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Guidance for gamma spectrometry measurement of radioactive waste

Lignes directrices pour le mesurage de déchets radioactifs par spectrométrie gamma

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear fuel cycle*.

This first edition of ISO 19017 cancels and replaces ISO 14850-1:2006, which, in particular, did not take into account segmented measurements performed with collimators, the possible use of numerical simulation for calibration and uncertainty assessment, and gamma radiation detectors other than high-purity germanium semiconductors.

Introduction

A variety of non-destructive assay techniques are routinely used within the nuclear industry to measure or provide information to otherwise enable quantification of the radionuclide inventory of packages containing radioactive materials. This International Standard specifically considers gamma spectrometry measurements made on packages containing radioactive waste.

The methods and techniques discussed within this International Standard find application in the routine assay of various types of radioactive waste, packaged in a variety of ways, employing a variety of container sizes, and types. They range from basic techniques, which have been in use for many years, through to state of the art techniques that have been developed because of the increasing variety and forms being assayed and the demands to satisfy increasingly challenging performance criteria.

Where guidance is provided, this is viewed as best current practice and is based on experience of operating quantitative gamma spectrometry measurement systems, within a variety of applications, for the purpose of providing radionuclide identification and activity information.

The objective of this International Standard is to promote a consistent approach to gamma spectrometry measurements made on packages containing radioactive waste.

Guidance for gamma spectrometry measurement of radioactive waste

1 Scope

This International Standard is applicable to gamma radiation measurements on radioactive waste.

Radioactive waste can be found in different forms and exhibit a wide range of characteristics, including the following:

— raw or unconditioned waste, including process waste (filters, resins, control rods, scrap, etc.) and waste from dismantling or decommissioning;

— conditioned waste in various forms and matrices (bitumen, cement, hydraulic binder, etc.);

— very low level (VLLW), low level (LLW), intermediate level (ILW) and high level radioactive waste (HLW);

— different package shapes: cylinders, cubes, parallelepipeds, etc.

Guidance is provided in respect of implementation, calibration, and quality control. The diversity of applications and system realizations (ranging from research to industrial systems, from very low level to high level radioactive waste, from small to large volume packages with different shapes, with different performance requirements and allowable measuring time) renders it impossible to provide specific guidance for all instances; the objective of this International Standard is, therefore, to establish a set of guiding principles. Ultimately, implementation is to be performed by suitably qualified and experienced persons and based on a thorough understanding of the influencing factors, contributing variables and performance requirements of the specific measurement application.

This International Standard assumes that the need for the provision of such a system will have been adequately considered and that its application and performance requirements will have been adequately defined through the use of a structured requirements capture process, such as data quality objectives (DQO).

It is noted that, while outside the scope of this International Standard, many of the principles, measurement methods, and recommended practices discussed here are also equally applicable to gamma measurements of items other than radioactive waste (e.g. bulk food, water, free-standing piles of materials) or to measurements made on radioactive materials contained within non-traditional packages (e.g. in transport containers).

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE   Definitions presented here are confined mainly to those terms not defined in common nuclear material glossaries or whose use is specific to this document. Important key terms are repeated here for the convenience of the reader.

2.1
assay
procedure to determine quantitatively the amount of one or more radionuclides of interest contained in a package

2.2
attenuation
physical process based on interaction between a radiation source and matter placed in the path of the radiation that results in a decrease in the intensity of the emitted radiation

Note 1 to entry: Attenuation experienced in *non-destructive assay* (NDA)(2.27) of waste packages includes *self-attenuation* (2.37) by the radioactive material itself as well as attenuation effects in the *waste matrix* (2.23), internal barrier(s) and external container(s).

2.3
attenuation correction factor
used to correct (compensate) for the effect of attenuation within an NDA measurement equal to the ratio between the un-attenuated and the attenuated radiation flux

Note 1 to entry: After attenuation correction the measured quantity is considered to be representative of the un-attenuated activity of the radioactive substance assayed.

2.4
bias
estimate of a systematic measurement error

2.5
calibration standard
primary standard
designated or widely acknowledged as having the highest metrological qualities and whose value is accepted without reference to other standards of the same quantity

Note 1 to entry: The calibration standard should be physically, radiologically, and chemically similar to the items to be assayed, for which the activity of the radionuclide(s) of interest and all relevant properties to which the measurement technique is sensitive are known with sufficient accuracy.

[SOURCE: www.french-metrology.com]

2.6
calibration
set of operations that establish, under specific conditions, the relationship between values of quantities indicated by a measuring system, or values represented by a material measure or a reference material and the corresponding values realized by Standards

Note 1 to entry: The result of a calibration permits either the assignment of values of measurands to the indications or the determination of indications with respect to indications.

Note 2 to entry: A calibration may also determine other metrological properties such as the effect of influence quantities.

Note 3 to entry: The result of a calibration may be recorded in a document, sometimes called a calibration certificate or a calibration report.

[SOURCE: www.french-metrology.com]

2.7
collimation
method to restrict the field of view of the detector to specific parts of the item to be measured

Note 1 to entry: A shield around the side of the detector that still allows the detector to view the entire item is technically not a collimator. Such shielding does not change the efficiency of the detector due to its presence.

2.8
collimator
device for collimating the radiation beam, usually constructed from highly attenuating material(s) such as tungsten or lead. Collimators can be of parallel wall type or divergent

2.9
collimated (detection) geometry
measurement configuration where only a part of a waste package can contribute to the response of the detection system

Note 1 to entry: The whole activity is measured by scanning the entire package, or by assuming that the part of the package within the detector's field of view during one or more measurements is representative of the entire package.

2.10
compton continuum
continuous pulse amplitude spectrum due to Compton electrons released in a detector

Note 1 to entry: The full-energy peaks are superimposed to this continuum and their "net areas" are determined by subtracting the average Compton level estimated below each peak, as detailed in ISO 11929 for instance.

[SOURCE: IEC 60050-395:2014]

2.11
container
vessel into which the *waste form* (2.41) is placed for handling, transport, storage and/or eventual disposal

Note 1 to entry: Also the outer barrier protecting the waste from external intrusions.

[SOURCE: IAEA Radioactive Waste Management Glossary 2003 Edition]

2.12
coverage factor
although the combined standard deviation is used to express the uncertainty of many measurement results, for some commercial, industrial, and regulatory applications (e.g. when health and safety are concerned), what is often required is a measure of uncertainty that defines an interval about the measurement result within which the value of the measurand can be confidently asserted to lie

Note 1 to entry: The measure of uncertainty intended to meet this requirement is termed expanded uncertainty and is obtained by multiplying the standard deviation by a coverage factor, suggested symbol k. In general, the value of the coverage factor k is chosen on the basis of the desired level of confidence to be associated with the interval within which the true value is supposed to lie.

[SOURCE: http://physics.nist.gov/cuu/Uncertainty/coverage.html]

2.13
data quality objectives process
DQO
seven stage requirements capture process used to determine the type, quantity, and quality of data needed to support a decision

Note 1 to entry: The purpose of this process (published by the US Environmental Protection Agency) is to provide general guidance to organizations on developing data quality criteria and performance specifications for decision making.

2.14
dead time
non-operative time of the detection system during the measurement period

Note 1 to entry: The length of time, directly following an instance of detection, associated with signal processing, during which the system is not able to process further gamma events. This is a system performance parameter which is usually expressed as a percentage of the measurement period. The measured counts would be less than the actual counts due to the dead time and hence needs to be corrected.

2.15
decision threshold
DT
value of the estimator of the measurand, which when exceeded by the result of an actual measurement using a given measurement procedure of a measurand quantifying a physical effect, one decides that the physical effect is present

Note 1 to entry: The decision threshold is defined, such that in cases, where the measurements result, y, exceeds the decision threshold, y^*, the probability that the true value of the measurand is zero is less or equal to a chosen probability, α.

Note 2 to entry: If the result, y, is below the decision threshold, y^*, the result cannot be attributed to the physical effect; nevertheless it cannot be concluded that it is absent.

[SOURCE: ISO 11929:2010, 3.6]

2.16
detection geometry
describe the extent of detector collimation with respect to the item to be measured

Note 1 to entry: Two principle assay configurations are distinguished in this guideline: collimated geometry and open geometry.

2.17
detection limit
DL
smallest true value of the measurand which ensures a specified probability of being detectable by the measurement procedure

Note 1 to entry: With the decision threshold defined above, the detection limit is the smallest true value of the measurand for which the probability of wrongly deciding that the true value of the measurand is zero is equal to a specified value, β, when, in fact, the true value of the measurand is not zero.

[SOURCE: ISO 11929:2010, 3.7]

2.18
emission computed tomography
ECT
NDA method which allows the distribution of nuclide activity to be determined within sections of the waste package

Note 1 to entry: The technique is based upon the measurement spectra from segments of the waste matrix which the detector views through a collimator. In order to obtain accurate results, it is necessary to know the matrix density distribution within the section (or in 3D), typically by *Transmission Computed Tomography* (TCT) (2.38).

Note 2 to entry: ECT is also referred to as *Tomographic Gamma Scanning* (TGS) (2.39).

2.19
full-energy peak
peak of the gamma spectrum corresponding to the complete deposition of the energy of a photon emitted by a radionuclide

Note 1 to entry: No energy loss has occurred by photon interaction in the waste package or by the escape of secondary photons from the detector following the interaction(s) of the primary photon leading to its detection.

2.20
full width at half maximum
FWHM
width of a gamma-ray peak at half of the maximum of the peak distribution

Note 1 to entry: This parameter is used to describe energy resolution. FWHM is often quoted when defining detector performance (e.g. FWHM for a given energy, such as 662 keV). FWHM can be given in energy units (e.g. keV) or in % if normalized to the gamma-ray energy.

2.21
intrinsic detection efficiency
number of counts in the *full-energy peak* (2.19) at a given energy E (net area after subtraction of the Compton continuum and other sources of background in the gamma spectrum) divided by the number of photons at that energy that enter the detector

2.22
live time
difference between the measurement period and the dead-time

2.23
matrix
waste matrix
non-radioactive materials inside a *waste package* (2.29) in which the radioactive substances are dispersed

2.24
measurand
particular quantity subject to measurement

[SOURCE: ISO 11929:2010, 3.2]

2.25
measurement accuracy
closeness of agreement between a measured quantity value and a true quantity value of a measurand

2.26
measurement period
time frame over which the measurement is made

2.27
non-destructive assay
NDA
procedure based on the observation of spontaneous or stimulated nuclear radiation, interpreted to estimate the content of one or more radionuclides in the item which is under investigation, without affecting the physical or chemical form of the material

2.28
open (detection) geometry
measurement configuration where all parts of a *waste package* (2.29) can contribute to the response of the detection system

2.29
package
waste package
product of conditioning that includes the *waste form* (2.41) and any container(s) and internal barriers

[SOURCE: ISO 12749-3:2015, 3.5.2]

2.30
precision
statistical precision
generic term used to describe the dispersion of a set of measured values under reproducible measurement conditions

2.31
radioactive waste
material for which no further use is foreseen that contains or is contaminated with radionuclides

[SOURCE: ISO 12749-3:2015, 3.7.1]

2.32
radioactivity
phenomenon whereby atoms undergo spontaneous random disintegration, usually accompanied by the emission of radiation

[SOURCE: IAEA Radioactive Waste Management Glossary 2003 Edition]

2.33
radionuclide
nucleus (of an atom) that possesses properties of spontaneous disintegration (*radioactivity* (2.32))

Note 1 to entry: Nuclei are distinguished by their mass and atomic number.

[SOURCE: IAEA Radioactive Waste Management Glossary 2003 Edition]

2.34
scanning profile
distribution of recorded system responses as a function of successive scan positions

2.35
segment (gamma) spectrum
emission gamma spectrum collected from only a part of a *waste package* (2.29)

2.36
segmented gamma scanning
SGS
procedure to measure one or more *segment spectra* (2.35) of a waste package

Note 1 to entry: Segmented gamma scanning requires the use of a *collimated detection geometry* (2.9). There are several manifestations of SGS which are currently in use. For this International Standard we distinguish vertical, horizontal and angular scanning, see Figure 3, which can be combined or used partly (in practice SGS usually refers to the combination of vertical scanning and continuous rotation).

