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

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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 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 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 preamplifer, 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 µA. 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 progency 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 energy and efficiency calibration must be accomplished with radioactive sources covering the entire energy range of interest (**6, 7** and Test Methods E181). Subsequent efficiency calibrations and source analyses are performed with the same gain settings and the same high-voltage setting. Prepare efficiency calibration standards by weighing an appropriate amount of a radionuclide standard solutions containing 100 to 10 000 Bq each onto a soil matrix in an appropriate container, drying it, and mixing thoroughly. Standardized dried soil and bottom sediment are also available from the U.S. National Institute of Standards and Technology (NIST) or other appropriate sources which can be used directly or diluted with ambient soil to a measured weight or volume. Prepare blank sources containing the same quantity of unspiked soil to account for any naturally occurring radionuclides that may be present. Commercially available epoxy soil-equivalent standards with an appropriate mixture of radionuclides can also be used. It should be noted that soils that contain high atomic number materials will significantly alter the expected self-attenuation.

9.1.2 Follow the manufacturer's instructions, limitations, and cautions for the setup and the preliminary testing for all of the spectrometry equipment to be used in the analysis. This equipment would include, as applicable, detector, power supplies, preamplifiers, amplifiers, multichannel analyzers, and computing systems. For example, ensure that the detector has had ample time (typically 6–8 h) to cool down after the first filling with liquid nitrogen before turning on the high voltage. Also, ensure that the high-voltage bias supply is set for the recommended operating voltage and the correct polarity.

9.1.3 Place an appropriate weight of standardized dried soil in an appropriate soil matrix in a sealed container and place the container at a desirable and reproducible source-to-detector distance. The standard (traceable to a designated standards organization) should provide enough counts in each calibration peak (typically 20 000 or more, see Test Methods E181 or ANSI N42.14) in a reasonable amount of time (4–12 h). In all radionuclide measurements, the volumes, shape, and physical and chemical characteristics of all the samples and standards and their containers must be as identical as practical for the most accurate results. For situations where it is not possible or practical to produce standards that are identical to the samples, standard matrices that are different from the sample matrices have been found to provide acceptable results when coupled with attenuation correction methods.

9.2 *Energy and Shape Calibration:*

9.2.1 The energy and shape calibration (the peak gamma-ray energy versus channel number of the multichannel analyzer and peak shape versus the peak gamma-ray energy) of the detector system is determined at a specific gain setting (typically 0.5 keV/channel) using standards containing known radionuclides. The peak shape calibration may involve only calculating the peak resolution (full-width-at-half-maximum, or FWHM), or include other, nonsymmetrical components as well. The standards should be in sealed containers and should emit at least eight different gamma-ray energies covering the range of interest, usually from 20 to 2000 keV, in order to test for system linearity. If the calibration is performed with only the radionuclides of interest, fewer gamma-ray energies can also be used. Energy and shape calibration can be performed without NIST traceable sources.

9.2.2 Verify the radionuclide purity of the standard periodically to ensure against accidental contamination or the presence of long-lived impurities by comparing the observed gamma rays with the data published in the literature. Careful adherence to precautions and certificate calibration instructions are necessary when using the calibration standards.

9.2.3 Calibrate a multichannel analyzer for energy, shape, and efficiency to cover the energy range or interest. If the range of interest is from 20 to 2000 keV, adjust the gain of the system until the centroid of the cesium-137 photopeak, 661.6 keV, is about one-third full-scale. Leaving the gain constant, locate at least three other photopeaks of different energies within the energy range of interest. Determine and record the peak centroid for each of the four gamma energies. A linear relationship between the gamma-ray energies and their channel numbers should be observed if the equipment is operating properly. Calculate the slope and intercept of the line using a least-squares calculation. If the spectrometry system is computerized, follow the appropriate manufacturer input instructions for the determination of the slope and intercept.

9.2.4 If the system is being calibrated with the radionuclides of interest, fewer lines may be used for calibration and the linearity of the MCA is not an issue as long as the peaks of interest are identified and quantified consistently.

