terminology

### 3.1 Definitions:

3.1.1 *acceptance test, n*—for a neutron absorber material, quality control, tests, and inspections conducted to determine whether a specific production lot meets selected specified material properties, characteristics, or both, so that the lot can be accepted.

3.1.2 *areal density, n*—for neutron absorber materials with flat parallel surfaces, the density of the neutron absorber times the thickness of the material ( $\text{g}/\text{cm}^2$ ).

3.1.3 *durability, n*—the ability of neutron absorber materials to withstand service conditions without physical changes that would render them unable to perform their design functions.

3.1.4 *lot, n*—a quantity of a product or material accumulated under conditions that are considered uniform for sampling purposes. **E 456**

3.1.5 *moderator, n*—a material used to reduce neutron energy by scattering without appreciable capture.

3.1.6 *neutron absorber, n*—a nuclide that has a large thermal neutron absorption cross section (also known as a neutron poison).

3.1.7 *neutron-absorber material, n*—a compound, alloy, composite or other material that contains a neutron absorber.

3.1.8 *neutron attenuation test, n*—for neutron absorber materials, a process in which a material is placed in a thermal neutron beam, and the number of neutrons transmitted through the material in a specified period of time is counted. The neutron count can be converted to areal density by performing the same test on a series of appropriate calibration standards and comparing the results.

3.1.9 *neutron cross section, [barn], n*—a measure of the probability that a neutron will interact with a nucleus in the absorbing medium and is a function of the neutron energy.

3.1.10 *open porosity, n*—the volume fraction of all pores, voids, and channels within a solid mass that are interconnected with each other and communicate with the external surface,

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.03 on Neutron Absorber Materials Specifications.

Current edition approved July 15, 2007. Published August 2007.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

and thus are measurable by gas or liquid penetration. **C 242, C 21**

3.1.11 *packaging, n*—in transport of radioactive material, the assembly of components necessary to enclose the radioactive contents completely.<sup>3</sup>

3.1.12 *probability sampling, n*—a sample selection procedure in which the sampling units are selected by a chance process such that, at each step of the selection, a specified probability of selection can be attached to each sampling unit available for selection. **E 456**

3.1.13 *qualification, n*—for neutron absorber materials, the process of evaluating, testing, or both, a material produced by a specific manufacturing process to demonstrate uniformity and durability for a specific application.

3.1.14 *systematic sampling, n*—a sample selection procedure in which every  $k$ th element is selected from the universe or population, for example,  $u, u + k, u + 2k, u + 3k$ , etc., where  $u$  is in the interval 1 to  $k$ . **E 456**

### 3.2 Definition of Term Specific to This Standard:

3.2.1 *designer, n*—the organization responsible for the design or the license holder for the dry cask storage system or transport packaging. The designer is usually the purchaser of the neutron absorber material, either directly or indirectly (through a fabrication subcontractor).

## 4. Significance and Use

4.1 For criticality control of nuclear fuel in dry storage and transportation, the most commonly used neutron absorber materials are borated stainless steel alloys, borated aluminum alloys, and boron carbide aluminum alloy composites. The boron used in these neutron absorber materials may be natural or enriched in the nuclide  $^{10}\text{B}$ . The boron is usually incorporated either as an intermetallic phase (for example,  $\text{AlB}_2$ ,  $\text{TiB}_2$ ,  $\text{CrB}_2$ , etc.) in an aluminum alloy or stainless steel, or as a stable chemical compound particulate such as boron carbide ( $\text{B}_4\text{C}$ ), typically in an aluminum MMC or cermet.

4.2 While other neutron absorbers continue to be investigated,  $^{10}\text{B}$  has been most widely used in these applications, and it is the only thermal neutron absorber addressed in this standard.

4.3 In service, many neutron absorber materials are inaccessible and not amenable to a surveillance program. These neutron absorber materials are often expected to perform over an extended period.

4.4 Qualification and acceptance procedures demonstrate that the neutron absorber material has the necessary characteristics to perform its design functions during the service lifetime.

4.5 The criticality control function of neutron absorber materials in dry cask storage systems and transportation packagings is only significant in the presence of a moderator, such as during loading of fuel under water, or water ingress resulting from hypothetical accident conditions.