— **vertical scanning** [see Figure 3 a)] consists in acquiring vertically segmented gamma spectra representative of stacked slices of the package. The mechanical movement can be step-by-step, with an acquisition for each slice, or continuous with a time-segmented acquisition (mechanics is simpler and measurement time is shorter, but interpretation is more complex). Vertical scanning is most commonly used in combination with continuous rotation.

— **horizontal scanning** [see Figure 3 b)], is most commonly used in combination with angular and vertical scanning for TGS, and also for objects without rotational symmetry in combination with vertical scanning.

— **angular scanning** [see Figure 3 c)], is rarely used alone but as part of TGS systems. This can be functionally accomplished with a single detector or multiple detectors to limit acquisition time (as shown), and with step rotation or continuous rotation with timely segmented acquisition.

2.37
self-attenuation
self-absorption
attenuation of the gamma radiation in a nuclear material itself (like Pu or U)

Note 1 to entry: This effect is here distinguished from the attenuation of the gamma radiation in nonnuclear materials like the waste matrix, internal shields, container, external shields, collimators, etc.

2.38
transmission computed tomography
TCT
gamma or X-ray transmission technique to determine the matrix density distribution within sections of the waste package, by angular and horizontal scanning, as for ECT and in 3D with an additional vertical scanning

Note 1 to entry: 3D densitometry allows more accurate corrections for attenuation of gamma radiation within non-uniform matrices.

Note 2 to entry: Both in ECT and TCT, 2D sections can be reconstructed by angular and horizontal scanning, and the complete 3D information can be obtained by superimposing the slices vertically or by performing a continuous helical scan.

2.39
tomographic gamma scanning
TGS
typically a combination of emission computed tomography (ECT) and transmission computed tomography (TCT)

2.40
total detection efficiency
number of counts in the full-energy peak (net area) per photon of energy (E) emitted in the waste package

2.41
waste form
physical and chemical form after treatment or conditioning prior to packaging and which is a component of the *waste package* (2.29)

[SOURCE: ISO 12749-3:2015, 3.7.6]

3   Application

3.1   General

Measurement of gamma radiation emissions provides a non-destructive method of establishing the inventory of gamma-emitting radionuclides inside a waste package.

Gamma measurements can be performed using relatively unsophisticated techniques (such as Open Detector Geometry, see 4.2) and measurement procedures where the waste and matrix are well understood or where source and matrix can be considered to be uniformly distributed (such that a simple form of measurement can provide a representative result).

Alternatively, there may be little or no knowledge of the sources present, the activity distributions, the matrix composition or homogeneity; in these cases, it is often necessary to consider more complex techniques (such as Collimated Detector Geometries, see 4.3).

Depending on gamma irradiation level, shields and/or a collimated geometry may also be necessary to keep the detector and acquisition system count rates within operating limits.

3.2   Typical applications

Gamma radiation measurement systems are currently employed in a variety of radioactive waste package measurement applications, such as the following:

— inventory assignment ahead of waste processing, storage or transport;

— inventory verification ahead of waste processing, storage or transport;

— waste inspection during interim storage or final disposal,

— quality checking of waste conditioning processes;

— free release measurements.

NOTE   Gamma spectroscopy is used in many applications beyond the scope of this International Standard, such as process control, radioactivity assessment of environmental media (soil, vegetation, water, etc.), characterization of post-accident clean-up debris, bulk material measurements, etc. The same principles and good practices may often apply in these fields.

Radionuclides to be detected by this method must emit gamma radiation with sufficient intensity and energy to penetrate the surrounding materials and escape the containment before they can be measured.

The useful energy range is dependent on a number of factors such as the composition and distribution of the matrix; the source position and/or source distribution inside the package and the type and dimension of the container. For most applications, the gamma radiation energies of interest in waste assay lie within the range from a few tens keV to 3 MeV. The energy of the gamma radiations that may be successfully detected in different applications and under different conditions may have a reduced range.

4   Measurement equipment

4.1   General

A number of different types of system are currently used to perform gamma radiation measurements on packages containing radioactive waste. It is not the intention of this International Standard to focus on the specific design of any type of system. The objective is to concentrate on the general aspects relevant for implementation in specific measurement configurations and for performance assessment. Some examples of measurement systems, currently in use in assay applications are given in Annex A. The contents of Annex A are provided for information only; they should not be considered to be mandatory; neither should they be considered exhaustive.

In instances where measurements are made on packages containing radioactive waste, the objective of the measurement is generally to enable the operator to establish the activity of radionuclides of interest within the package, within the context of the application. The information required can vary from application to application. For instance, the information required for criticality control within the confines of the site of origin may be a sub-set of the total radionuclide inventory of the package including only fissile isotopes (e.g. ^{235}U, ^{239}Pu, ^{241}Pu); a more complete radionuclide inventory may be required to enable transport through the public domain (e.g. a number of beta and alpha activities) and this may be different from the information required for ultimate disposal (whole inventory including for instance long-lives isotopes). Equally, the performance requirements of the system may vary from application to application. However, in all instances, the functionality and performance requirements for the system shall be established prior to development of the system.

This Clause describes the basic characteristics of systems currently employed to perform gamma radiation measurements on packages containing nuclear waste. Systems currently in use range from simple systems (incorporating a single, uncollimated detector) through to complex systems (incorporating multiple detectors, advanced scanning techniques, and state of the art counting equipment).

For waste packages with revolution symmetry, a common feature of most gamma measurement systems is a turntable to rotate the package during the measurement. Box-shaped packages are commonly measured several times from multiple locations and sides. These multiple measurements and rotation are primarily performed to average variations in system response from non-homogeneous waste.

Measurement systems can be broadly classified according to the detection geometry and measurement procedure as

— open detector geometry, and

— collimated detector geometry.

Gamma spectrometry systems may use single detectors or multiple detectors, to increase system throughput. Throughout this International Standard, reference will only be given to single-detector instruments because the performance characteristics of both types show no principal differences despite the superior efficiency of multiple-detector systems.

4.2   Open detector geometry

The basic configuration for this type of measurement involves one or more detectors, which are located in a fixed position relative to the waste package. The open geometry configuration is set-up so that all

parts of a package contribute to the response of the detector (see Figure 1). The package may be rotated during the measurement or multiple measurements made from different directions can be averaged to reduce the measurement uncertainty in case of non-uniform radioactivity in the package. The decision to rotate the package or to perform multiple view acquisitions depends on the heterogeneity of the waste (materials and activity) and its impact on uncertainty. The choice may be the result of a trade-off between uncertainty objectives and practical limitations (e.g. for cylindrical packages, rotation is the most common practice, while for cubic or parallelepipedic packages each face is generally measured).

Systems based on this type of configuration have the advantage of simpler hardware and generally higher detection efficiency compared to systems that employ collimated geometry and a scanning system. Practical experience is that open geometry measurement systems usually yield significantly lower detection limits; however, the results from this method are generally more sensitive to the distribution of activity and variations in the density of the waste matrix.

If waste material and activity distributions are known to be quite homogeneous, a gamma transmission technique can be used to correct for matrix attenuation (density and composition effects). The clause of the waste interrogated by the transmission source shall be as representative as possible of the entire volume. Representation can be improved by using multiple external transmission sources, placed so as to interrogate the upper portion, at half height, and the bottom portion of the package; alternatively, a continuous vertical scan can be implemented (however, this complicates both hardware and software). The package may be rotated during the transmission measurement; alternatively, multiple measurements can be made from different directions, and averaged.

Key
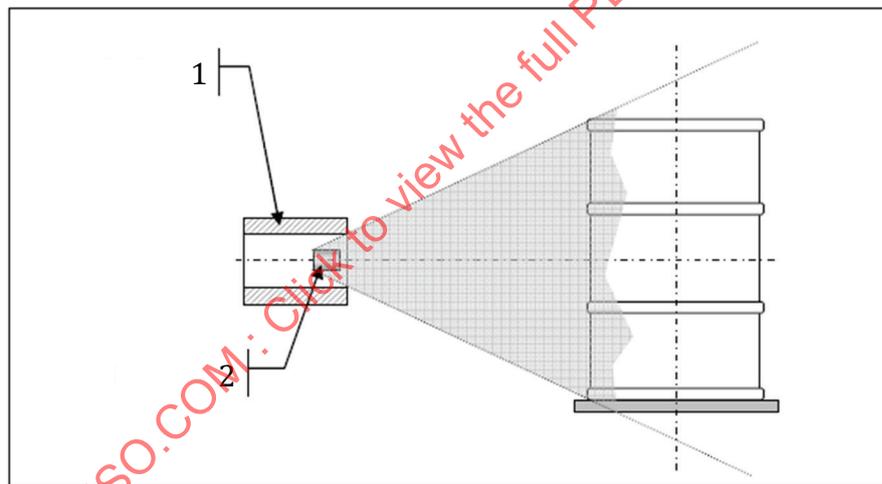
1   shielding

2   detector

NOTE   A background reduction shield, surrounding the side and sometimes the back of the detector is desirable. However, this is to be designed to keep the entire package within the field of view of the detector.

Figure 1 — Open detector geometry (transmission correction source not shown)

Open detector geometry is applicable when variations in activity distribution within the package and other waste characteristics (in particular density distribution) will not result in punitively large measurement uncertainty.[1]

If the waste is heterogeneous, the measurement uncertainty may be punitively large, even with package rotation or multiple measurements made from different directions, and with gamma transmission

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1)   Rotating the package during acquisition allows reducing the uncertainty due to radial heterogeneity.

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measurement. In this instance, collimated detector geometry coupled with techniques like segmented gamma scanning (SGS) or tomographic gamma scanning (TGS) discussed below may be more appropriate.

NOTE   Measurement uncertainty is discussed in 6.4.

4.3   Collimated detector geometry

Collimation may be used to restrict the field of view of the detector, or detectors, to specific parts of the waste package (see Figure 2). It restricts the size and angle of the beam of radiation falling on the detector.

Key
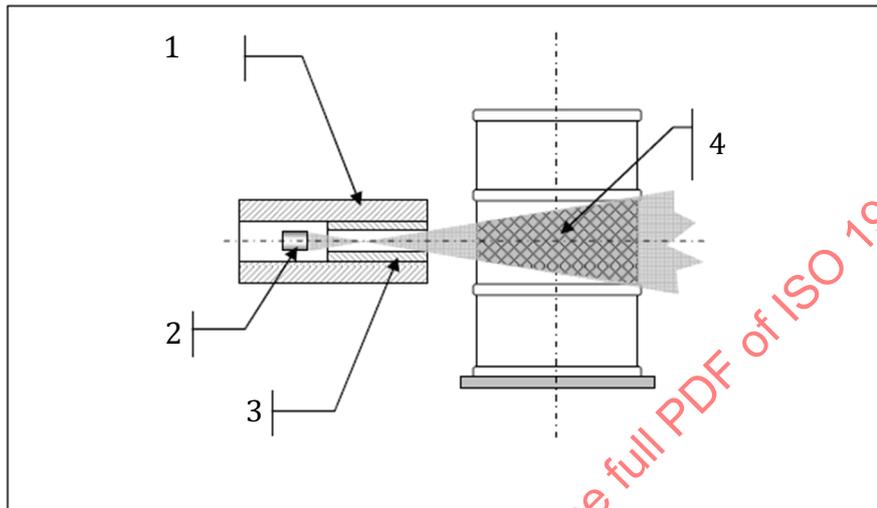
1   shielding
2   detector
3   collimator
4   measured volume

Figure 2 — Collimated detector geometry

This technique is an essential component of SGS,[2] which requires the field of view of the detector to be restricted such that the spectrum collected is the result of the activity contributions from species present in specific portions (segments) of the package, rather than the package as a whole.

Since collimated geometries only view a small portion of the package, they are almost always combined with some other method to obtain a representative view of the full package. These methods include horizontal scanning, vertical scanning, angular scanning, or continuous rotation and multiple detectors in fixed positions. Combinations of these methods are frequently used.

The geometry of the collimator is a function of the type of scanning employed and the positional resolution required.

There are several manifestations of SGS2 which are currently in use:

— vertical scanning [see Figure 3 a)], consists of acquiring vertically segmented gamma spectra, representative of stacked slices of the package. The mechanical movement can be step-by-step, with an acquisition for each slice, or continuous with a time-segmented acquisition (mechanics are simpler and measurement time is shorter, but interpretation is more complex). Vertical scanning is most commonly used in combination with continuous rotation2; which are also typically coupled to a gamma transmission measurement, to correct for (variable) matrix attenuation;

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2)   In practice, the term "SGS" usually refers to the simple combination of vertical scanning and continuous rotation (contrary to the step rotation of angular scanning) of the package. We use here this term in its general sense.