9.3 *Efficiency Calibration:*

9.3.1 Efficiency calibration must be performed with sources that are traceable to a national standards laboratory, such as NIST. A mixed gamma-ray standard for both energy and efficiency calibration containing Am-241, Cd-109, Co-57, Ce-139, Hg-203, Sn-113, Sr-85, Cs-137, Y-88, and Co-60 is available from many commercial source manufacturers who provide NIST traceable sources. The gamma-ray energies of this mixed standard as well as some other commercially available NIST traceable radionuclides that are suitable for efficiency calibration (and energy and shape calibration) are shown in Table 1. As another example, an antimony-125/europium-154,155 mixture from NIST (SRM 4275B or its replacement) has 19 major photopeaks between 100 and 1600 keV.

TABLE 1 Radionuclides Useful for Energy Calibration

Radionuclide	Gamma-Ray Energy, keV
Lead-210	46.5
Americium-241	59.5
Cadmium-109	88.0
Cobalt-57	122.1 and 136.5
Cerium-141	145.5
Cerium-139	165.9
Mercury-203	279.2
Chromium-51	320.1
Barium-133	356.0
Tin-113	391.7
Strontium-85	514.0
Cesium-137	661.6
Manganese-54	834.8
Sodium-22	511.0 and 1274.5
Yttrium-88	898.0 and 1836.0
Cobalt-60	1173.2 and 1332.4

9.3.2 For environmental or low-activity samples (0.01 to 1 Bq/g), typically, 300 to 500 g of prepared soil are used. If a fixed volume is used, the mass will vary according to the density. High-density samples may cause significant self-absorption of low-energy gamma rays and degrade the higher-energy gammas. Therefore, it is important to calibrate the detector with standards of the same geometry, composition, and density, or use appropriate attenuation and geometry correction algorithms.

9.3.3 Accumulate a gamma-ray spectrum using sealed, calibrated radioactivity standards until there are approximately 20 000 net counts (see Test Methods E181 or ANSI N42.14) in each full-energy calibration peak provided that this does not require an excessive amount of time. To achieve reasonable count times, smaller peak sizes may also be used. Compare the length of the count to the half-life of the radionuclide of interest. If the duration of the count is a significant portion of the half-life (>10 %), a correction factor must be applied for decay during the count. Refer to Standard Test Methods E181 or Ref **(6)** for additional information. To correct the results to the start of the measurement due to decay during spectrum acquisition, use the following equation:

$$K = \frac{\lambda T}{1 - e^{-\lambda T}} \tag{1}$$

where:

K = multiplicative corrective for peak area or intensity,
λ = nuclide decay constant (in the same time units as T), and
T = data collection elapsed clock time (that is, real time, not live time).

9.3.4 The equation shown in 9.3.3 is not sufficient if the count rate changes significantly during data acquisition, as might happen if a short-lived nuclide is the main source of the activity. Other methods, such as the Virtual Pulser **(25)** and add "N" Method **(26)** may be used for a varying count rate situation.

9.3.5 Correct the radioactivity standard source gamma-ray emission rate for the decay from the time of standardization to the start of data acquisition. Many commercial and noncommercial data analysis software packages will do this automatically as part of the efficiency calibration.

9.3.6 Calculate the full-energy peak efficiency, E_f, as follows:

$$E_f = \frac{N_p}{N_g} \tag{2}$$

where:

E_f = full-energy peak efficiency (counts recorded per gamma ray emitted),
N_p = net gamma-ray count rate in the full-energy peak of interest (counts per second), and
N_g = gamma-ray emission rate (gamma rays per second).

9.3.7 If the standard source is calibrated for activity rather than emission rate, the gamma-ray emission rate is given as follows:

$$N_g = A \cdot P_g \tag{3}$$

where:

A = number of nuclear decays per second, and
P_g = emission probability for the gamma ray per nuclear decay.

9.3.8 Many modern spectrometry systems are computerized, and the determination of the gamma-ray efficiencies is accomplished automatically at the end of an appropriate counting interval. Refer to the manufacturer's instructions for specific input requirements. It is necessary for the user to determine the basis of the system analysis and its limitations.

9.3.9 Plot the values for the full-energy peak efficiency (as determined in 9.3.6) versus gamma-ray energy. The plot will allow the determination of efficiencies at energies for which standards are not available. Many computerized systems provide such plotting capabilities as part of the overall functionality of the system. Computerized systems also typically provide a variety of different calculational models to automatically calculate the efficiencies at any energy. The calibration curve, regardless of whether it is calculated by hand or by a computerized system, generally should not be used for peak energies beyond the first and last calibration points. If it is necessary to use the calibration curve in such a manner, one must pay particular attention to establishing appropriate uncertainties for any peak energies outside the calibration range. Alternatively, calibrate using standards of the radionuclides of interest to obtain direct calibration factors for them without establishing an efficiency curve.

