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Standard Guide for High-Resolution Gamma-Ray Spectrometry of Soil Samples¹

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1. Scope

1.1 This guide covers the identification and quantitative determination of gamma-ray emitting radionuclides in soil samples by means of gamma-ray spectrometry. It is applicable to nuclides emitting gamma rays with an approximate energy range of 20 to 2000 keV. For typical gamma-ray spectrometry systems and sample types, activity levels of about 5 Bq (135 pCi) are measured easily for most nuclides, and activity levels as low as 0.1 Bq (2.7 pCi) can be measured for many nuclides. It is not applicable to radionuclides that emit no gamma rays such as the pure beta-emitting radionuclides hydrogen-3, carbon-14, strontium-90, and becquerel quantities of most transuranics. This guide does not address the in situ measurement techniques, where soil is analyzed in place without sampling. Guidance for in situ techniques can be found in Ref (1) and (2).² This guide also does not discuss methods for determining lower limits of detection. Such discussions can be found in Refs (3), (4), (5), and (6).

1.2 This guide can be used for either quantitative or relative determinations. For quantitative assay, the results are expressed in terms of absolute activities or activity concentrations of the radionuclides found to be present. This guide may also be used for qualitative identification of the gamma-ray emitting radionuclides in soil without attempting to quantify their activities. It can also be used to only determine their level of activities relative to each other but not in an absolute sense. General information on radioactivity and its measurement may be found in Refs (7), (8), (9), (10), and (11) and Standard Test Methods E181. Information on specific applications of gamma-ray spectrometry is also available in Refs (12) or (13). Practice D3649 may be a valuable source of information.

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 may involve hazardous material, operations, and equipment. 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.

1.5 *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.*

2. Referenced Documents

2.1 ASTM Standards:³

- C859 Terminology Relating to Nuclear Materials
- C998 Practice for Sampling Surface Soil for Radionuclides
- C999 Practice for Soil Sample Preparation for the Determination of Radionuclides
- C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry
- D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water
- D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
- E181 Test Methods for Detector Calibration and Analysis of Radionuclides
- IEEE/ASTM-SI-10 Standard for Use of the International System of Units (SI) the Modern Metric System

2.2 ANSI Standards:⁴

- N13.30 Performance Criteria for Radiobioassay
- N42.14 Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
- N42.23 American National Standard Measurement and Associated Instrumentation
- IEEE-325 Standard Test Procedures for Germanium Gamma-Ray Detectors

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² The boldface numbers in parentheses refer to the 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.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

4. Summary of Guide

4.1 High-resolution germanium detectors and multichannel analyzers are used to ensure the identification of the gamma-ray emitting radionuclides that are present and to provide the best possible accuracy for quantitative activity determinations.

4.2 For qualitative radionuclide identifications, the system must be energy calibrated. For quantitative determinations, the system must also be shape and efficiency calibrated. The standard sample/detector geometries must be established as part of the efficiency calibration procedure.

4.3 The soil samples typically need to be pretreated (for example, dried), weighed, and placed in a standard container. For quantitative measurements, the dimensions of the container holding the sample and its placement in front of the detector must match one of the efficiency-calibrated geometries. If multiple geometries can be selected, the geometry chosen should reflect the detection limit and count rate limitations of the system. Qualitative measurements may be performed in non-calibrated geometries.

4.4 The identification of the radionuclides present is based on matching the energies of the observed gamma rays in the spectrum to computer-based libraries of literature references [see Refs (14), (15), (16), (17), or (18)]. The quantitative determinations are based on comparisons of observed count rates to previously obtained counting efficiency versus energy calibration data, and published branching ratios for the radionuclides identified.

5. Significance and Use

5.1 Gamma-ray spectrometry of soil samples is used to identify and quantify certain gamma-ray emitting radionuclides. Use of a germanium semiconductor detector is necessary for high-resolution gamma-ray measurements.

5.2 Much of the data acquisition and analysis can be automated with the use of commercially available systems that include both hardware and software. For a general description of the typical hardware in more detail than discussed in Section 7, see Ref (19). For best practices on set-up, calibration, and quality control of utilized spectrometry systems, see Practice D7282.