— horizontal scanning [see Figure 3 b)], either continual or step-wise, is most commonly in combination with rotation and vertical scanning for TGS; it is also commonly used for objects without rotational symmetry such as box-shaped containers;

— angular scanning [see Figure 3 c)], rarely used alone but as part of TGS systems. This can be functionally accomplished with a single detector or multiple detectors to limit acquisition time (as shown), and with step rotation or continuous rotation with timely segmented acquisition.

The uncertainty due to heterogeneity in rotationally symmetric objects can be reduced by continuously rotating the package. The uncertainty can also be reduced by multiple measurements, made at different locations on the package. Identification of the presence of hot spots, or the confirmation of homogeneity, is possible by observing the instantaneous count rate during the rotation or at the separate measurement locations.

The major advantage of an SGS, as compared to an open detector geometry measurement of a rotating drum, is when the segment emission data are combined with a transmission measurement of the segment, as this allows correction for different vertical layers of density in the item. This is especially important where the radionuclide emits low energy gamma radiation, and where there are medium-high atomic number materials in the waste (e.g. iron, lead, uranium).

The scanning geometries described above can be utilized in combination, depending on waste heterogeneity and required measurement uncertainty.

When the rotational segments fully cover 360° plus when there is both lateral and vertical scanning, and when the emission and the transmission of each small segment are determined separately, then 3D coupled emission-transmission computed tomography (ECT-TCT also referred to as TGS) is possible. This reduces the measurement uncertainty when there are spatially distinct areas of both radioactivity and attenuation.

Tomography has the benefit of greatly reducing measurement uncertainty because the location of the radioactivity and matrix non-homogeneities can be established more precisely and therefore more precise efficiency corrections can be applied.

However, this technology comes at an increased level of complexity, cost and measurement time.

a) Vertical scanning of a drum

b) Horizontal scanning of a drum

c) Angular scanning of a drum
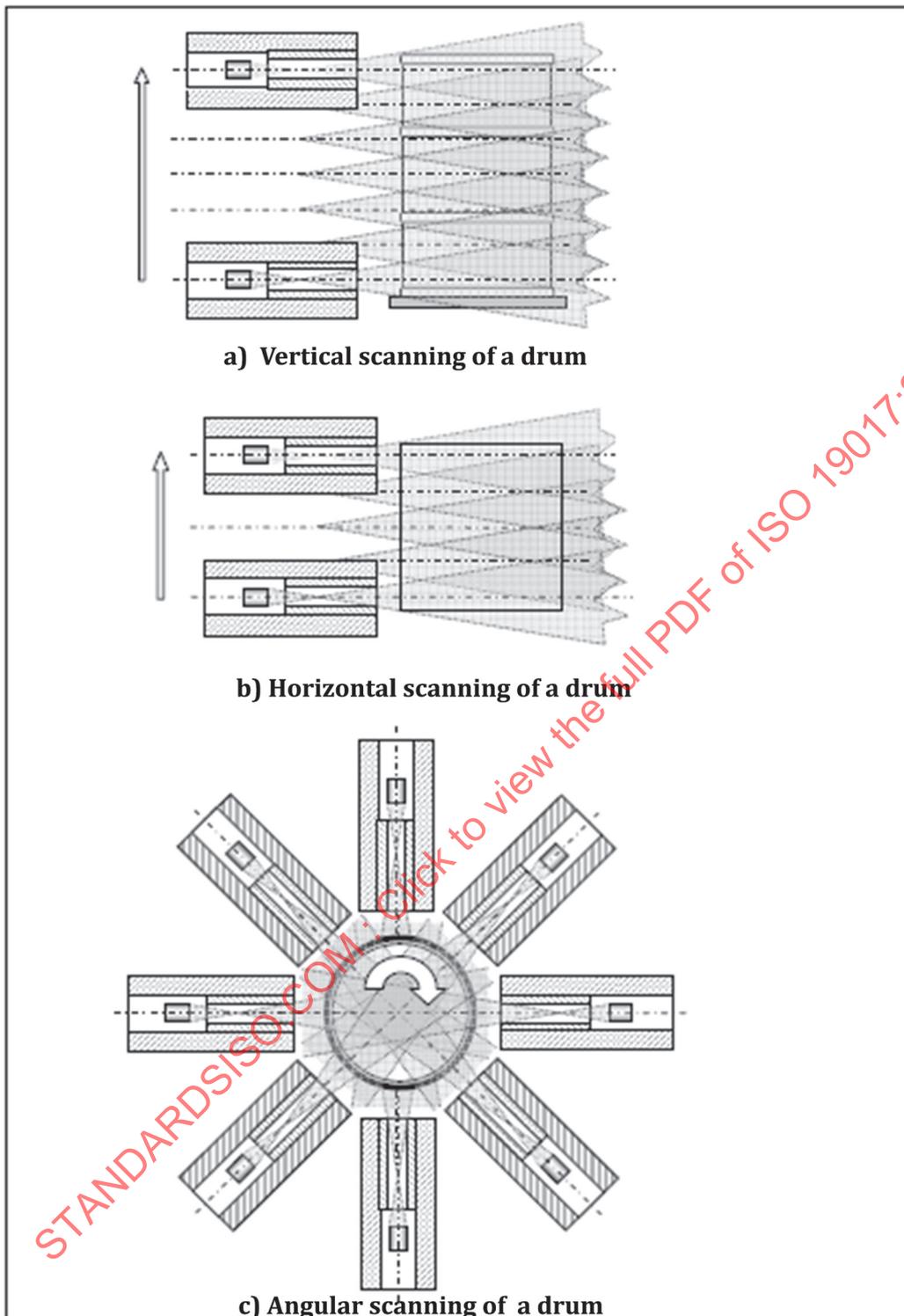
Figure 3 — Alternative approaches to segmented gamma scanning commonly in use

NOTE 1   Figures 1, 2, 3 a) and 3 c) depict typical scanning configurations for waste packages within cylindrical containers (such as drums). There may be differences in detector numbers and configurations employed for the assay of waste packaged within cuboid containment, like the box of Figure 3 b). Size and shape may be preclusive of using a turntable; so configurations which employ detectors which view each face may be employed (this alternative configuration is not represented in the diagrams above).

NOTE 2   Scanning may be achieved either by moving the waste package, or by a combination of detector and waste package movements. For instance, in many SGS systems the drum is in continuous rotation, while the detector/collimator assembly scans the drum vertically. In compact TGS systems, the drum is generally in translation and rotation while the detector scans the package vertically. Additionally, an array of collimated detectors can be used to reduce measurement time, and possibly to avoid the necessity for some mechanical movements (e.g. a vertical array covering the whole height of the package can be used as an alternative to a mechanical device to raise and lower the detector).

The acquisition of segment spectra can be performed continuously, as the waste package is rotated, or at a predefined, discrete set of positions. The activity of the package, as a whole, is reconstructed by summing the results of the individual segments, rather than as the result of a single measurement. In this way, a higher degree of precision can be obtained since each individual spectrum need only be considered representative of the volume of the segment from which it was acquired, rather than volume of the entire waste package.

The SGS measurement procedure can be combined with a segment specific determination of *attenuation factors* (i.e. transmission measurement). Therefore, SGS offers the possibility of segment specific attenuation correction. This usually results in the measurement procedure being less sensitive to non-uniform activity and density distributions, than open collimation detection geometry.

Segment specific spectra can be used to check the adherence of the waste package to underlying assumptions, such as uniformity of source and matrix distribution. This facilitates the definition and evaluation of criteria for conformity checking, which is especially important for waste forms which have not originated from well-defined processes.

Collimated SGS measurements are most useful for waste packages where the matrix effects are known to be or suspected of being, heterogeneous and where the measurement uncertainty obtained using simpler techniques is unacceptably large.

In cases where the waste can be considered to be homogeneous (e.g. for processed waste forms), where there is little benefit in using segmented techniques, it is possible to implement collimated detector geometry as a means of restricting the count rate seen by the detector, where this would otherwise lead to saturation of the signal processing electronics (possibly leading to excessive dead time).

4.4   Components of gamma measurement system

Within the scope of this International Standard only the core components of gamma spectrometry measurement systems are mentioned. Typical systems incorporate the following:

— mechanical equipment to provide positioning capabilities for the detector and/or waste package, as well as tools for container loading;

— radiation detection equipment;

— data acquisition and analysis unit;

— electrical control;

— additional equipment.

4.4.1   Mechanical equipment

Mechanical equipment generally comprises the following:

— structures to hold the detector and collimator;

— mechanical system to accommodate the waste package and perform any needed movement (continuous or step-by-step rotation, vertical and horizontal scanning);

— manual or automated waste package handling equipment;

— mechanical system for positioning the detector relative to the waste package (or vice versa).

4.4.2 Radiation detection equipment

Radiation detection equipment is application specific, but generally comprises the following:

— gamma detector: high resolution, low resolution and medium resolution detectors are available, such as the high purity germanium (HPGe) and cadmium-zinc-telluride (CdZnTe or CZT) semiconductors, or the thallium activated sodium iodide NaI(Tl) or the cerium-activated lanthanum bromide $LaBr_3(Ce)$ scintillators;

— cooling equipment (for high resolution HPGe detectors);

— collimator/shield configuration (as appropriate).

4.4.3 Data acquisition and analysis unit

Data acquisition processing functions are application specific, but generally comprise the following:

— high voltage power supply;

— photomultiplier (scintillation detector only);

— pre-amplifier (usually close-coupled to the detector);

— gamma spectroscopy system, consisting of signal conditioning and digitizing electronics, typically consisting of shaping main-amplifier, analog-to-digital converter, and multi-channel buffer and storage. These are commonly integrated today into a single unit, but can also be created from independent modules;

— hardware to control operation of the gamma spectroscopy system;

— software to perform spectral evaluation;

— user interface.

4.4.4 Electrical control

Electrical control comprises all components necessary to control and synchronize the mechanical equipment with the measurement and data acquisition unit in combination with the data acquisition system. Control is also performed to ensure safe operation of the complete system.

4.4.5 Additional equipment

Additional equipment may include (but not be limited to) the following:

— weighing unit (balance) for the waste package, with an appropriate range;

— dose rate meter(s);

— power input conditioning units.

5 Calibration

5.1 General

The objective of calibration is to establish the relationship between the radionuclide activity of a known source (or set of sources) and the measured values of the source (or sources), i.e. the response of the system.

Calibration is an essential part of any quality and metrology assurance programme. The calibration procedure, for a gamma measurement system, generally comprises the following:

— peak energy and peak shape calibration;

— efficiency calibration;

— calibration of additional instruments.

Calibration is dependent on the source of radiation and the geometrical condition of the target waste package. Calibration sources should cover the energy range of the gamma radiation expected in the waste package. Efficiency calibration should take into account the measurement geometry and the density and composition of the waste materials (matrix), as well as the distribution of radioisotopes within the waste (homogeneous or not). In many waste packages, both the matrix and radioactivity distributions are heterogeneous, which may be taken into account either in the calibration procedure, for instance, by measuring or calculating efficiencies covering all the expected situations, or by using specific correction techniques based on segmented gamma transmission and detection measurements (see 5.3 and 5.4).

5.2 Peak energy and shape calibration of the gamma spectrometry system

This includes the following:

— energy calibration (establishing the relationship between the individual gamma energies and the channels of the analyser), which is generally linear with high purity germanium (HPGe) detectors, but shows nonlinearity, at low energies, with inorganic scintillators like NaI(Tl);

— peak shape calibration (including energy resolution). Energy resolution determines the ability to discriminate between individual peaks with close energy in the gamma spectrum. Energy resolution is essentially a function of the type of detector employed: high resolution can be achieved with HPGe, resolution is poorer in NaI(Tl), and intermediate in $LaBr_3(Ce)$.

NOTE   Energy resolution is generally quoted in terms of full width half maximum, FWHM(E) in keV or relative resolution, FWHM(E)/E in %, at a specific energy E.