10. Measurement Control

10.1 A properly run laboratory must have a measurement control program to verify that the detection system is in calibration. See Guide C1009 or Ref **(27)** for further guidance on laboratory measurement control programs.

10.2 As a minimum, the following periodic checks should be made.

10.2.1 Each day or prior to each measurement, energy, resolution, and efficiency response should be checked using at least two different gamma-ray energies. If the energy calibration slope and intercept are essentially unchanged, the energy calibration data are assumed to remain valid. If an appreciable change in the slope or intercept is evident, the reason should be determined and corrected.

10.2.2 Once the efficiencies for the various sample sizes, matrices, and source-to-detector distances have been established, it is not necessary to repeat the process unless there is a change in resolution or system configuration, or a new sample size, matrix, or geometry is added, or the detector has failed and has been returned to service after it has been repaired. However, a complete check of the efficiency and energy calibrations should be done periodically (typically annually). Similar tests should be used to determine loss of resolution or efficiency **(28)**.

10.2.3 Ideally, a measurement of the room background should be made before and after any series of determinations.

10.2.4 A measurement of a standard or sample with a known concentration to provide a measurement bias check. Radioactive decay of the standard must be taken into account.

10.2.5 Periodic replicate measurements of a standard or sample to determine that the precision reported by the analysis method, such as a computer program, is appropriate.

10.3 It is recommended that control charts and other periodic statistical analysis of the precision and bias data be used.

11. Sample Measurements

11.1 After the spectrometer system has been set up and the energy and efficiency calibrations performed, unknown samples can be measured.

11.2 Soil samples are collected by methods in accordance with Practice C998 and prepared for analysis in accordance with Practice C999. An appropriate aliquot of soil is transferred to the sample container (8.1) and positioned in the same manner as was done during system calibration (Section 9).

11.3 Measure the activity of the sample for a period of time long enough to acquire a gamma-ray spectrum which will meet the minimum acceptable counting uncertainty for the radionuclides of interest.

12. Calculation

12.1 Ambient background peak areas must be subtracted if their contributions are not negligible. In many experiments, the background may not affect the results but is still monitored to ensure the integrity of the system. The method presented here is not the only acceptable one but is compatible with available computational hardware and should be used to verify the validity of commercial software.

12.2 The underlying aim of this method is to subtract the continuum or baseline from the spectral data where it underlies a photopeak of interest. For operator-directed calculations, the choice of the baseline level may be straightforward. The simplest way, using a plot of the spectral data, is to draw a straight line, using judgement and experience, that best describes the baseline. The baseline data can be read directly from the plot and subtracted. A variety of computer programs have accomplished this but details are not included in this guide.

12.3 Photopeaks lying on a sloping baseline, or one with curvature, will be analyzed, regardless of method, with increased uncertainty. Use of data from these peaks should be limited to those cases where there is no other alternative. Photopeaks that overlap with each other will also increase the uncertainty of the final result. In the case where use of overlapping peaks cannot be avoided and software programs are not available, the experimenter may estimate the areas by assuming that the ratio of the peak areas is equal to the ratio of the peak heights, but this may introduce a sizable error. Computer programs separating overlapping peaks with varying degrees of success may be found in Refs (**29**), (**30**). The current quality of peak reduction and deconvolution software available from the major counting system manufacturers is adequate for most situations.

12.4 The radioactive decay process is governed by Poisson statistics. In Poisson statistics, the variance in *N* accumulated events acquired with a detector is simply *N*. The standard deviation is the square root of the variance.

12.5 The areas of well-resolved spectral peaks can be determined by summing the data above the underlying continuum. For narrow peaks, the continuum can be well-approximated by interpolation of the background line. The peak area can then be calculated from the following equation:

$$C = P - \left[\frac{N_p}{2} \left(\frac{B1}{N_{B1}} + \frac{B2}{N_{B2}} \right) \right] \tag{4}$$

where:

C = net peak area (counts),
P = total counts in the peak including the continuum,
N_p = number of channels summed for P,
$B1$ and $B2$ = number of counts in continuum regions on either side of peak, and
N_{B1} *and* N_{B2} = number of channels summed to determine $B1$ and $B2$, respectively.

