Designation: D3649 – 23

Standard Practice for High-Resolution Gamma-Ray Spectrometry of Water[1]

This standard is issued under the fixed designation D3649; 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 practice covers the measurement of gamma-ray emitting radionuclides in water by means of gamma-ray spectrometry. It is applicable to nuclides emitting gamma-rays with energies greater than 45 keV. For typical counting systems and sample types, activity levels of about 40 Bq are easily measured and sensitivities as low as 0.4 Bq are found for many nuclides. Count rates in excess of 2000 counts per second should be avoided because of electronic limitations. High count rate samples can be accommodated by dilution, by increasing the sample to detector distance, or by using digital signal processors.

1.2 This practice can be used for either quantitative or relative determinations. In relative counting work, the results may be expressed by comparison with an initial concentration of a given nuclide which is taken as 100 %. For quantitative measurements, the results may be expressed in terms of known nuclidic standards for the radionuclides known to be present. This practice can also be used just for the identification of gamma-ray emitting radionuclides in a sample without quantifying them. General information on radioactivity and the measurement of radiation has been published (**1**,**2**).[2] Information on specific application of gamma spectrometry is also available in the literature (**3-5**). See also the referenced ASTM Standards in 2.1 and the related material section at the end of this standard.

1.3 *This standard does not purport to address 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 limitation prior to use.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1066 Practice for Sampling Steam
D1129 Terminology Relating to Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Flowing Process Streams
D3648 Practices for the Measurement of Radioactivity
D4448 Guide for Sampling Ground-Water Monitoring Wells
D7902 Terminology for Radiochemical Analyses
E181 Guide for Detector Calibration and Analysis of Radionuclides in Radiation Metrology for Reactor Dosimetry

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1129 and Terminology D7902. For terms not defined in this practice or in Terminology D1129 or Terminology D7902, reference may be made to other published glossaries.

4. Summary of Practice

4.1 Gamma ray spectra are measured with modular equipment consisting of a detector, high-voltage power supply, preamplifier, amplifier and analog-to-digital converter (or digital signal processor), multichannel analyzer, as well as a computer with display.

4.2 High-purity germanium (HPGe) detectors, p-type or n-type, are used for the analysis of complex gamma-ray spectra because of their excellent energy resolution. These germanium systems, however, are characterized by high cost and require cooling. Liquid nitrogen or electromechanical cooling, or both, can be used.

4.3 In a germanium semiconductor detector, gamma-ray photons produce electron-hole pairs. The charged pair is then

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved June 1, 2023. Published July 2023. Originally approved in 1978. Last previous edition approved in 2014 as D3649 – 06 (2014) which was withdrawn January 2023 and reinstated in June 2023. DOI: 10.1520/D3649-23.

[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[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.

collected by an applied electric field. A very stable low noise preamplifier is needed to amplify the pulses of electric charge resulting from gamma photon interactions. The output from the preamplifier is directly proportional to the energy deposited by the incident gamma-ray. These current pulses are fed into an amplifier of sufficient gain to produce voltage output pulses in the amplitude range from 0 V to 10 V.

4.4 A multichannel pulse-height analyzer is used to determine the amplitude of each pulse originating in the detector, and accumulates in a memory the number of pulses in each amplitude band (or channel) in a given counting time. Computerized systems with stored programs and interface hardware can accomplish the same functions as hardwired multichannel analyzers. The primary advantages of the computerized system include the capability of programming the multi-channel analyzer functions and the ability to immediately perform data reduction calculations using the spectral data stored in the computer memory or mass storage device. For a 0 MeV to 2 MeV spectrum, 4000 or more channels are typically needed in order to fully utilize a germanium detector's excellent energy resolution.