5.3 Efficiency calibration of the gamma spectrometry system

The preferred method of efficiency calibration is to construct a physically, chemically, and radiologically equivalent (size, density, chemical composition, radionuclides) surrogate of the radioactive waste sample to be measured, using well-known (traceable) radioactive sources of the appropriate energy. An energy versus efficiency curve is constructed which is then applied to the samples to be measured. While this method is highly desirable, it is rarely practical for packaged waste measurements. Constructing physical radioactive calibration standards for waste packages is expensive, takes time, requires the appropriate radioactive materials licenses, has some degree of radiological hazard, and creates a radioactive materials storage and disposal issue.

The following alternatives are in common use for waste assay systems.

— Filling a physically identical container with radioactive material that is representative of the waste package contents, extracting representative samples, and measuring the radioactivity in those samples, in a traceable manner; thus establishing a calibration for that type of waste. If the waste matrix to be measured is homogeneous and easily sampled, this can be a very accurate calibration method and quite practical. However, it is not easy to extend the calibration to other matrices, densities, or gamma radiation energies.

— Constructing a simulated waste package, using non-radioactive material(s) that is/are representative of the attenuation characteristics of the matrix to be measured, and inserting many point or line sources into the matrix, in a controlled manner. This has the advantage of only needing physically small sealed radioactive sources, which can be used in many different simulations. Common examples are the use of line sources in boxes or rotating drums, and constructing complex shapes

out of a large number of small boxes with a point source in each of the boxes. The disadvantage is that the efficiency is biased low, as compared to a true homogeneous source, and that appropriate matrix materials are not always available. Numerical simulations could be used to derive a correction factor for the bias.

— Using a single point source to determine the intrinsic efficiency of the detector, and then applying a series of mathematical correction factors to compute the efficiency for the waste package. This is the traditional SGS calibration method. Correction factors for various geometric and physical parameters to be applied include: container wall attenuation, matrix correction, detector-container geometry corrections, collimator correction, and self-absorption in nuclear materials corrections. An illustration of this method is included below.

— Using mathematical modelling to create realistic mathematical simulations of the waste package, and then computing the emission from that package and full-energy interaction probability with the detector. Compared to simulations constructed using radioactive sources, these can be quicker, lower cost, and have zero storage and disposal costs. As with the other methods, the quality of the results is determined by how well the radiosensitive elements of the package are known (matrix, density, radionuclide, source location, etc.). In addition, the mathematical description of the detector must be well known, and is best determined by reference to traceable sources. When performed correctly, with good methods, mathematical calibrations can have equal or better accuracy than most other large volume calibration methods.

The example below illustrates the method of using a single point source to determine the intrinsic efficiency of the detector, and then establishing a series of mathematical correction factors, relating to the system response. These correction factors can then be applied to measurements made on "real" waste packages, to quantify the activity (A) of a certain radionuclide (i), emitting gamma radiation of energy (E), with a probability of emission (ρ); given in Formula (1):

$$A_{i,E} = (N_{i,E} - B_{i,E}) \cdot K_i \frac{1}{\rho_{i,E}} \cdot \frac{1}{\varepsilon_E} \cdot f_{i,E}(k_1, k_2, ...) \tag{1}$$

where

$A_{i,E}$ the activity (Bq) of radionuclide i, at energy E;

$N_{i,E}$ the net area of the full-energy peak of radionuclide i, at energy E, **present in the spectrum of the waste package**, (number of counts per second expressed in s^{-1}); the net area is obtained by subtracting the Compton Continuum under the peak (see 6.2);

$B_{i,E}$ the net area of the full-energy peak of radionuclide i, at energy E, **present within the background spectrum**, (number of counts per second expressed in s^{-1}); this is best performed with a package of the same size and mass but without the analytes of interest, as the package can reduce the empty shield background; the net area is obtained by subtracting the Compton Continuum under the peak (see 6.2);

NOTE In order to avoid this situation, where a peak is present in the background spectrum at the same energy as the radionuclide of interest in the package, appropriate shields can be implemented around the package, to restrict the field of view of the gamma detector to the measured package.

$\rho_{i,E}$ is the probability of photon emission by radionuclide i at energy E (the fractional number of photons emitted per radioactive disintegration), as for instance reported in Reference [13];

ε_E is the "intrinsic detection efficiency" at energy E, i.e. (the fractional number of counts detected in the full-energy peak net area per photon of energy E entering the detector);

K_i is the correction factor for the radioactive decay during the measurement for radionuclide i; it is defined as $A_0 = K_i \times \bar{A}$, where A_0 and $\bar{A}$ are respectively the activity at the beginning of the measurement and average during the acquisition; K_i is given by:

$$K_i = \frac{\dfrac{t_{\text{acquisition}}}{T_i} \times \ln 2}{1 - \exp(\dfrac{t_{\text{acquisition}}}{T_i} \times \ln 2)}$$

where $t_{\text{acquisition}}$ is the acquisition time and T_i is the half-life of radionuclide i. For most practical applications, K_i is almost equal to 1 because acquisition time is generally very small compared to T_i and can be disregarded;

$f_{i,E}(k_1, k_2, \ldots)$ is the correction factor for variations in the geometric and physical k_n parameters, such as:

k_1 is the materials and dimensions of the container;

k_2 is the detector-container relative position (distance, angles);

k_3 is the collimation effects;

k_4 is the position of the waste materials within the container (matrix geometry);

k_5 is the chemical and physical characteristics of the matrix (material type, density);

k_6 is the localization of radioactive materials in the container (e.g. homogeneous, hot spots, surface or bulk contamination of a part or of the entire waste materials) leading to combined matrix and localisation attenuation effects;

k_7 is the chemical and physical characteristics of the radioactive material (e.g. non-homogeneous sources significantly different from the matrix in density and atomic number Z, which could lead to self-absorption in the radioactive material itself that is not corrected by the matrix density correction).

The calibration should span the whole range of activities (A), the whole range of energies (E) and the dynamic ranges of all the parameters k.

The radioactive sources used in the calibration should be, whenever possible traceable to national or international standards.

The calibration of the measurement device consists of determining the product $\dfrac{1}{\varepsilon_E} \cdot f_{i,E}(k_1, k_2, \ldots)$

which represents the "total detection efficiency" (number of counts in the full-energy net peak area per photon of energy E emitted in the waste package).

This can either be determined

— by considering ε_E and $f_{i,E}(k_1, k_2, \ldots)$ separately; whereby ε_E can be determined through the measurement of reference sources; $f_{i,E}(k_1, k_2, \ldots)$ being determined through calculation or simulation;

NOTE   The calibration geometry (point or linear reference sources) may differ from the measurement geometry (distribution of radionuclides in the waste package), leading to different incoming angles for the photons entering the detector. This can be taken into account by a correction factor, either in ε_E or in $f_{i,E}(k_1, k_2, \ldots)$.

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— by considering the entire term, as a single entity which can be evaluated through measurements of reference standards, contained within representative packages, or by the use of numerical simulations.

In both cases, package(s) which is (are) measured or simulated shall be representative of the package(s) to be characterized in terms of geometry, activity, distribution of the radioactive materials, and matrix characteristics (k_n parameters).

5.4 Attenuation correction techniques

Some calibration methods create the full efficiency directly (e.g. numerical simulations, full container surrogates). Where these preferred methods cannot be used, partial calibrations can be used, and correction factors applied. A major correction factor is due to attenuation. Methods routinely used to perform attenuation correction include the following:

— *correction based upon average density*: this is the simplest attenuation correction technique and involves the determination (or estimation) of the average density of the waste matrix; this density determination can then be used to determine the attenuation at the energies of interest.

   The process of determining the density usually involves measuring the weight of the package (the dimensions of the package are generally known); the volume of the matrix is often estimated, based on an assumed filling level. Some systems currently in use obtain fill information using radiography techniques. Often, determination of the waste volume is not the prime purpose for the supply of radiographic systems, but where present for other purposes, it is considered good practice to make use of the available information;

— *correction based upon differential peak absorption*: if gamma radiation with sufficiently different energies can be detected from species that can be considered co-located, then an attenuation correction factor can be derived. This is an especially useful method when there is self-attenuation in small lumps of nuclear material (e.g. U or Pu), and where the density and Z of these small lumps are not representative of the matrix. In this instance, the relative attenuation of gamma rays at different energies can provide useful information about the attenuation properties of the nuclear materials being assayed;

— *correction based upon transmission measurements or imaging techniques*: an external gamma (or X-ray) radiation source is used to measure the transmission at one or more energies, at one or more locations across the matrix. This attenuation correction technique is often used by SGS systems and TGS systems.

6 Data evaluation

6.1 Data processing steps

In order to determine the inventory of gamma emitting radionuclides with their corresponding activities or the detection limits in case of non-detected gamma rays, the following sequence of procedures is carried out:

— calculation of the full-energy net peak areas;

— calculation of net peak count rates ($N_{i,E}$ — $B_{i,E}$) in 5.3;

— calculation of gamma activity inventory of the waste package;

— calculation of measurement uncertainty;

— calculation of detection limits for non-detected gamma rays.

6.2 Calculation of net peak count rates

The following corrections should be considered:

— background correction: subtraction of the Compton continuum. This is applicable to both the waste package spectrum and the background spectrum (present as a result of natural radioactivity, cosmic radiation, radioactive materials stored in the vicinity of the measurement system, etc.);
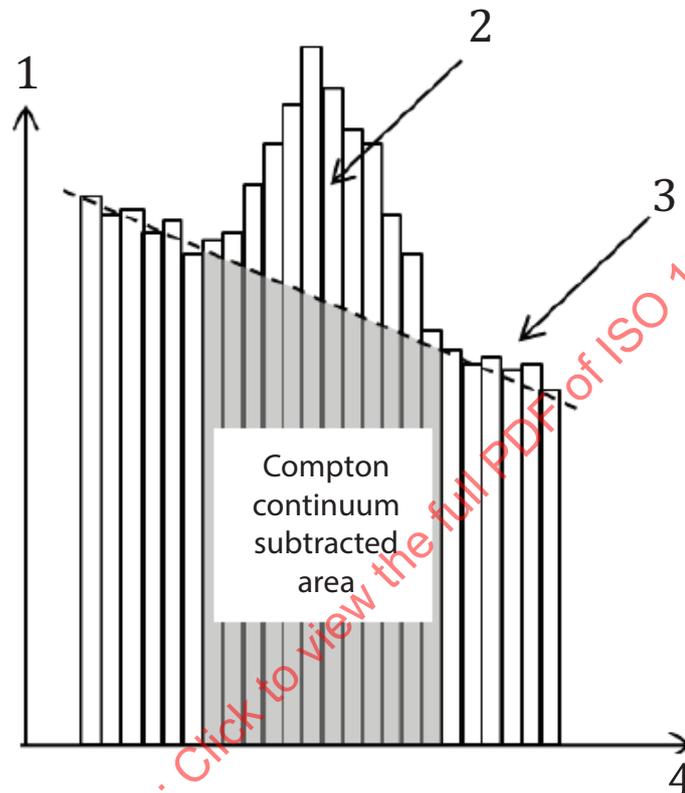
Key

1   counts (or count rate)
2   net peak area
3   compton continuum
4   channels

Figure 4 — Example of Compton continuum subtraction based on a simple trapeze extrapolation of the Compton background in the left and right channels

— subtraction of the "peaked background" (i.e. $B_{i,E}$ in 5.3). It shall be warned, however, that if interfering peaks from outside radioactive sources are present at the same energy as those from sources in the package, the simple subtraction of $B_{i,E}$ may be incorrect depending upon the location of the outside source and the size/shape/density of the package and its contents. The exact quantity to subtract is generally not well controlled and, as far as possible, peaks interfered by background peaks should be avoided, or background peaks eliminated by changing the measurement geometry, shielding, environment;

— correction for interference (between radionuclides present in the waste package). If the gamma radiation emitted by different radionuclides have energies which are located too close to one another within the gamma spectrum, resolution of the respective net peak areas may not be possible; the effect of this is normally an over-estimate of one or both of the radionuclides involved: corrections may be needed;

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— dead-time correction (correction for high count rates, which may otherwise result in "missed counts" are normally made within the counting electronics). Excessive dead time can result in under-reporting; it is normal practice to place limits on the amount of dead time that is acceptable. A reference radioactive source or a pulse generator can be used to monitor count losses and dead time corrections by the electronics;

— coincidence summing correction (i.e. efficiency loss in a peak of energy E due to the simultaneous detection of two gamma rays emitted in cascade during a single disintegration; usually this correction only needs to be considered in applications where the source and detector are very close and where the package is very small).