4.6 The expected users of this standard include designers, neutron absorber material suppliers and purchasers, govern-

ment agencies, consultants and utility owners. Adherence to this standard does not guarantee regulatory approval; a government regulatory authority may require different tests or additional tests, and may impose limits or restrictions on the use of a neutron absorber material.

## 5. Procedure

5.1 *Determination of Service Conditions and Design Requirements for the Neutron Absorber Material*—The designer shall specify the service conditions and design requirements, including environmental conditions, mechanical properties and areal density or equivalent measure of neutron absorber content. Selection of environmental and service conditions that are important for neutron absorber material performance and qualification should take into consideration known failure modes and industry experience.

5.1.1 Environmental conditions to be considered include but are not limited to water chemistry, water temperature, paired dissimilar materials, hydrostatic pressure, duration of immersion, gamma and fast neutron flux, heat-up rate after draining, and maximum temperature.

5.1.2 For structural applications, specify the mechanical properties required by the structural analysis. For non-structural uses of the neutron absorber material, specify mechanical properties sufficient to assure material durability under the service conditions for which it is designed.

5.1.3 Specify other design properties, for example, thermal conductivity, surface finish, etc., as required for the application.

5.1.4 Product or feed material chemistry shall be specified.

5.2 *Neutron Absorber Material Qualification*—Qualification shall consist of three components: (1) verify durability for the intended service as defined in 5.2.5, (2) verify that the physical characteristics of components meet their design requirements defined in 5.2.6, and (3) verify that the uniformity of the  $^{10}\text{B}$  distribution in the neutron absorber material is within acceptable bounds as specified by the designer as described in 5.2.6.

5.2.1 Qualification is needed:

5.2.1.1 When the neutron absorber material has not been previously qualified,

5.2.1.2 When a new supplier is producing a qualified neutron absorber material, or

5.2.1.3 When any key process or process control, as defined in 5.2.7, is altered for production of a qualified neutron absorber material.

5.2.2 The key processes and process controls for producing neutron absorber material for qualification should be the same as those to be used for commercial production. Differences shall be justified per 5.2.7.

5.2.3 Re-qualification for a qualified neutron absorber material produced by a new supplier may consist of review of key processes and process controls to verify that they have been adequately replicated by the new supplier.

5.2.4 If a neutron absorber material can not be qualified completely by reference to prior testing with similar neutron absorber materials for similar design functions and service conditions, complete the qualification by performing testing or portions thereof as described in 5.2.5 and 5.2.6.

<sup>3</sup> "Regulations for the Safe Transport of Radioactive Material," Safety Series Standards No. TS-R-1, International Atomic Energy Agency, Vienna, Austria.

5.2.5 *Environmental Qualification Tests*—For these tests, verify by visual and dimensional inspection, mechanical testing, neutron attenuation testing, etc., as appropriate, that the neutron absorber material does not undergo physical changes that would render it unable to perform its design functions.

5.2.5.1 For radiation and thermal testing, expose the neutron absorber material to the service conditions or equivalent accelerated conditions.

5.2.5.2 Corrosion testing shall consist of exposing test specimens of the neutron absorber material to simulate in-service immersion conditions.

5.2.5.3 If the neutron absorber material has open porosity, test it under simulated loading and service conditions using bounding pressure, temperature, time, and vacuum.

5.2.6 *Mechanical, Absorber Uniformity, and Other Qualification Testing*:

5.2.6.1 Perform tensile tests according to Test Method **B 557**, **B 557M**, **E 8**, or **E 21**. Perform any other mechanical testing, for example, fracture toughness testing, bend testing, etc., in accordance with the appropriate ASTM test method. For neutron absorber materials where standardized testing is not appropriate, such as for laminates, develop the mechanical test appropriate for the materials.

NOTE 1—Most neutron absorbers are non-structural and are held in place during service by structural components. If the absorber material is intended as a structural member, other tests may be necessary to conform to a structural code (for example, ASME Boiler and Pressure Vessel Codes). It may also be necessary to consider the long term service temperature and the effect of aging on the tensile strength of aluminum alloy-based absorber materials.