5.3 Both qualitative and quantitative analyses may be performed using the same measurement data.

5.4 The procedures described in this guide may be used for a wide variety of activity levels, from natural background levels and fallout-type problems, to determining the effectiveness of cleanup efforts after a spill or an industrial accident, to tracing contamination at older production sites, where wastes were purposely disposed of in soil. In some cases, the combination of radionuclide identities and concentration ratios can be used to determine the source of the radioactive materials.

5.5 Collecting samples and bringing them to a data acquisition system for analysis may be used as the primary method to detect deposition of radionuclides in soil. For obtaining a representative set of samples that cover a particular area, see Practice C998. Soil can also be measured by taking the data acquisition system to the field and measuring the soil in place (in situ). In situ measurement techniques are not discussed in this guide.

6. Interferences

6.1 In complex mixtures of gamma-ray emitters, the degree of interference of one nuclide in the determination of another is governed by several factors. Interference will occur when the photopeaks from two separate nuclides overlap within the resolution of the gamma-ray spectrometer. Most modern analysis software can deconvolute multiplets where the separation of any two adjacent peaks is more than 0.5 FWHM (see Refs (20) and (21)). For peak separations that are smaller than 0.5 FWHM, most interference situations can be resolved with the use of automatic interference correction algorithms (22).

6.2 If the nuclides are present in the mixture in very unequal radioactive portions and if nuclides of higher gamma-ray energy are predominant, the interpretation of minor, less energetic gamma-ray photopeaks becomes difficult due to the high Compton continuum and backscatter.

6.3 True coincidence summing (also called cascade summing) occurs regardless of the overall count rate for any radionuclide that emits two or more gamma rays in coincidence. Cobalt-60 is an example where both a 1173-keV and a 1332-keV gamma ray are emitted from a single decay. If the sample is placed close to the detector, there is a finite probability that both gamma rays from each decay interact within the resolving time of the detector resulting in a loss of counts from both full energy peaks. Coincidence summing and the resulting losses to the photopeak areas can be considerable (>10 %) before a sum peak at an energy equal to the sum of the coincident gamma-ray energies becomes visible. Coincidence summing and the resulting losses to the two individual photopeak areas can be reduced to the point of being negligible by increasing the source to detector distance or by using a small detector. Coincidence summing can be a severe problem if a well-type detector is used. See Test Methods E181 and (7) for more information.

6.4 Random summing is a function of count rate (not dead time) and occurs in all measurements. The random summing rate is proportional to the total count squared and to the resolving time of the detector and electronics. For most systems, uncorrected random summing losses can be held to less than 1 % by limiting the total counting rate to less than 1000 counts/s. However, high-precision analyses can be performed at high count rates by the use of pileup rejection circuitry and dead-time correction techniques. Refer to Test Methods E181 for more information.

7. Apparatus

7.1 *Germanium Detector Assembly*—The detector should have an active volume of greater than 50 cm³, with a full width at one half the peak maximum (FWHM) less than 2.0 keV for

the cobalt-60 gamma ray at 1332 keV, certified by the manufacturer. A charge-sensitive preamplifier should be an integral part of the detector assembly.

7.2 Sample Holder Assembly—As reproducibility of results depends directly on reproducibility of geometry, the system should be equipped with a sample holder that will permit using reproducible sample/detector geometries for all sample container types that are expected to be used at several different sample-to-detector distances.

7.3 Shield—The detector assembly should be surrounded by a radiation shield made of material of high atomic number providing the equivalent attenuation of 100 mm (or more in the case of high background radiation) of low-activity lead. It is desirable that the inner walls of the shield be at least 125 mm distant from the detector surfaces to reduce backscatter and annihilation radiation. If the shield is made of lead or has a lead liner, the shield should have a graded inner shield of appropriate materials, for example, 1.6 mm of cadmium or tin-lined with 0.4 mm of copper, to attenuate the induced 88-keV lead fluorescent X-rays. The shield should have a door or port for inserting and removing samples. The materials used to construct the shield should be prescreened to ensure that they are not contaminated with unacceptable levels of natural or man-made radionuclides. The lower the desired detection capability, the more important it is to reduce the background. For very low activity samples, the detector assembly itself, including the preamplifier, should be made of carefully selected low background materials.