6.3 Calculation of gamma activity inventory of the waste package

Ideally the gamma activity inventory of the waste package would be returned by the system, according to the calibration function in Formula (2):

$$A_{i,E} = (N_{i,E} - B_{i,E}) \cdot K_i \frac{1}{\rho_{i,E}} \cdot \frac{1}{\varepsilon_E} \cdot f_{i,E}(k_1, k_2, ...) \tag{2}$$

However, it is usually necessary to perform further processing and manipulation of the data before the package inventory can be declared.

For example:

It is usually desirable to combine the information in several peaks, originating from a single radionuclide to improve the quality of the results, both in terms of counting statistics and in terms of efficiency checking at different energies (i.e. validity of efficiency calibration).

It may be necessary to convert from activity to concentration or to mass of the radioactivity.

For segmented systems it is necessary to combine the information contained in several spectra to extract the activity for the whole waste package. In this instance specific consideration (which is ideally obtained by experiment, but where this is not practical, may be obtained by simulation) needs to be given to compensating for overlaps between spectra (to avoid over-estimating the package inventory due to double-counting).

Different applications (summed spectrum, combined activities of individual spectra...) inevitably require specific calibrations and different data manipulations to be performed. For this reason, it is outside the scope of this International Standard to provide further guidance on either data manipulation or data usage.

6.4 Calculation of measurement uncertainty

The objective of any measurement is to establish the value of some parameter of interest. Given the statistical nature of radioactive decay, the measurement result can only ever be an estimate of the value of that parameter; hence, the measurement result is incomplete without a statement of uncertainty.

When considering measurement uncertainty, it is reasonable to determine the range of all likely errors and to estimate the risk that the actual error could be outside the range.

Hence, it is apparent that two statements are needed to quantify measurement uncertainty. The first of these (known as the "coverage interval") provides the range within which we believe the actual error lies. The second (known as "confidence level") provides a statement of how sure we are that the "actual" measurement error lies within the defined interval.

For variables that are distributed according to a Gaussian (Normal) distribution, there is a fixed relationship between standard deviation and confidence, quoted as a percentage. However, it is important to note that when elements of the uncertainty are caused by radiation attenuation, the distribution is typically not Gaussian, even non-symmetric and non-centred, as for instance a log-normal distribution.

In practice, measurement uncertainty comprises contributions from a number of sources. Estimation of measurement uncertainty requires the analyst to assess the effects (on the result) of all significant sources of uncertainty. Sources of uncertainty typically considered when assembling error budgets for gamma measurement systems are the following:

— number of radiation induced events detected;

— number of background radiation events detected;

— calibration factor;

— correction factors

— measurement geometry (e.g. variation in container location);

— container size and wall thickness;

— fill height of the sample inside the container (frequently not visible);

— activity distribution;

— matrix effects (e.g. matrix composition, density distribution within matrix);

— self-absorption in the radioactive materials (if not already taken into account in the matrix effects);

— isotopic composition;

— dead time effects;

— precision of nuclear data.

Some effects are caused by irregular events that, although they are potential sources of measurement error, are not generally considered when estimating uncertainty. Instead, we seek to minimize (or ideally avoid) occurrence by the adoption of good practice. These includes the following:

— operator error;

— instrumental malfunction or drift;

— software errors and faults.

ISO has published ISO/IEC Guide 98-3:2008. Among other things it explains how to combine the various partial estimates of uncertainty. This does not preclude the use of other models or methodologies.

For most waste measurement systems, total measurement uncertainty is dominated by lack of precise knowledge about the distributions of activity and density, within the package. A convenient approach to understanding the error distribution is to use computer modelling. This approach usually produces a data set that can be analysed using statistical techniques. A common example is the use of numerical simulations to perform probabilistic uncertainty analyses. Here a combined uncertainty of the modelled items is determined, along with the distribution function of the uncertainty. This would typically include the uncertainty from the efficiency and various correction factors.

6.5 Calculation of detection limit

The random, fluctuating nature of both source and background gives rise to measurement parameters that are termed "detection limit" (DL) and "decision threshold" (DT).

Within the field of gamma radiation measurement, the detection limit may be defined as the minimum activity of a radionuclide that can be detected with a good confidence (the probability β to declare that it is not detected is low, see Figure 5, for a given measurement configuration, given the probability α to falsely declare its presence when nothing is present, which is also low). The DL can vary from radionuclide to radionuclide and from measurement to measurement; its value is usually calculated within standard gamma spectroscopy software.

The DL is an important performance parameter to compare one system to another system, The DL often dictates the design of the system, since it is sensitive to choice of detector, detector configuration, quantity and choice of shielding material, etc. Therefore, it is usually necessary to calculate it, at an early stage of system design, against a set of agreed parameters to gain confidence that the "as built" system will deliver the performance requirement.

Whether calculated during design or at the time of measurement, the method of calculation is the same.

NOTE   Determination of DL is application specific. For this reason, only the general principles are described within this International Standard. Further guidance can be found in ISO/IEC Guide 98-3 and ISO 11929.

In order to determine the DL, it is first necessary to establish a parameter termed the "decision threshold" (DT).

The Decision Threshold is the threshold above which the net signal will be interpreted as being due to the presence of some radionuclide within the waste package, rather than being due to random fluctuations of the background. For a given gamma peak of a radionuclide, the threshold is set such that an acceptably small portion of the signal distribution lies above it when the radionuclide is not present in the waste package, see α portion in Figure 5 which represents the risk of false alarm (false positive).[3]

The Detection Limit represents the minimum activity that can be detected by the measurement system, i.e. which will induce a net signal larger than DT with a sufficient probability. The DL is such that an acceptably small portion of the net signal distribution lies below the DT, see β portion in Figure 5 which represents the risk of non-detection (false negative).[3]
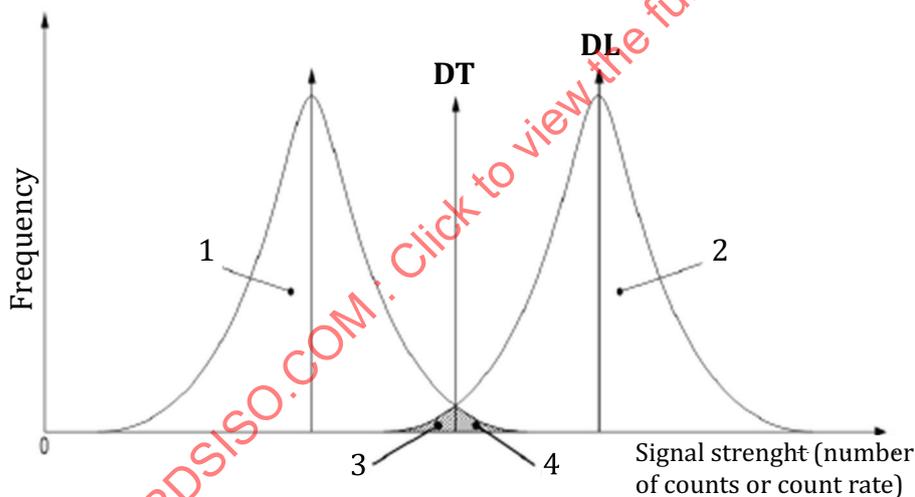
Key

1   background distribution
2   signal distribution
3   non detection β proportion
4   false

Figure 5 — Illustration of the relationship between decision threshold (DT) and detection limit (DL)

With respect to Figure 5, the left hand curve represents the distribution of counts in the region of interest where a peak would be if the radionuclide were present and the right hand curve represents

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3)   The "acceptable" portion is application specific. Typical values used are 0,01, 0,05 and 0,1. This does not preclude the selection of other values as appropriate to the application.

the total counts in the peak region (net plus background signal) when the activity of the radionuclide (present within the waste) is equal to the DL.

The DL is an "a-priori" value. It combines both the probability of detection and the probability of non-detection. The DL is best used to compare performance of one system or counting arrangement to another. After the sample has been counted and analysed, one of the two probabilities has been eliminated, activity of a radionuclide is either detected, for a given gamma ray, or not detected, depending on whether the net signal is above or below DT. If radioactivity is detected, it should be reported along with the uncertainty. If it is not detected, the analyst is often required to provide an estimate of the size of source that may have been present even if the net signal is below the DT.

In this instance, the analyst may choose arbitrarily to declare the measured value and its uncertainty, or the DT together with a level of confidence, or the DL, depending on context and system performance requirements.

NOTE   The DL is often declared in security and safety applications. However, this approach systematically overestimates the activity, which may be an issue regarding storage or repository accountability.

7   Quality assurance

7.1   General

It is an expectation of this International Standard that all relevant measured data, applied calibration and calculated correction factors as well as deduced results will be documented and recorded.

It is an expectation of this International Standard that gamma measurement systems will be specified, designed, built, operated, maintained and de-commissioned in accordance with recognized and appropriate processes and procedures.

It is outside of the scope of this International Standard to consider every stage within the system lifecycle in explicit detail. The expectation of this International Standard is that each stage within the lifecycle will be governed and performed in accordance with recognized and appropriate processes and procedures.

It is an expectation of this International Standard that "appropriate" Quality Control performance tests will be designed and executed, and the results documented, so as to demonstrate that the system performs as required when installed and continues to perform as required throughout its operational life.

ISO/IEC 17025 specifies general requirements that are relevant regarding quality insurance.

It is, however, appropriate to consider the following areas in more detail.

7.2   Record of calibration, validation, and waste measurements

It is recommended that the raw and processed data from all performance demonstration (Quality Control) testing be recorded to aid detection of deviation of the equipment from its normal operation. It is recommended that these Quality Control results remain available for the operational life of the equipment (as a minimum), e.g. for audit purposes.

It is recommended that the measurements of the radioactive waste packages be recorded for purposes of traceability, as well as to allow post analysis in case of unexpected results or deviation of the equipment.

The following is provided by way of an example of the information which could be recorded, either in full or as a reference to a separate unique data set:

— waste package identification number;

— waste package weight;

— date and time of the acquisition;

— data acquisition parameters (high voltage, threshold, dead time, electronics settings, etc.);

— energy and peak shape calibration functions (see 5.1);

— "background spectrum" (see 5.2);

— nuclear data (peak intensities, radioactive periods, etc.);

— gamma spectrum of the waste package;

— net peak areas after background subtraction;

— peak efficiency calibration function

— attenuation and other correction parameters (see 5.2);

— activity of the radionuclides of interest;

— uncertainties;

— DL or other value for non-detected radionuclides.

7.3   Documentation and procedures

Documents should be developed describing the system, how it was calibrated, and how it was proven to be suitable for the intended application (fit for purpose). These documents should be peer reviewed, and approved as part of the equipment commissioning process. The documents should be stored and available for future reference and audits.

Written and approved procedures should be available for routine sample assay operations and for periodic performance demonstration (Quality Control) testing.

7.4   Quality control

The validation of the measurement system, proving its ability to assess with exactitude and accuracy the target activity in the waste package, shall be demonstrated during calibration. Then periodic quality control (QC) tests shall be performed to verify that the system does no shift or dysfunction, and that it still fulfills its initial requirements.

QC procedures should describe the tests to be done, the frequency of performance, the methods to evaluate the data to establish control limits, and what to do when the control limits have been exceeded.

It is necessary to evaluate the full system, and determine what could go wrong, and how that would affect the results. Then the appropriate QC tests are created to show that those things did not go sufficiently wrong and therefore that the measurement results are good. Typical Quality Control procedures include Spikes (to show that the instrument can perform consistent measurements at normal levels), Blanks (to show that the instrument can measure low levels), and Duplicates (to show that measurements of real samples are repeatable).

For nuclear measurement systems relevant here, Spikes are usually done as a single item measured repeatedly. It is best if the item closely resembles a real sample, so that the full system can be checked for consistency (weight measurements, transmission measurements, energy calibration, shape calibration, spectral analysis procedure, efficiency, and correction factor consistency). Blanks can be empty package measurements if there are no peaks from the radionuclides of interest in the background, but it is best if a typical sample size and mass is measured. Duplicates can be a part of the process, as for instance assaying a waste package a second time periodically.