5.2.6.2 Assess the uniformity of the neutron absorber distribution in the neutron absorber material by measuring either: (1) the neutron absorber density ( $\text{g}/\text{cm}^3$ ) and thickness, or (2) the areal density ( $\text{g}/\text{cm}^2$ ) by either: (1) probability sampling or (2) systematic sampling throughout the test material, provided that the systematic sampling method is conservative. Determine the lower tolerance limit of the measurements as described in item #3 below. If uniformity testing for areal density will be by means other than neutron attenuation, the user of the proposed method shall confirm that the proposed method is acceptable to the designer.

(1) Neutron attenuation is a convenient method of determining the  $^{10}\text{B}$  content of the neutron absorber material and may be used to analyze the  $^{10}\text{B}$  distribution. For neutron attenuation testing, compare thermal neutron attenuation through the product with attenuation through calibration standards. If materials with discrete absorber particles or phases are used for the calibration standards, then the size of the particles containing the neutron absorber shall be small enough so that neutron streaming and self-shielding is insignificant.<sup>4</sup> The attenuation tests on the calibration standards and on the product shall use the same test equipment and configuration.

(2) Neutron attenuation measurements shall be performed in accordance with written procedures that address, as a minimum, the following:

- (a) The calibration standards used and their validation,
- (b) The frequency of calibration as required to account for neutron beam intensity variation, or source decay,
- (c) Neutron source and beam collimation, if any,
- (d) Method of interpolation between calibration points, and
- (e) Neutron detection instrumentation.
- (f) If the material used for calibration standards contains neutron absorbing or scattering nuclides not present in the neutron absorber material to be examined, the procedure shall address the effect of these nuclides on the accuracy of the measurements.

(3) Using a goodness of fit test, determine if the set of measurement data is normally distributed. For a normal distribution, calculate the lower tolerance limit,  $T$ , for the areal density or neutron absorber density for each lot, that is, a number  $T$  such that at least a proportion,  $P$ , of the lot is greater than  $T$  with confidence  $\gamma$ . Usually,  $P \geq 95\%$  and  $\gamma \geq 95\%$ . Calculate  $T$  as follows:

$$T = x(\text{bar}) - K \cdot s \quad (1)$$

where:

- $T$  = the lower tolerance limit,  $\text{g}/\text{cm}^2$  for areal density or  $\text{g}/\text{cm}^3$  for neutron absorber density,
- $K$  = the one-sided tolerance limit factor for a normal distribution with probability  $P$  and confidence  $\gamma$ ,<sup>5</sup> dimensionless,
- $x(\text{bar})$  = the sample average,  $\text{g}/\text{cm}^2$  or  $\text{g}/\text{cm}^3$ , and
- $s$  = the sample standard deviation,  $\text{g}/\text{cm}^2$  or  $\text{g}/\text{cm}^3$ .

NOTE 2—If the data set is not normally distributed, then a nonparametric lower tolerance limit may be used. In this case, the method must be documented.

NOTE 3—The user is advised to consider the effect of the statistical uncertainty in neutron counting, which may contribute to the standard deviation of the measurements and thus affect the lower tolerance limit. In neutron attenuation testing, the fractional statistical uncertainty of counting is equal to the square root of the number of counts divided by the number of counts.

5.2.6.3 Verify other properties as required by the design, for example, thermal conductivity (Test Method **E 1225** or **E 1461**), chemical analysis, surface finish, mechanical properties, etc.

5.2.7 *Key Processes and Process Controls*—Identify key processes and process controls so that the product delivered for use will be consistent with the qualification test material in all respects that are important to the design function of the neutron absorber material. Prior to a change in any key process or process control, the supplier shall submit the proposed change and evaluation of its potential effects on the material properties to the designer who shall determine if any retesting is required. Re-testing need only evaluate those characteristics likely to be adversely affected by the change. The following paragraphs provide examples of key processes and process controls.

5.2.7.1 A change in a key process or process control that could adversely affect mechanical properties, for example, increasing the boron content over that qualified by testing, reducing powder metal compact density, etc., require re-qualification by applicable testing (5.2.5 and 5.2.6, as appropriate).

<sup>4</sup> Turner, S. E., "Reactivity Effects of Streaming Between Discrete Boron Carbide Particles in Neutron Absorber Panels for Storage or Transport of Spent Nuclear Fuel," *Nuclear Science and Engineering*, Vol 151, Nov. 2005, pp. 344-347.

