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Measurement of radioactivity — Gamma emitting radionuclides — Rapid screening method using scintillation detector gamma-ray spectrometry

Mesurage de la radioactivité — Radionucléides émetteurs gamma — Méthode d'essai de dépistage par spectrométrie gamma utilisant des détecteurs par scintillation

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical committee ISO/TC 85, *Nuclear Energy, nuclear technologies, and radiological protection*, Subcommittee SC 2, *Radiological protection.*

Introduction

Everyone is exposed to natural radiation. The natural sources of radiation are cosmic rays and naturally occurring radioactive substances which exist in the earth and within the human body. Human activities involving the use of radiation and radioactive substances add to the radiation exposure from this natural exposure. Some of those activities, such as the mining and use of ores containing naturally-occurring radioactive materials (NORM) and the production of energy by burning coal that contains such substances, simply enhance the exposure from natural radiation sources. Nuclear power plants and other nuclear installations use radioactive materials and produce radioactive effluent and waste during operation and on their decommissioning. The use of radioactive materials in industry, agriculture and research is expanding around the globe.

All these human activities give rise to radiation exposures that are only a small fraction of the global average level of natural exposure. The medical use of radiation is the largest and a growing man-made source of radiation exposure in developed countries. It includes diagnostic radiology, radiotherapy, nuclear medicine and interventional radiology.

Radiation exposure also occurs as a result of occupational activities. It is incurred by workers in industry, medicine and research using radiation or radioactive substances, as well as by passengers and crew during air travel and for astronauts. The average level of occupational exposures is generally below the global average level of natural radiation exposure[11].

As uses of radiation increase, so do the potential health risk and the public's concerns. Thus, all these exposures are regularly assessed in order to

a) improve the understanding of global levels and temporal trends of public and worker exposure

b) to evaluate the components of exposure so as to provide a measure of their relative importance, and

c) to identify emerging issues that may warrant more attention and study.

While doses to workers are mostly directly measured, doses to the public are usually assessed by indirect methods using radioactivity measurements results performed on various sources: waste, effluent and/or environmental samples.

To ensure that the data obtained from radioactivity monitoring programs support their intended use, it is essential that the stakeholders (for example, nuclear site operators, regulatory and local authorities) agree on appropriate methods and procedures for obtaining representative samples and then handling, storing, preparing and measuring the test samples. A assessment of the overall measurement uncertainty needs also to be carried out systematically. As reliable, comparable and ‘fit for purpose’ data are an essential requirement for any public health decision based on radioactivity measurements, international standards of tested and validated radionuclide test methods are an important tool for the production of such measurement results. The application of standards serves also to guarantee comparability over time of the test results and between different testing laboratories. Laboratories apply them to demonstrate their technical qualifications and to successfully complete proficiency tests during interlaboratory comparison, two prerequisites for obtaining national accreditation. Today, over a hundred international standards, prepared by Technical Committees of the International Standardization Organization, including those produced by ISO/TC85, and the International Electrotechnical Commission (IEC), are available for application by testing laboratories to measure the main radionuclides.

Generic standards help testing laboratories to manage the measurement process by setting out the general requirements and methods to calibrate and validate techniques. These standards underpin specific standards which describe the test methods to be performed by staff, for example, for different types of sample. The specific standards cover test methods for:

— Naturally-occurring radionuclides (including ^{40}K, ^{3}H, ^{14}C and those originating from the thorium and uranium decay series, in particular ^{226}Ra, ^{228}Ra, ^{234}U, ^{238}U, ^{210}Pb) which can be found in materials from natural sources or can be released from technological processes involving naturally occurring radioactive materials (e.g. the mining and processing of mineral sands or phosphate fertilizer production and use);

— Human-made radionuclides, such as transuranium elements (americium, plutonium, neptunium, and curium), ^{3}H, ^{14}C, ^{90}Sr and gamma emitting radionuclides found in waste, liquid and gaseous effluent, in environmental matrices (water, air, soil, biota) and food and feed as a result of authorized releases into the environment and of fallout resulting from the explosion in the atmosphere of nuclear devices and accidents, such as those that occurred in Chernobyl and Fukushima.