Care should be taken in establishing Warning and Action Levels for these various QC parameters. It is appropriate to acquire and save many QC parameters in the QC record. These can be useful for retrospective evaluations of minor instrument trends that are not sufficient to affect the quality of the results, but might predict future preventative action. But only a few key parameters should be

implemented with Warning and Action Levels. Warning Levels and Action Levels should be set based upon how that parameter can affect results, not simply upon how stable the last N measurements of that parameter have been. For example, the FWHM may be consistent at ±Δ % of the nominal value, but the Activity results are not affected until it is more than 10 × Δ % different than the nominal value; therefore an Action Level that requires stopping operations at 2 × Δ % is not warranted, but should be closer to 10 × Δ %.

7.5 Competence

Personnel involved in specifying, delivering, testing, operating and maintaining gamma radiation measurement systems should be demonstrated to be competent to discharge those responsibilities. An assessment of competence shall be based on formal training, previous experience and qualifications; these should be relevant and appropriate to both the nature and duty of the equipment, and to the role being undertaken.

Annex A
(informative)

Examples of application of the techniques and methods discussed within this International Standard

A.1 General

This Annex includes examples of how the methods and techniques discussed within this International Standard have been implemented on systems which are currently in use.

Since the precise implementation may differ from application to application, the contents of this Annex should be considered to be illustrative rather than prescriptive.

In addition to these examples, References [1] to [3] provide theoretical and practical information for the implementation of gamma ray spectroscopy.

A.2 Example of a box measurement in open geometry

A.2.1 Problem statement

A waste box containing low level radioactive waste was scanned using a 37 % relative efficiency HPGe detector (see Figure A.1). Open detector geometry was utilized. Note that the detector was shielded but no collimator was utilized. Particulars are given in Table A.1.
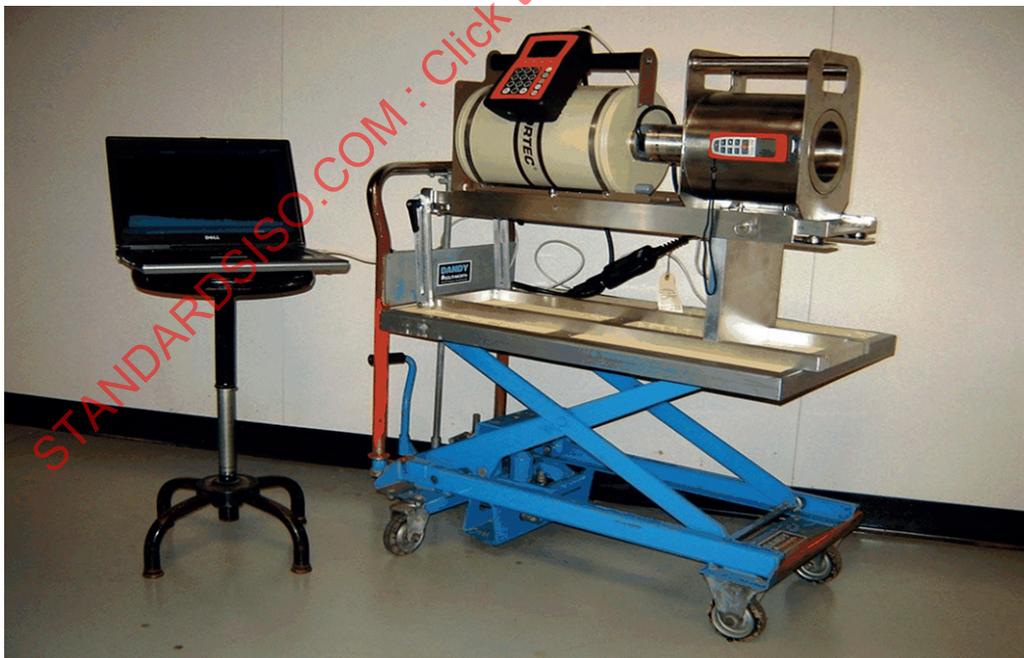

Figure A.1 — Mobile HPGe spectroscopy system

Table A.1 — Particulars of waste box and measurement geometry

Length of box: 191 cm
Width of box: 127 cm
Height of box: 73 cm
Wall Thickness: 0,267 cm
Material of construction of box: Steel
Average density of waste in box: 0,19 g/cm3
Distance of measurement from large face of box: 1 m

A.2.2 Detector's field of view

Figure A.2 shows the projected field of view through the opening in the detector's shielding. The diameter of the projection was estimated based on simple geometrical considerations and compared with the size of the waste box to be scanned to ensure that the entire box was within the field of view.
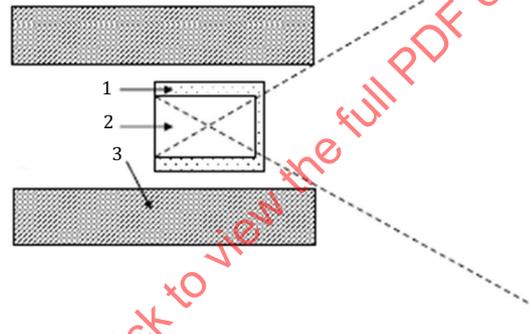
Key

1 detector housing
2 detector crystal
3 detector shielding

Figure A.2 — Projected field of view of detector

A.2.3 Measurement of box source activity and point source efficiency calibration

The detector was placed in front of the large face of the box source and counted for a suitable duration depending on the level of activity present. The axis of the detector is coincident with the centre of the box.

A point source efficiency calibration plot was also prepared by counting a mixed radionuclide activity calibration source at a distance of 30 cm from the face of the detector's shield on its central axis.

A.2.4 Methodology for data interpretation

This subclause presents the methodology used to determine the activity of a box source from its measured count rate. The methodology is based on the use of the point source efficiency calibration data (without the waste package) and the application of the MicroShield shielding code to model the waste container geometry and the corresponding photon attenuation.

NOTE   Microshield is a commercial product; alternative products exist (Annex A).

For a point source calibration (without waste package), Formula (A.1) relationship applies:

$$I_p\left(E\right) = \frac{S_p\left(E\right)}{4\pi R^2} = \frac{A_p\rho\left(E\right)}{4\pi R^2} \tag{A.1}$$

where I_p is the photon flux (photons.cm^{-2}·s^{-1}) from a point source located at a distance R from the detector on its axis. The source has an activity A_p (Bq) and emits photons with multiple emission energies. The source strength corresponding to emission energy E (MeV) is $S_p(E)$ (photons/s); this equals the product of the activity A_p (Bq) and $\rho(E)$ the fractional number of photons of energy E emitted per disintegration.

Efficiency of the point source can be expressed in Formula (A.2)

$$\in_p\left(E\right) = \frac{N_p\left(E\right)}{A_p\rho\left(E\right)} \tag{A.2}$$

where N_p represents the net counts/s measured by the detector in the peak of energy E. It follows from the above that

$$I_p\left(E\right) = K_p\left(E\right)N_p\left(E\right) \tag{A.3}$$

where $K_p(E)$ the energy dependent constant is given by

$$K_p\left(E\right) = \frac{1}{\in_p\left(E\right)4\neq R^2} \tag{A.4}$$

$K_p(E)$ (cm^{-2}) which represents the ratio between photon flux reaching the detector and measured count rate, should also be applicable to a box source. In fact, a simplification is made in considering that the difference of photon incoming angles in the detector between the point source and box geometries does not have a significant effect on its intrinsic detection efficiency. Therefore,

$$I_B\left(E\right) = N_B\left(E\right)K_p\left(E\right) \tag{A.5}$$

where $I_B(E)$ represents the flux of photons (photons.cm^{-2}·s^{-1}) of energy E from the box source which corresponds to the measured count rate $N_B(E)$ at the applicable distance of measurement.

Based on a MicroShield code representation of the box source counting geometry (the attenuation property of the waste is assumed to approximate that of carbon), the flux of photons of energy E (photons.cm^{-2}·s^{-1}) from the box source can be represented as

$$I_B\left(E\right) = K_B\left(E\right)V_B S_B\left(E\right) \tag{A.6}$$

where $K_B(E)$ (cm^{-2}) is an energy dependent function, V_B is the volume of the box (cm^3) and $S_B(E)$ is the box volumetric source strength (photons.cm^{-3}·s^{-1}) corresponding to emission energy E. MicroShield calculations with a source strength S_B of 1 photon.cm^{-3}·s^{-1} will yield a value for $I_B(E)$ (photons.cm^{-2}·s^{-1}); this represents the value of the product $K_B V_B$. Combining Formulae (A.5) and (A.6) yields

$$K_B \cdot \left(E\right) = \frac{I_B\left(E\right)}{V_B S_B\left(E\right)} = \frac{I_B\left(E\right)}{V_B A_B\rho\left(E\right)} = \frac{N_B K_p}{V_B A_B\rho} \tag{A.7}$$

where A_B (Bq. cm^{-3}) represents the volumetric box activity. Formula (A.7) can be re-arranged and combined with Formula (A.4) to yield the final expression for calculating the box activity corresponding to photon energy E.

$$A_B = \frac{N_B}{K_B V_B \rho 4\pi R^2 \in_p} \tag{A.8}$$

NOTE   Distance notation R, as discussed earlier, refers to the calibration point source measurement and not to the box source measurement.

A.2.5   Determination of Box Source Activity

Table A.2 illustrates the application of Formula (A.8) for calculating the activity of a box source whose particulars are given in Table A.1; the box was assumed to be 100 % filled; the waste attenuation property being approximated by that of carbon. Note that the activity of radionuclides with multiple emissions is calculated for each of its emission energies. An average activity can be deduced based on results for multiple emissions. In some cases, results based on an emission with a relatively low value of intensity ρ (%) may be disregarded because of its lower reliability. An average weighted by the inverse of the relative uncertainty of each peak can also be used. The last column of Table A.2 shows the estimated box source efficiency calculated analogous to Formula (A.2).

Table A.2 — Calculation of box source activity based on Formula (A.8)

Radionuclide	Energy (keV)	Photons per disintegration ρ (%)	Point source Eff. ϵ	Box count rate N_B (c/s)	Estimate for $K_B V_B$	Box activity A_B (Bq/cm³)	Box Eff. $\in_B = \dfrac{N_B}{V_B A_B \rho}$
Mn-54	834,8	99,98	$4,8 \times 10^{-4}$	$1,92 \times 10^{-1}$	2,26	$3,5 \times 10^{-2}$	$6,95 \times 10^{-6}$
Co-60	1332,5	99,99	$3,3 \times 10^{-4}$	$2,89 \times 10^{-1}$	2,70	$7,7 \times 10^{0}$	$5,76 \times 10^{-6}$
Zn-65	1115,5	50,60	$3,8 \times 10^{-4}$	$6,35 \times 10^{-2}$	2,52	$2,9 \times 10^{-2}$	$6,18 \times 10^{-6}$
Zr-95	756,7	54,00	$5,2 \times 10^{-4}$	$5,52 \times 10^{-1}$	2,17	$1,7 \times 10^{-1}$	$7,23 \times 10^{-6}$
Nb-94	871,1	100,00	$4,7 \times 10^{-4}$	$3,75 \times 10^{-1}$	2,29	$7,1 \times 10^{-2}$	$6,83 \times 10^{-6}$
Nb-95	765,8	100,00	$5,2 \times 10^{-4}$	$1,73 \times 10^{0}$	2,18	$3,0 \times 10^{-1}$	$7,19 \times 10^{-6}$
Ag-110	884,7	72,20	$4,6 \times 10^{-4}$	$4,05 \times 10^{-2}$	2,31	$1,1 \times 10^{-2}$	$6,78 \times 10^{-6}$
Sb-124	1691,0	47,79	$2,8 \times 10^{-4}$	$6,85 \times 10^{-2}$	3,00	$4,6 \times 10^{-2}$	$5,30 \times 10^{-6}$
Sb-125	427,9	30,00	$8,1 \times 10^{-4}$	$2,80 \times 10^{-2}$	1,76	$1,0 \times 10^{-2}$	$9,17 \times 10^{-6}$
Cs-134	795,9	85,53	$5,0 \times 10^{-4}$	$3,80 \times 10^{-2}$	2,22	$7,9 \times 10^{-3}$	$7,08 \times 10^{-6}$
Cs-137	661,7	85,10	$5,8 \times 10^{-4}$	$3,24 \times 10^{0}$	2,07	$5,8 \times 10^{-1}$	$7,64 \times 10^{-6}$
Eu-152	344,3	27,00	$9,7 \times 10^{-4}$	$1,05 \times 10^{-2}$	1,64	$3,6 \times 10^{-3}$	$1,01 \times 10^{-5}$
Eu-154	1274,4	35,19	$3,5 \times 10^{-4}$	$2,90 \times 10^{-2}$	2,65	$2,1 \times 10^{-2}$	$5,86 \times 10^{-6}$