4.5 The distribution of the amplitudes (pulse heights) of the pulses can be separated into two principal components. One of these components has a nearly Gaussian distribution and is the result of total absorption of the gamma-ray energy in the detector. This peak is normally referred to as the full-energy peak or photopeak. The other component is a continuous one lower in energy than that of the photopeak. This continuous curve is referred to as the Compton continuum and is due to interactions wherein the gamma photons deposit only part of their energy in the detector. These two portions of the curve are shown in Fig. 1. Other peaks, such as escape peaks, backscattered gamma rays or X rays from shields, are often superimposed on the Compton continuum. Escape peaks will be present when gamma-rays with energies greater than 1.02 MeV are emitted from the sample. The positron formed in pair production is usually annihilated in the detector and one or both of the 511 keV annihilation quanta may escape from the detector without interaction. This condition will cause single or double escape peaks at energies of 0.511 MeV or 1.022 MeV less than the photopeak energy. In the plot of pulse height versus count rate, the size and location of the photopeak on the pulse height axis are proportional to the number and energy of the incident photons, respectively, and are the basis for the quantitative and qualitative application of the spectrometer. The Compton continuum serves no useful purpose in photopeak analysis and must be subtracted when peaks are analyzed.

4.6 If the analysis is being directed and monitored by an online computer program, the analysis period may be terminated by prerequisites incorporated in the program. If the analysis is being performed with a modern multichannel analyzer, analysis may be terminated when a preselected time or total counts in a region of interest or in a specified channel is reached. Visual inspection of a display of accumulated data can also be used as a criterion for manually terminating the analysis on either type of data acquisition systems.

4.7 Upon completion of the analysis, the spectral data are interpreted and reduced to radionuclide activities in becquerels (Bq) or other units suited to the particular application. At this time the spectral data may be inspected to identify the gamma-ray emitters present. This is accomplished by reading the channel number from the *x*-axis and converting to gamma-ray energy by multiplying by the appropriate keV/channel (system gain). In some systems the channel number or gamma-ray energy in keV can be displayed for any selected channel. Identification of nuclides may be aided by catalogs of gamma-ray spectra and other nuclear data tabulations (**3, 6 and 7**).

4.7.1 Computer programs for data reduction have been used extensively although calculations for some applications can be performed effectively with the aid of a scientific calculator. Data reduction of spectra taken with germanium spectrometry systems is usually accomplished by integration of the photopeaks above a definable background (or baseline) and subsequent activity calculations using a library which includes data

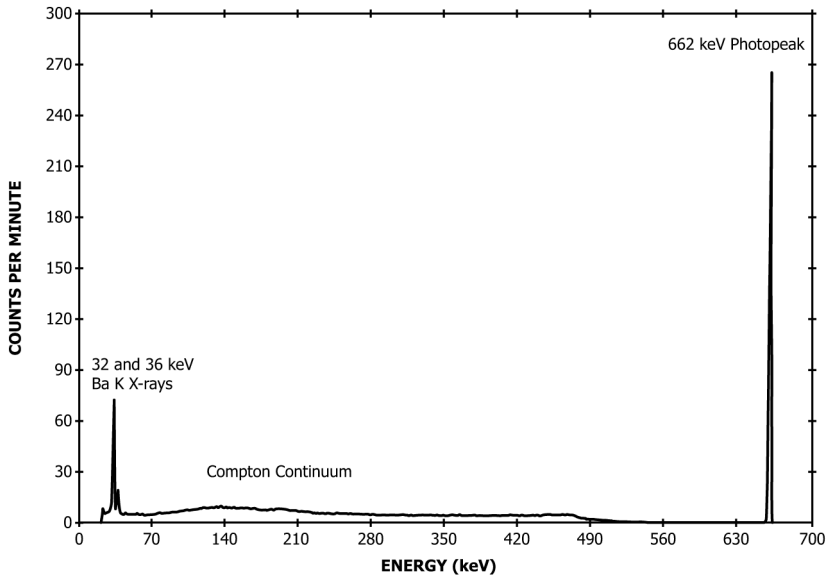

FIG. 1 Cesium-137 Spectrum