Environmental materials, including foodstuffs, thus may contain radionuclides at activity concentrations which could present a risk to human health. In order to assess the potential human exposure to radioactivity and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, the environment and foodstuffs are routinely monitored for radioactivity content as recommended by the World Health Organization (WHO). Gamma-emitting radionuclides are usually quantified in environmental and food samples by gamma-ray spectrometry using High Purity Germanium (HPGe) gamma-ray spectrometry. Following a nuclear accident, a screening approach based on rapid test methods is recommended to help the decision makers to decide whether activity concentrations in environmental samples, feed and food samples are above or below operational intervention levels (OILs)[12] that are specifically set up to manage nuclear and radiological emergency. During nuclear emergency response, these default radionuclide specific OILs for food, milk and water concentrations from laboratory analysis would be used to measure the effectiveness of protective actions and contribute to determining any further actions required[12][13].

In 1989, following the Chernobyl accident, the first version of the Codex Guideline Levels (GLs) for Radionuclides in Foods Contaminated Following a Nuclear or Radiological Emergency (in the following referred to as “Codex GLs”) was adopted. The Codex GLs were reviewed in 2006 and are included in the General Standard for Contaminants and Toxins in Food and Feeds[14][15]. During a nuclear emergency situation, the Codex GLs for gamma-emitting radionuclides such as $^{106}Ru/^{106}Rh$ and ^{131}I is 100 $Bq\cdot kg^{-1}$; the GL for ^{60}Co, ^{103}Ru, ^{137}Cs and ^{134}Cs, ^{144}Ce is higher at 1000 $Bq\cdot kg^{-1}$ but a lower limit of 100 $Bq\cdot kg^{-1}$ still applies for foods for infants. Default radionuclide specific OILs for food, milk and water concentrations from laboratory analysis set up by FAO, IAEA, ILO, OECD/NEA, PAHO, OCHA, WHO were recently revised[16].

NOTE The Codex GLs are the activity concentration in foods that would result in an effective dose of 1 mSv/year for members of the Public (infant and adult) in accordance with the most recent recommendations of the International Commission on Radiological Protection (ICRP) considering that 550 kg of food is consumed per year by an adult and 200 kg of food and milk is consumed per year by an infant, with 10 % of the diet is of imported food, all of which is contaminated giving an import to production factor of 0,1. For convenience the GL values were rounded, and radionuclides with ingestion dose coefficients of similar magnitudes grouped and given similar GLs values. However, separate GLs were derived for infants and adults due to differences in radionuclide absorption, metabolism and sensitivity to radiation.

Emergency preparedness should include planning for the implementation of optimized test methods that can provide rapid estimates of activity concentration to be checked against OILs. Thus, an international standard on a screening method using Gamma-Ray Spectrometry is justified for use by testing laboratories carrying out measurements of gamma-emitting radionuclides during an emergency situation. Such laboratories are intended to obtain a specific accreditation for radionuclide measurement in environmental and/or food samples.

This document describes, after proper sampling, sample handling and preparation, a screening method to quantify rapidly the activity concentration of iodine and caesium in environmental, feedstuffs and foodstuffs samples using scintillation spectrometer during an emergency situation.

This document is one of a set of generic international standards on measurement of radioactivity.

Measurement of radioactivity — Gamma emitting radionuclides — Rapid screening method using scintillation detector gamma-ray spectrometry

WARNING — Persons using this document should be familiar with normal testing laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a screening test method to quantify rapidly the activity concentration of gamma-emitting radionuclides, such as ^{131}I, ^{132}Te, ^{134}Cs and ^{137}Cs, in solid or liquid test samples using gamma-ray spectrometry with lower resolution scintillation detectors as compared with the HPGe detectors (see IEC 61563).