12.6 The variance in quantity *f* that is a function of *n* independent variables *x* is approximated by the following equation:

$$\sigma^2 (f) = \sum_{i=1}^{n} \left(\frac{\alpha f}{\alpha x_i} \right)^2 \sigma^2 (x_i) \tag{5}$$

12.7 Hence, the propagated variance of the area *C* is given by the following expression (remembering that the variance of $P = P$, variance of $B1 = B1$, and variance of $B2 = B2$):

$$\sigma^2 (C) = P + \left[\left(\frac{N_p}{2 \cdot N_{B1}} \right)^2 \cdot B1 \right] + \left[\left(\frac{N_p}{2 \cdot N_{B2}} \right)^2 \cdot B2 \right] \tag{6}$$

where:

$\sigma(C)$ = standard deviation of Peak Area C.

12.8 In order to determine radionuclide activity concentrations, the photopeak areas, corrected for background and interferences, are divided by the count time and efficiency for the energy of the gamma ray being calculated to yield gammas per second for the peak of interest. If, as is the case for some radionuclides, the gamma-ray abundance is 100 %, division by the detector efficiency converts counts per second for the photopeak of interest to decays per second (bequerels) for the nuclide. If not, the gammas per second are converted to disintegrations per second by dividing the gammas per second by the gammas per disintegration, for the nuclide and photopeak of interest. The results are then corrected for sampling or decay, or both, as demanded by the application. The activity of a particular radionuclide may be calculated using the following equation:

$$A = \frac{C}{T \cdot \varepsilon \cdot K_\mu \cdot B \cdot W \, e^{-\lambda T_w}} \tag{7}$$

where:

A = activity concentration, Bq/g,
ε = efficiency of the spectrometer for the gamma ray of interest,
K_μ = correction factor to accommodate for the attenuation in the sample matrix compared to the attenuation in the matrix of the standard source,
B = number of gamma rays emitted per decay,
$e^{-\lambda Tw}$ = decay correction,

W = weight of sample, g,
T = count time, s, (live time), and
T_w = decay time.

The energies, half-lives, and gammas per disintegration for typical radionuclides that might be present in soil samples are available in Refs (**14-18**).

12.8.1 The activity of the *i*th radionuclide can also be calculated as follows:

$$A_i = \frac{k_i \; C}{T \cdot W \; e^{-\lambda T_w}} \qquad (8)$$

where:

k_i = calibration factor for the *i*th radionuclide.

13. Precision and Bias

13.1 *Precision:*

13.1.1 Precision of the method is influenced by random counting uncertainties, and interferences in the spectra of the individual components with each other as well as the sampling uncertainty. The more complex the spectrum, the greater are the errors, and in general, major components can be determined more precisely than minor ones. Precision may be improved with increased counting time and by taking as large a sample of the soil volume being analyzed as possible. Variations in sample vial geometry and positioning will affect the precision of the measurement as well.

13.1.2 To illustrate typical precision of a gamma-ray spectrometry system, a check source was counted ten consecutive times without removing the source from the detector system. The ten single operator counts of ten minutes each provide a measure of system repeatability. The results are listed in Table 2.

13.1.3 The same source was used and another set of 10 measurements of 10 min each were made on successive days to capture more sources of variation. This process involved the normal day-to-day system operational checks and should provide a measure of the variability of operating procedures. The results are listed in Table 3.

13.2 *Bias:*

13.2.1 The calibration of standard sources, including errors introduced in using a standard radioactive solution or aliquot thereof, to prepare a working standard for bias correction may result in a bias. The full-energy peak efficiency at a given energy determined from the calibration function may introduce a bias.

13.2.2 The single-operator bias of a gamma-ray spectrometry system was estimated by measuring the NIST Rocky Flats Soil Number 1 (SRM 4353) and the NIST River Sediment (SRM 4350 B) six different times. The results are shown in Table 4.

13.3 *Sources of Error:*

13.3.1 Variation of the physical geometry of the sample and its relationship with the detector will produce both qualitative and quantitative variations in the gamma-ray spectrum. To adequately account for these geometry effects, efficiency calibrations (and occasionally also energy calibrations) should be designed to duplicate all conditions including source-to-detector distance, sample shape and size, and sample matrix. When it is not possible to have a calibration source that duplicates the sample matrix, the difference between the calibration matrix and the sample matrix as well as its height can be corrected for by a transmission measurement and a calculation and use of a geometry dependent attenuation factor (see Ref (**31**)).