<sup>5</sup> Natrella, M. G., "Experimental Statistics," *National Bureau of Standards Handbook 91*, National Bureau of Standards, Washington, DC, 1963.

5.2.7.2 A change in equipment, mixing variables, or precipitate phase that could adversely affect the  $^{10}\text{B}$  distribution may require repetition of the qualification testing for  $^{10}\text{B}$  uniformity (5.2.6.2).

### 5.3 Neutron Absorber Material Acceptance Testing:

5.3.1 Establish the lot definition, statistical sampling plan, coupon locations (if applicable), and test locations. Acceptance tests shall be performed on each lot of manufactured product.

5.3.2 Test locations shall be representative of the finished product with regard to the characteristic to be tested, or they shall be selected such that they are conservative, that is, they are more likely to fail the test for that characteristic. Tests may be performed directly on the final product or on coupons removed contiguous to the final product. The tests should be performed at locations that are selected by probability sampling or systematic sampling throughout the lot.

5.3.3 Establish the consequences of acceptance testing failure, for example, sheet or lot rejection, in terms of re-measurement and disposition of non-conforming product.

5.3.4 *Acceptance Tests for Neutron Absorber Content and Uniformity*—Acceptance testing for  $^{10}\text{B}$  density or areal density is generally performed on small samples that are representative of a much larger area of sheet or plate, a large number of pellets, etc. The acceptance testing must include some validation that the same measurement, if performed on 100 % of the material, would satisfy the specified  $^{10}\text{B}$  density or areal density minimum with a specified degree of probability and confidence, usually at 95 % and 95 % or better. This can be achieved in several ways: (1) Determine the lower tolerance limit of measured  $^{10}\text{B}$  density or areal density as described in item #3 of 5.2.6.2, and compare the lower tolerance limit to the specified minimum as the basis for lot acceptance as described in 5.3.4.1 through 5.3.4.5. (2) Measure the density using a test method or a sample location that will yield a  $^{10}\text{B}$  density or areal density that is lower than the value anywhere else in the product. In this case, statistical analysis of the acceptance test results is not needed, but the material qualification must validate that the test method and sample location will yield a  $^{10}\text{B}$  density or areal density that is lower than the value anywhere else in the product.

5.3.4.1 Measure the amount of absorber in the neutron absorber material. Two common methods are: (1) chemical

analysis supplemented by isotopic analysis of the fraction of  $^{10}\text{B}$  in the boron (Test Method C 791), and (2) neutron attenuation testing. Neutron attenuation testing is primarily applicable to sheet or plate materials that are characterized by areal density, and does not readily lend itself to other material forms such as pellets. Neutron attenuation testing shall be performed in accordance with 5.2.6.2.

5.3.4.2 Areal density may be divided by the material thickness at the location of the areal density measurement to calculate  $^{10}\text{B}$  density.

5.3.4.3 Coupons which do not meet dimensional, visual or other specified physical criteria may be removed from the sample before being tested for  $^{10}\text{B}$  density or areal density.

5.3.4.4 Determine the lower tolerance limit in  $^{10}\text{B}$  density or areal density for all remaining sampled coupons as described in item #3 of 5.2.6.2.

5.3.4.5 Any lot whose lower tolerance limit or lower bound for  $^{10}\text{B}$  density or areal density is below the specified minimum shall be treated as non-conforming in accordance with 5.3.3.

5.3.5 *Quality Inspections for Other Characteristics*—Verify other product characteristics as required by the designer.

### 5.4 Quality Assurance:

5.4.1 The supplier shall maintain a quality assurance system to control manufacturing in accordance with the key process controls, as described in 5.2.7.

5.4.2 As a condition of material acceptance, the supplier shall permit the designer to review manufacturing records to verify conformance to the key process controls.

## 6. Report

6.1 Prepare a material qualification report that addresses all of the items in 5.2.5 and 5.2.6.

6.2 Verify that the neutron absorber material qualification report satisfies the design requirements of the dry cask storage system or transportation packaging.

6.3 The results of the acceptance testing will be documented in an acceptance test report.

## 7. Keywords

7.1 acceptance testing; dry cask storage; isotopic analysis; neutron absorber; neutron attenuation; neutron poison; qualification testing; transportation packaging

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