This test method can be used for the measurement of any potentially contaminated environmental matrices (including soil), food and feed samples as well as industrial materials or products that have been properly conditioned. Sample preparation techniques used in the screening method are not specified in this document, since special sample preparation techniques other than simple machining (cutting, grinding, etc.) should not be required. Although the sampling procedure is of utmost importance in the case of the measurement of radioactivity in samples, it is out of scope of this document; other international standards for sampling procedures that can be used in combination with this document are available (see References [1],[2],[3],[4],[5],[6]).

The test method applies to the measurement of gamma-emitting radionuclides such as ^{131}I, ^{134}Cs and ^{137}Cs. Using sample sizes of 0,5 l to 1,0 l in a Marinelli beaker and a counting time of 5 min to 20 min, decision threshold of 10 Bq·kg^{-1} can be achievable using a commercially available scintillation spectrometer [e.g. thallium activated sodium iodine (NaI(Tl)) spectrometer 2” ϕ × 2” detector size, 7 % resolution (FWHM) at 662 keV, 30 mm lead shield thickness].

This test method also can be performed in a “makeshift” laboratory or even outside a testing laboratory on samples directly measured in the field where they were collected.

During a nuclear or radiological emergency, this test method enables a rapid measurement of the sample activity concentration of potentially contaminated samples to check against operational intervention levels (OILs) set up by decision makers that would trigger a predetermined emergency response to reduce existing radiation risks[12].

Due to the uncertainty associated with the results obtained with this test method, test samples requiring more accurate test results can be measured using high-purity germanium (HPGe) detectors gamma-ray spectrometry in a testing laboratory, following appropriate preparation of the test samples[7][8].

This document does not contain criteria to establish the activity concentration of OILs.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11929, *Determination of the characteristic limits (decision threshold, detection limit and limits of the confidence interval) for measurements of ionizing radiation — Fundamentals and application*

ISO 80000-10, *Quantities and units — Part 10: Atomic and nuclear physics*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

IEC 61453, *Nuclear instrumentation — Scintillation gamma ray detector systems for the assay of radionuclides – Calibration and routine tests*

3 Terms and definitions

For the purposes of this document, the terms, definitions, and the symbols and abbreviations given in ISO 80000-10 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at http://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
blank sample
sample, liquid or solid, with very low to no activity for radiation of the same type and region of interest, with a mass and a composition as close as possible to those of the test sample

3.2
emergency
non-routine *situation* that necessitates prompt action, primarily to mitigate a hazard or adverse consequences *for human life and health*, property and the environment

[SOURCE: IAEA safety glossary 2016 Rev.]

Note 1 to entry: This includes nuclear and radiological emergencies and conventional emergencies such as fires, release of hazardous chemicals, storms or earthquakes. It includes situations for which prompt action is warranted to mitigate the effects of a perceived hazard[12].

3.3
operational intervention level
OIL
set level of a measurable quantity that corresponds to a generic criterion

[SOURCE: IAEA safety glossary 2016 Rev. Mod]

Note 1 to entry: OILs are calculated levels, measured by instruments or determined by laboratory analysis, that corresponds to an intervention level or action level. These are typically expressed in terms of dose rates or of activity of radioactive material released, time integrated air activity concentrations, ground or surface concentrations, or activity concentrations of radionuclides in environmental, food or water samples. OILs are used immediately and directly (without further assessment) to determine the appropriate protective actions on the basis of an environmental measurement[12].

3.4
reference level

<emergency exposure situation or existing exposure situation> level of dose, risk or activity concentration above which it is not appropriate to plan exposures to occur and below which optimization of protection and safety would continue to be implemented

[SOURCE: IAEA safety glossary 2016 Rev.]

Note 1 to entry: The chosen value for a reference level depends upon the prevailing circumstances of the exposure under consideration[13]. Above the reference level, it is judged that the risks from exposure are not justified and therefore is not allowed to occur. Below the reference level, optimization of personnel protection needs to be implemented to keep exposures as low as reasonably achievable (ALARA).