Figure A.3 presents a plot of the derived box source efficiency versus energy curve. For comparison, the point source efficiency plot is also shown. As shown, the box source efficiencies are significantly lower. Note that the slope of the box source efficiency curve is similar to that of the point source efficiency curve because of the relatively low density of the waste in this case. In general, it would be flatter for higher density waste.
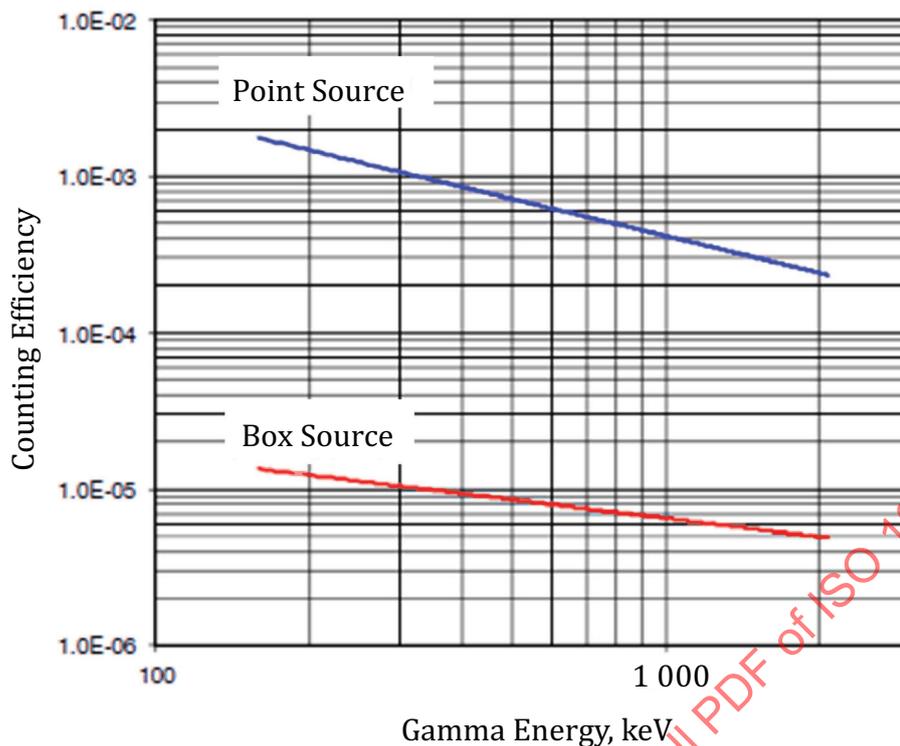
NOTE   It is not necessary to plot all points; only those points covering the range of energy of interest need to be plotted.

Figure A.3 — Sample plot, illustrating the difference between point source and box source efficiencies

A.3   Example showing a drum measurement system, configured in open geometry, used for the characterization of the plutonium content of the waste

A.3.1   Problem statement

A waste drum containing between 1 mg and 100 g of plutonium is measured using a 26 % relative efficient BEGE HPGe detector (see Figure A.4). An uncollimated (open) detector configuration is utilized. Note that the detector was shielded but no collimator was used. Particulars are given in Table A.2.
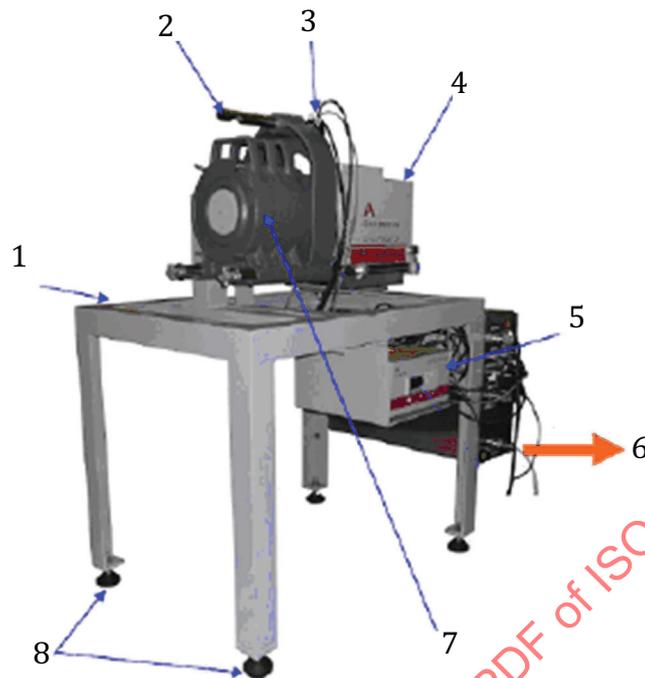
Key

1	supporting frame	5	data acquisition and processing
2	laser positioner	6	ethernet link
3	preamp HV-inhibit	7	lead rings (shielding)
4	cryogenics detector	8	adjustment feets

Figure A.4 — HPGe spectroscopy system

A mechanical system facilitates the location of up to four plates of tin (each 1 mm in thickness) in front of the detector, for the purpose of limiting the count rate due to the 59,5 keV gamma ray of ^{241}Am (to prevent saturation of the detector counting chain).

This gamma spectrometry station is coupled with a passive counting neutron cell. Figure A.5 shows the assembly of both measurement stations.

Key

1 gamma spectro station

2 neutron cell

Figure A.5 — Gamma and neutron measurement system

Table A.3 — Particulars of waste drum and measurement geometry

Height of drum: 76 cm
Radius of drum: 23 cm
Wall Thickness: 0,1 cm
Drum material: Steel
Average density of waste in drum: 0,01 to 1 g/cm^3
Distance of the detector to the axis of the drum: 1,23 m

A.3.2 Detector's field of view

Figure A.6 shows the projected field of view through the opening in the detector's shielding. The diameter of the projection was estimated based on simple geometrical considerations and compared with the size of the waste drum to ensure that the entire drum is within the field of view. The dimensions of the tin plates cover completely the solid angle of the drum with regard to the detector.
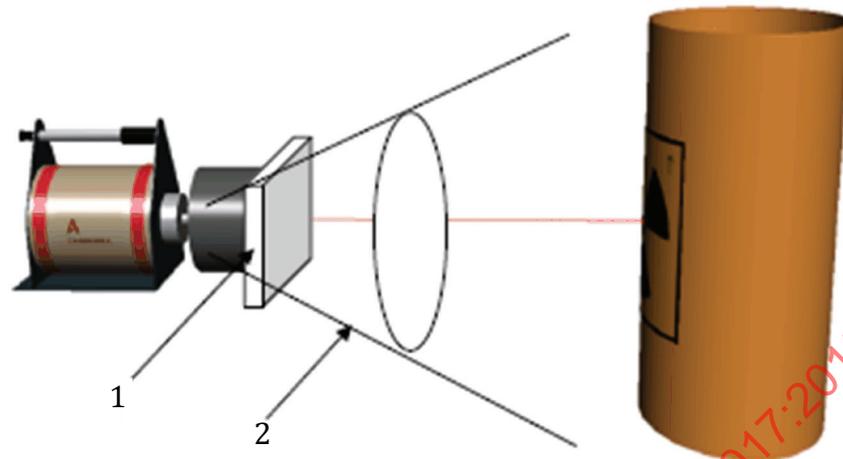
Key

1   tin plates
2   solid angle

Figure A.6 — Projected field of view of detector

A.3.3   Measurement process and efficiency calibration

The drum is placed on the rotating tray then put in rotation. The gamma and neutron measurements are launched simultaneously on a duration of acquisition of 1 800 s. The axis of the detector is coincident with the centre of the drum.

The calculation of the efficiencies is realized by means of a computer code. A beam of efficiencies curves is determined for a series of bulk densities of the waste 0,01 g/cm^3, 0,1 g/cm^3, 0,25 g/cm^3, 0,5 g/cm^3, 0,75 g/cm^3, and 1 g/cm^3. This beam of curves is calculated for five matrix compositions and for five configurations of tin plates (0 to 4 of 1 mm thickness) on a range of energy from 50 keV to 2 000 keV.

The model of calculation of the efficiencies is based on a linear source with the height of the drum placed on the axis of the drum filled with a type of homogeneous supposed matrix.

In the hypothesis of a random distribution of the contamination in drums and for a large number of drums, this geometrical configuration tends to voluntary overestimate the average measured activity, in order not to risk an underestimation.

A.3.4   Determination of drum activity

This subclause presents the methodology used to determine the activity of a drum from its measured count rate. The methodology is based on use of the pre-calculated line source efficiency calibration according to the container and detector geometry. It is important to underline that duration of 30 days is systematically waited between the closure of the drum and the measurement. This lapse of time allows making sure that ^{237}U (six days of period) is almost in radioactive equilibrium with ^{241}Pu. Indeed the gamma rays of ^{237}U are very intense and numerous.

Before beginning the acquisition, the operator selects the type of the matrix and the number of tin plates. He informs the filling height and the gross mass of the drum.

The system determines the bulk density of the waste by subtracting the mass of the empty drum.

The system performs a 120 s preliminary acquisition and indicates to the operator if it is necessary to add or to remove a shield of tin. This information is calculated to optimize the gamma spectrum in the

analysis of the plutonium isotopic composition with a dedicated software (height peak of 59 keV γ-ray of ^{241}Am identical to the peaks in the 100 keV region).

Another 1 800 s acquisition is then launched and the gamma spectrum is analysed automatically to determine the plutonium isotopic composition and the various peaks found in the spectrum (centroid and area). Figure A.7 shows an example of gamma spectrum.
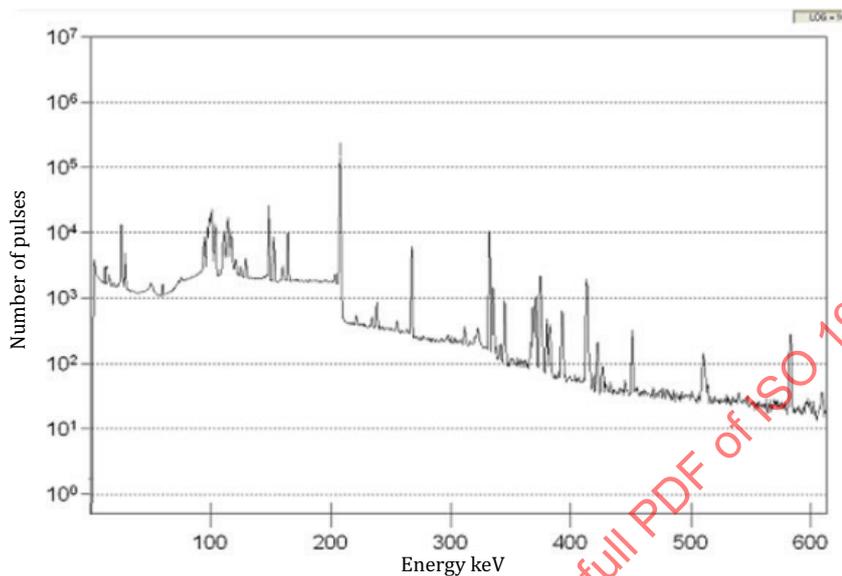

Figure A.7 — Example of gamma spectrum for a drum containing plutonium

Table A.2 below presents the list of the potentially exploitable gamma lines in the spectrum. The most intense lines are in bold characters.

NOTE   Both within Table A.4, and in the rest of this paragraph, it is assumed that ^{237}U is in equilibrium with ^{241}Pu and so considered as ^{241}Pu.

