



Designation: E3063 – 24

Standard Test Method for Antimony Content Using Neutron Activation Analysis (NAA)¹

This standard is issued under the fixed designation E3063; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method covers the measurement of antimony concentration in plastics or other hydrocarbon or organic matrix by using neutron activation analysis (NAA). The sample is activated by irradiation with neutrons from a research reactor and the subsequently emitted gamma-rays are detected with a germanium semiconductor detector. The same system may be used to determine antimony concentrations ranging from 1 ng/g to 10 000 $\mu\text{g/g}$ with the lower end of the range limited by numerous interferences and the upper limit established by the demonstrated practical application of NAA.

1.2 This test method may be used on either solid or liquid samples, provided that they can be made to conform in size and shape during irradiation and counting to a standard sample of known antimony content using very simple sample preparation. Several variants of this method have been described in the technical literature. A monograph is available which provides a comprehensive description of the principles of neutron activation analysis using reactor neutrons (1).²

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

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautions are given in Section 9.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This test 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 The boldface numbers in parentheses refer to a list of references at the end of this standard.

2. Referenced Documents

2.1 ASTM Standards:³

E170 Terminology Relating to Radiation Measurements and Dosimetry

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 U.S. Government Document:⁴

Code of Federal Regulations, Title 10, Part 20

2.3 Joint Committee for Guides in Metrology (JCGM) Reports:⁵

JCGM 100:2008, GUM 1995, with minor corrections Evaluation of measurement data—Guide to the expression of uncertainty in measurement

3. Terminology

3.1 Definitions: See also Terminology E170.

3.1.1 *comparator standard*—a reference standard of known antimony content whose specific activation and counting sensitivity (counts (mg of antimony)^{−1}) may be used to quantify the antimony content of a sample irradiated and counted under the same conditions. Often, a comparator standard is selected to have a matrix composition, physical size, density, and shape very similar to the corresponding parameters of the sample to be analyzed. Differences in size, density, shape, and matrix composition between sample and standard may be corrected for using physical or empirical models.

3.1.2 *gamma-ray spectrometer*—a system comprising a detector which detects individual gamma-rays and converts their energy into an electronic pulse whose voltage is proportional to the energy deposited in the detector, and a multichannel

³ 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.

⁴ Available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁵ Document produced by Working Groups of the Joint Committee for Guides in Metrology (JCGM). Available free of charge at BIPM website (<http://www.bipm.org>).

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

pulse-height analyzer which measures the pulse heights, assigns a digital value, and stores the individual counts in the channels of a gamma-ray spectrum according to the digital values assigned.

3.1.3 *intensity*—the probability of emission of a gamma-ray of a given energy per decay. Another commonly used term is gamma abundance.

3.1.4 *monitor*—any type of detector or comparison reference material that can be used to produce a response proportional to the neutron fluence rate in the irradiation position, or to the radionuclide decay events recorded by the sample detector.

3.1.4.1 *Discussion*—An aluminum wire with 1 mg/g Au content is often used as a fluence rate monitor. Iron wires are used as well. It is important to distinguish that the monitor is not a standard used to scale the antimony content of the samples to be measured, but rather is used to normalize the analysis system among samples irradiated simultaneously at different positions in the polyethylene irradiation sample container or among successive analytical passes within the procedure. When using reactors with highly reproducible fluence rate, such as those with 1 % variation over long periods of time, monitors may not be necessary for every irradiation.

3.1.5 *neutron fluence rate*—the fluence rate (see definition in Terminology E170) of neutrons. In this test method it refers to the value at the site in the reactor where sample and comparator standard are irradiated.

3.1.6 *pneumatic transfer system*—a system used to transport the sample to the irradiation site in the reactor and then to a sample receiver.

3.1.6.1 *Discussion*—It may also be used to transport the sample directly to the counting station where the activity of the sample is measured. For the measurement of antimony, where a long decay time between irradiation and counting is usually required, the samples are manually transferred from the receiver to the germanium semiconductor detector or to a mechanical sample changer which transports them one by one at the appropriate time to the counting position at the detector.

3.1.7 *research nuclear reactor, n*—a nuclear reactor that uses the fission of uranium to operate at a well-controlled power level and produces neutrons that can be used for experiments and for neutron activation analysis. The operational characteristics of reactor types believed to be applicable to this test method are given in Refs (2-6).

3.1.7.1 *Discussion*—Another term in common usage is research reactor. Reactor conditions which may make the reactor unsuitable for this test method (for example, very low neutron fluence rates or high operating temperature) are sample and reactor dependent. Such conditions should be considered prior to use of this test method.

3.1.8 *standard uncertainty*—measurement uncertainty of the results of a measurement expressed as a standard deviation (GUM, see 2.3).

4. Summary of Test Method

4.1 The test method can be applied directly to solid samples such as plastic pellets or cylindrical pieces of cable insulation.

The weighed sample to be analyzed is placed in a polyethylene container for transfer from the sample loading port to the irradiation site in the reactor. Several samples, standards, and monitors may be irradiated simultaneously provided that the self-shielding effects of multiple samples on each other are well understood (7). After irradiation for a pre-selected time, the samples are returned to the sample receiver. After an appropriate decay period to allow the decay of short-lived radio-isotopes, typically 24 h, the samples are manually unpacked and transferred from the receiver to the germanium semiconductor detector or to a mechanical sample changer which transports them one by one at the appropriate time to the counting position at the detector. The signals from the detector are sent to a multichannel pulse-height analyzer which measures the energies of the individual gamma-rays and places them in a gamma-ray spectrum. The spectrum has peaks at the characteristic energies of the elements present in the sample. The spectrum for each sample is stored for subsequent analysis.

4.2 The amount of total antimony (all chemical forms) in the sample is proportional to the corrected and normalized peak area and is quantified by use of the corrected and normalized peak area of the comparator standard(s).

4.3 When antimony is irradiated with neutrons, the atoms of the isotope ^{121}Sb capture neutrons and are converted to ^{122}Sb which is radioactive with a half-life of 2.72 days. ^{122}Sb decays by emitting a beta-ray and gamma-rays of several possible energies. From Ref (8), the main gamma-ray, at 564.2 keV, is emitted in 70.67 % of decays. The amount of total antimony (all chemical forms) in the sample is proportional to the corrected number of counts in the peak at 564.2 keV. The area of the peak at 564.2 keV is corrected for counts beneath the peak due to Compton scattered gamma-rays and for pulse losses (dead-time) in the combined detector-multichannel pulse-height analyzer system. All modern multichannel pulse-height analyzers accurately correct for pulse losses up to their maximum useable count-rates.

4.3.1 The detector must have good energy resolution because the peak at 564.2 keV must be well separated from nearby peaks such as those from ^{82}Br at 554.3 keV and ^{76}As at 559.1 keV.

4.4 In addition to ^{121}Sb capturing neutrons to produce ^{122}Sb , the ^{123}Sb isotope captures neutrons to produce ^{124}Sb , with a 60-day half-life. This isotope has strong gamma lines at 603 keV and 1691 keV. Measurement of both ^{122}Sb and ^{124}Sb provides an additional verification of the method's accuracy. This standard employs the most sensitive ^{122}Sb gamma-ray, but the same methods and equations apply equally to all gamma-rays of both isotopes.

5. Significance and Use

5.1 High levels of antimony are commonly used in flame retardant formulations for various materials. NAA is a test method that can be useful for verifying these levels and, for other materials, NAA can also be useful in establishing the amount of low level contamination, if any, with high sensitivity and high precision.