7.4 High-Voltage Power/Bias Supply—The bias supply required for germanium detectors usually provides a voltage up to ± 5000 V and 1 to 100 μ A. The power supply should be regulated to 0.1 % with a ripple of not more than 0.01 %. Noise caused by other equipment should be removed with r-f filters and power line regulators.

7.5 Amplifier—A spectroscopy amplifier which is compatible with the preamplifier. If used at high count rates, a model with pile-up rejection should be used. The amplifier should be pole-zeroed properly prior to use.

7.6 Data Acquisition Equipment—A multichannel pulse-height analyzer (MCA) with a built-in or stand-alone analog-to-digital converter (ADC) compatible with the amplifier output and pileup rejection scheme. The MCA (hardwired or a computer-software-based) collects the data, provides a visual display, and stores and processes the gamma-ray spectral data. The four major components of an MCA are: ADC, memory, control, and input/output. The ADC digitizes the analog pulses from the amplifier. The height of these pulses represents energy deposited in the detector. The digital result is used by the MCA to select a memory location (channel number) which is used to store the number of events which have occurred at the energy. The MCA must also be able to extend the data collection time for the amount of time that the system is dead while processing pulses (live time correction).

7.7 Count Rate Meter—It is useful but not mandatory to have a means to measure the total count rate for pulses above the amplifier noise during the measurement. If not provided by

the MCA, a separate count rate meter may be used for this purpose. In the absence of a rate meter, count rates that are too high to provide reliable results may also be detected by monitoring the system dead time or peak resolution, or both.

7.8 Pulser—Required only if random summing effects are corrected with the use of a stable pulser (23) and (24).

7.9 Computer—Most modern gamma-ray spectrometers are equipped with a computer for control of the data acquisition as well as automated analysis of the resulting spectra. Such computer-based systems are readily available from several commercial vendors. Their analysis philosophies and capabilities do differ from each other somewhat. See ANSI N42.14 for a series of tests on how to tell if a particular gamma-spectrometry software package has adequate analysis capabilities. In addition to the analysis capabilities, it is important to consider the overall user interface and architecture of the software. For small-scale operations, a few samples per week, a user interface that requires a lot of user intervention is sufficient. For larger-scale operations, with hundreds of samples per week on multiple detectors, a software package that permits some kind of batch processing and automated operation is recommended.

8. Container for a Test Sample

8.1 Sample holders and containers must have a reproducible geometry. Considerations include commercial availability, ease of use and disposal, and the containment of radioactivity for protection of the working environment, personnel, and the gamma-ray spectrometer from contamination. For small soil samples (up to a few grams), plastic bottles are convenient containers, while large samples (up to several kilograms), which require greater sensitivity, are frequently packaged in Marinelli beakers. For analyzing low-energy gamma rays at close geometries, the consistency of the wall thickness of the sample container facing the detector becomes an important factor in the variability of the analysis results.

8.2 Measurements may require precautions to prevent the loss of volatile radionuclides. For example, the direct determination of radium-226 in soil by the measurement of the 609-keV gamma ray of bismuth-214 assumes secular equilibrium between radium-226 and its bismuth-214 progeny and that the radon-222 daughter was not lost from the sample.

8.3 A beta absorber consisting of about 6 mm of aluminum, beryllium, or plastic should be placed between the detector and sample for samples that have significant quantities of high-energy beta emitters.

9. Calibration and Standardization

9.1 Overview:

9.1.1 Commission and operate the instrumentation and detector in accordance with the manufacturer's instructions and best practices such as may be contained in Practice D7282. Initial set-up includes all electronic adjustments to provide constant operating conditions consistent with the application and life expectancy of the calibrations. The analog-to-digital converter gain and range, amplifier gain, and zero-level must be adjusted to yield an optimum energy calibration. Both the