Table A.4 — Gamma energies which are often detectable

Energy (keV)	Radioelement
152,75	^{238}Pu
766,25	^{238}Pu
129,25	^{239}Pu
144,25	^{239}Pu
171,25	^{239}Pu
195,75	^{239}Pu
203,75	^{239}Pu
255,25	^{239}Pu
297,25	^{239}Pu
345,25	^{239}Pu
375,054	^{239}Pu
380,25	^{239}Pu
393,25	^{239}Pu
413,75	^{239}Pu

Table A.4 *(continued)*

Energy (keV)	Radioelement
422,75	^{239}Pu
451,25	^{239}Pu
598,08	^{239}Pu + ^{241}Am
160,308	^{240}Pu + ^{239}Pu + ^{241}Pu
642,25	^{240}Pu
94,75	^{241}Pu
117,75	^{241}Pu
148,567	^{241}Pu
164,58	^{241}Pu + ^{241}Am
208,25	^{241}Pu + ^{241}Am
267,75	^{241}Pu + ^{241}Am
332,25	^{241}Pu + ^{239}Pu + ^{241}Am
370,75	^{241}Pu + ^{241}Am
125,25	^{241}Am + ^{239}Pu
169,75	^{241}Am
335,4	^{241}Am + ^{239}Pu + ^{241}Pu
368,6	^{241}Am + ^{239}Pu + ^{241}Pu

The activity of the various radonuclides of interest is automatically determined from the Formulae (A.9) and (A.10) below.

$$A_E^i = \frac{Sn}{t \cdot I_E^i \cdot \varepsilon_E} \tag{A.9}$$

$$M_E = \frac{A_E^i}{P^i} \cdot W^i \tag{A.10}$$

where

A_e^i is the activity of radionuclide "*i*" (Bq) determined from its gamma ray of energy "*E*";

Sn is the net area of the peak of radioelement "*i*" to the energy "*E*" (count);

t is the acquisition time corrected by the dead time (i.e. live time in seconds);

I_E^i is the gamma emission intensity of radioelement "*i*" at energy "*E*";

ε_E is the probability of detection of a gamma ray of energy "*E*" (net number of counts in the peak per photon emitted in the waste). His value is determined according to the methodology described below.

For radionuclides corresponding to an isotope plutonium, the system determines M_E

where

M_E is the mass of plutonium (g) determined at energy "*E*";

P^i is the mass proportion of radioelement "i" (plutonium isotope) determined with the code Multi gamma analysis (MGA);

NOTE   MGA is a commercial product and not a part of the normative text (Annex A). Alternative products exist.

W^i is the specific mass of radioelement "i" (g/Bq).

The determination of efficiency ε_E is realized as follows.

The system looks for the beam of efficiency curves (according to the densities) for the type of chosen matrix and for the number of tin shields selected by the operator. Figure A.8 presents an example for a steel matrix and just one tin plate.
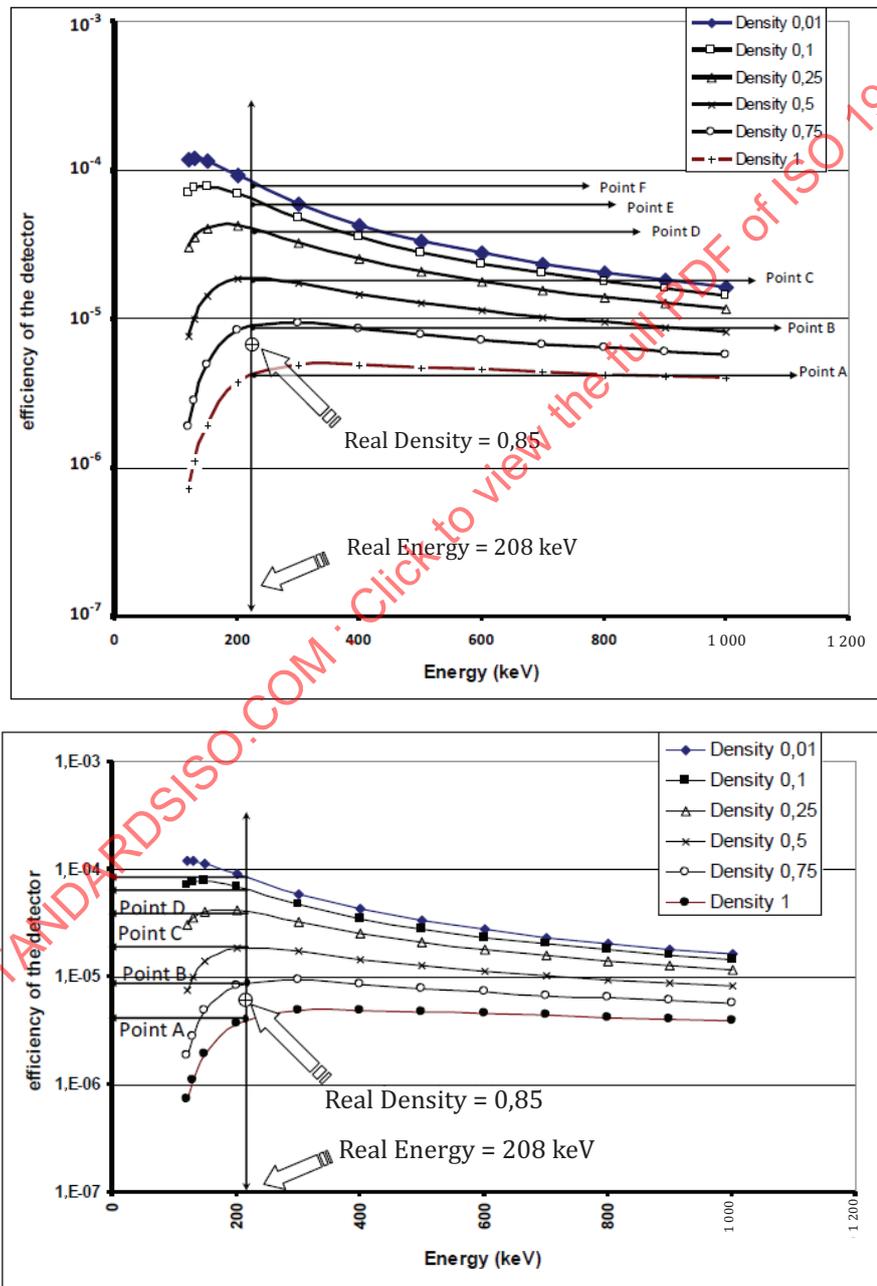

Figure A.8 — Efficiency curves for the steel matrix and one tin shield

For the energy of the gamma peak measured in the spectrum (for example 208 keV on Figure A.8), the system looks for the efficiencies of points A, B, C, D etc. Then the system reconstructs an efficiency curve according to density using the values of points A, B, C, D, as shown in Figure A.9.
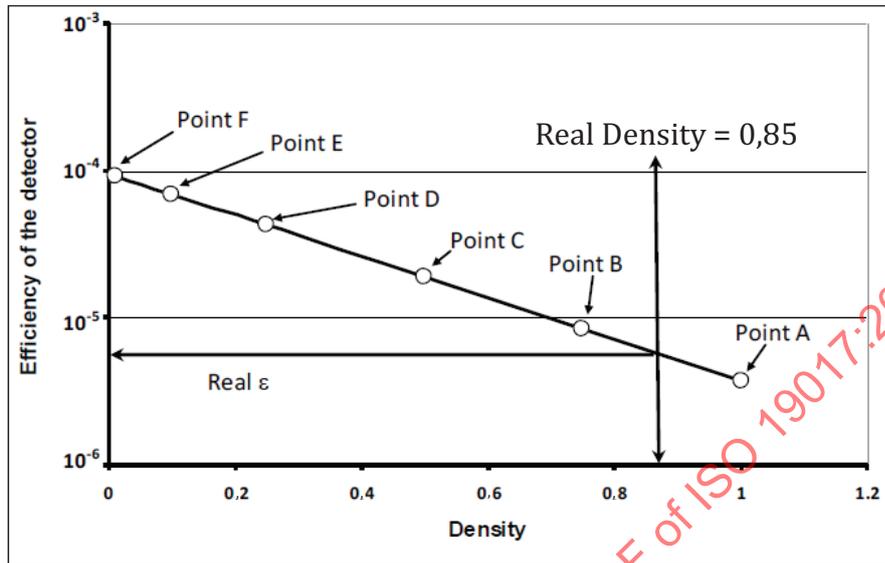

Figure A.9 — Efficiency curve as a function of density

The system then performs a logarithmic interpolation for the real measured density (0,85) of the drum.

However, efficiency curves do not take into account a possible self-absorption of the gamma rays in plutonium itself which could be significant in the case of a local concentration of plutonium.

The system includes a correction of this phenomenon as explained below.

From Formula (A.10), the system reconstructs a curve of M_E according to the logarithm of the inverse of energy for every peak found in the spectrum corresponding to an isotope of plutonium generally ^{239}Pu. Peaks of other isotopes can also be used if the isotopic composition of the nuclear material is a priori known, or has been measured.

Figure A.10 presents an example in which the self-absorption phenomenon is present in the drum.
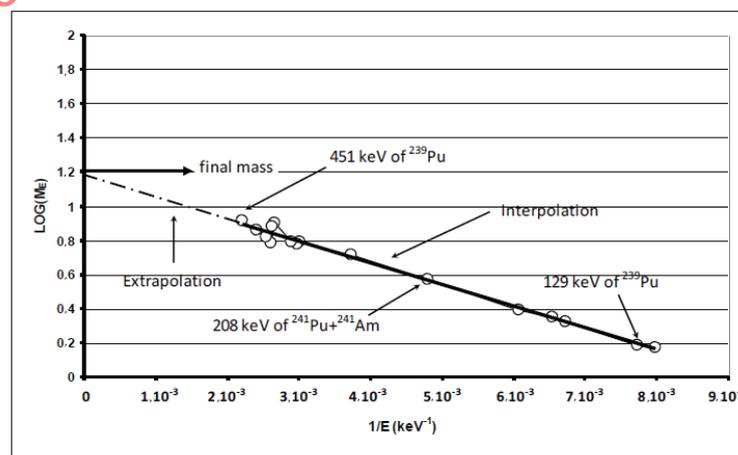

Figure A.10 — Mass plutonium as a function of 1/Energy

The method of correction is based on a linear adjustment of $\ln(M_E)$ according to $1/E$ by a least square method. For $1/E$ tends toward zero (i.e. infinite E), it is reasonable to think that the phenomena of self-absorption is negligible.

The underlying physical reason is that photoelectric effect is predominant in the energy range of interest. Therefore the curve $\ln(M_E) = f(1/E)$ can be extrapolated to "infinite energy", where photoelectric effect tends toward zero, to estimate the "un-attenuated true mass" of plutonium. The main limitation of the method is that the surface mass of the plutonium samples present in the waste does not practically exceed 10 g.cm^{-2}. Beyond this limit, the extrapolation underestimates the plutonium mass (because the photons emitted in the center of the sample are not detected).

This method imposes the use of the gamma lines of energy superior to 121,82 keV corresponding to plutonium's K-edge (i.e. electron binding energy on plutonium's atomic K shell). See Reference [4] for more details on the "infinite energy extrapolation" method.

A.4   Automated systems for general waste characterization

A.4.1   Problem statement

Consider the situation requiring a high throughput of drummed and/or boxed waste containers that must be individually assayed and quantified for sequestration and disposal. Containers may contain a variety of radionuclides with unknown source and matrix distributions. An automated system is necessary to manage the throughput.

Examples of such automated systems are presented in Figure A.11. Each of these systems consists of one or multiple germanium detectors that measure the sample container from one or more positions. The placement of the container with respect to the detectors is done by automated mechanical controls. The quantified analysis of the radioactive content of the container is typically done by analysing the sum of the measured spectra. Inhomogeneity of matrix and/or source distribution can be identified by analysing the individual spectra and comparing them against expected responses to homogeneous matrices. If necessary, corrections to each measurement can be applied to potentially reduce the total measurement uncertainty (TMU).

NOTE   Left: Q2, Middle: segmented gamma scanner, Right: box counter.

Figure A.11 — Examples of automated systems

A.4.2   Detector's field of view

While the details of the field of view depend on the design of the specific system, the detector(s) of the system can be collimated to reduce the overall field of view of the container. This allows inhomogeneity in the container to be more readily identified. An example of this restricted field-of-view is shown in Figure A.12. If the container matches the assumptions used in the efficiency calibration, a reduced field of view only has an impact in reducing the overall statistics in the measurement. This is often recovered by using multiple detectors or measuring with a single detector in multiple positions.