13.3.2 Since some spectrometry systems are calibrated for various size sources at many different source-to-detector distances, a wide range of activity levels can be measured by the same detector. For high-activity samples (for example, $>10^6$ Bq), which may have resulted from a spill or accident, a large source-to-detector distance (for example >1 m) may be used. The large source-to-detector distance for high-activity samples will reduce the overall rate and thus minimize the random summing problems in the spectrum. A larger source-to-detector distance will also remove coincidence summing, if present, regardless of count rate.

13.3.3 Electronic problems, such as loss of resolution and random summing, may be minimized by keeping the overall count rate below 2000 counts/s. For most soil samples, a high count rate is not a problem. Some care may be needed in preparing or purchasing calibration standards so that their count rate in the measurement geometries stays below the desired limit. Total counting time is governed by the radioactivity of the sample, the detector-to-source distance, and the acceptable Poisson counting distribution uncertainty.

TABLE 2 Precision of Repeated Gamma-Ray Measurements in Counts per Minute (CPM) Without Removing the Sample Between Measurements

Measurement Number	Am-241	Cs-137	Co-60 (I)	Co-60 (II)
1	12,139	9558	1647	1419
2	12,139	9583	1642	1413
3	12,097	9587	1653	1414
4	12,119	9574	1649	1428
5	12,091	9607	1622	1408
6	12,080	9581	1637	1421
7	12,062	9580	1627	1418
8	12,134	9573	1640	1424
9	12,073	9615	1641	1430
10	12,133	9598	1648	1424
Mean	12,107	9586	1641	1420
stdev, cpm	30	16	9	7
stdev, %	0.23	0.17	0.57	0.49

TABLE 3 Precision of Reproducibility of Gamma-Ray Measurements in Counts per Minute (CPM) While Removing the Sample from the Measurement Position Between Measurements

Measurement Number	Am-241	Cs-137	Co-60 (I)	Co-60 (II)
1	12,078	9589	1646	1425
2	12,043	9502	1623	1407
3	11,950	9499	1617	1428
4	12,056	9587	1606	1397
5	12,097	9646	1634	1412
6	12,069	9628	1635	1428
7	12,090	9583	1634	1430
8	12,129	9588	1645	1432
9	12,111	9602	1636	1434
10	12,151	9582	1625	1440
Mean	12,077	9581	1630	1423
stdev, cpm	55	45	12	13
stdev, %	0.44	0.47	0.74	0.91

13.3.4 The density of the sample is another factor that can affect quantitative results. Errors from this source can be avoided by preparing the standards for calibration in matrices with a composition and density comparable to the sample being analyzed. The important factor is the linear attenuation which is the product of the density and the mass attenuation. Attenuation correction methods to correct for differences in the densities of the calibration source and actual samples, such as the use of a transmission source, are acceptable to use if they can be demonstrated to give good results.

14. Keywords

14.1 coincidence summing; gamma-ray; high-purity germanium; HPGe; photopeak; Poisson; radionuclides; shield; soil

TABLE 4 Results of NIST Rocky Flats Soil Number 1 (SRM 4353) and NIST River Sediment (SRM 4350 B) (Each measured value is an average of 6 measurements of 1000 min each. The estimated measured uncertainty includes only the counting statistics. The NIST uncertainty is the total uncertainty.)

SRM 4353 (Bq/ g)			
Radionuclide	Measured Value	NIST Value	Ratio Measured/NIST
K-40	0.713 ± 0.033	0.723 ± 0.069	0.99
Cs-137	0.0190 ± 0.0010	0.0176 ± 0.0008	1.08
Ra-226 (Bi-214)	0.0430 ± 0.0030	0.0430 ± 0.0028	1.00
Th-228 (Tl-208)	0.083 ± 0.002	0.0708 ± 0.0036	1.17
Th-232 (Ac-228)	0.0690 ± 0.005	0.0693 ± 0.0035	1.00
SRM 4350 B (Bq/g)			
Co-60	0.00464 ± 0.0024	0.00464 ± 0.00023	1.00
Cs -137	0.031 ± 0.002	0.0290 ± 0.0018	1.07
Eu-152	0.037 ± 0.003	0.0305 ± 0.0012	1.21
Eu-154	0.0035 ± 0.0022	0.00378 ± 0.00057	0.93
Ra-226 (Bi-214)	0.034 ± 0.002	0.0358 ± 0.0036	0.95

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