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Standard Guide for
High-Resolution Gamma-Ray Spectrometry of Soil Samples[1]

This standard is issued under the fixed designation C 1402; 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 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 ~~energies greater than~~ 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 General Methods E-181~~and Standard Test Methods E 181. Information on specific applications of gamma-ray spectrometry is also available in Refs **(12)** or **(13)**. Practice D 3649 ~~is a valuable source of information.~~

~~1.3~~*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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C 998 Practice for Sampling Surface Soil for Radionuclides
C 999 Practice for Soil Sample Preparation for the Determination of Radionuclides
C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry
D 3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water
E 181 ~~General~~Test Methods for Detector Calibration and Analysis of Radionuclides
~~E380~~IEEE/ASTM-SI-10 ~~Practice~~Standard for Use of the International System of Units (SI) the ~~Modernized~~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 Measurement Quality Assurance for Radioassay Laboratories
ANSI/IEEE-645 Test Procedures for High Purity Germanium Detectors for Ionizing Radiation

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[1] This guide is under the jurisdiction of ASTM Committee ~~C-26~~C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.
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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* ~~, Vol 12.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

~~[4] Annual Book of ASTM Standards, Vol 11.02.~~

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

3. Summary of Guide

3.1 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.

3.2 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.

3.3 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.

3.4 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~~[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.

4. Significance and Use

4.1 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.

4.2 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 6, see Ref **(19)**.

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

4.4 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.

4.5 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 C 998. 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.

5. Interferences

5.1 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)**.

5.2 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.

5.3 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 ~~General~~Test Methods E 181 and **(7)** for more information.

5.4 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 count/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 ~~General~~Test Methods E 181 for more information.

6. Apparatus

6.1 *Germanium Detector Assembly* —The detector should have an active volume of greater than 50 cm^3, 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 ~~using low-noise field-effect transistors~~ should be an integral part of the detector assembly.

6.2 *Sample Holder Assembly*—As reproducibility of results depends directly on reproducibility of geometry, the system should