3.5
screening level
SL

values that are set up by the laboratory taking into account the characteristics of the measuring equipment and the test method to guarantee that the test result and its uncertainty obtained are fit for purpose for comparison with the operational intervention levels (OILs)

Note 1 to entry: The screening level is less than the OIL. Therefore food is safe for consumption if the screening level is not exceeded. Actions to take, if the food is not safe for consumption, are given in Reference[16].

4 Symbols and units

A	Activity of each radionuclide in reference source, at the measurement time, in becquerels
c_A	Activity concentration of each radionuclide expressed in becquerels per kilogram
$c_{A,\mathrm{SL}}$	Activity concentration that corresponds to the screening level of each radionuclide expressed in becquerels per kilogram
$c_{A,\mathrm{RL}}$	Activity concentration that corresponds to the OIL of each radionuclide expressed in becquerels per kilogram
c_A^*	Decision threshold, without and with corrections, in becquerels per kilogram
$c_A^\#$	Detection limit, without and with corrections, in becquerels per kilogram
$c_A^\triangleright$	Upper limits of the confidence interval, in becquerels per kilogram
R_i	the ratio of the indicated value of a spectrometer to the conventional true value of specific radionuclide, i
ε_E	Counting efficiency of the detector at energy, E
$\varepsilon_{i,E}$	Radionuclide-specific counting efficiency of the detector at energy, E, of specific radionuclide, i
$n_{N,E}$, $n_{N\mathrm{s},E}$, $n_{N,\mathrm{SL},E}$	Number of net counts in the gamma-ray energy region of interest, at energy E, in the sample spectrum, in the calibration spectrum and in the spectrum obtained from the measurement of reference sample having activity that corresponds to the screening level, respectively
$n_{\mathrm{g},E}$, $n_{\mathrm{gb},E}$, $n_{\mathrm{gs},E}$ $n_{\mathrm{g,SL},E}$	Number of gross counts in the gamma-ray energy region of interest, at energy E, in the sample spectrum, in the background spectrum, in the calibration spectrum and in the spectrum obtained from the measurement of reference sample having activity that corresponds to the screening level, respectively

P_E	Probability of the emission of a gamma ray with energy, E, of each radionuclide, per decay
R_i	Response of the detector/radiometer to the reference activity of radionuclide, i
t_g	Sample counting live time, in seconds
T_b	Background counting live time, in seconds
t_s	Reference source counting live time, in seconds
t_SL	Counting live time, in seconds, of a reference sample with an activity corresponding to a screening level
$t_{k\text{-}1,\alpha}$	The two sided t-distribution with k-1 degree of freedom and α two sides probability
$u(c_A)$	Standard uncertainty associated with the measurement result c_A, without and with corrections, in becquerels per kilogram
U	Expanded uncertainty calculated by $U = k \cdot u\,(c_A)$ with $k = 1, 2...$, in becquerels per kilogram
m	Mass of the sample for test, in kilograms
α, β	Probability of a false positive and false negative decision, respectively
$1-\gamma$	Probability for the coverage interval of the measurand

5 Principle

During a nuclear or radiological emergency, it is essential to measure rapidly the activity concentration in samples from the environment and potentially contaminated foodstuffs and feed to protect workers and the public, in accordance with international standards, by keeping doses below the dose reference levels[13]. It is recognized among organizations responsible for emergency management that good preparedness can substantially improve the emergency situation response. Thus default OILs for food are set up by national authorities, and measurement procedures using commonly available contamination screening equipment are implemented to meet the OILs criteria. This should be carried out as part of the emergency preparedness process. The process of assessing radionuclide concentrations in food, milk and water is shown in Figure 1. During the process of assessing radionuclide concentrations in food, milk and water the potentially contaminated food should be screened over a wide area and analysed to determine promptly the activity concentration of gross and/or individual radionuclides. If the OIL are not exceeded, the food, milk and water are safe for consumption during the emergency phase. If an OIL is exceeded, the radionuclide specific concentrations in the food, milk or water should be determined. Finally, as soon as possible the guidance in Reference[17] should be used to determine whether the food, milk or water is suitable for international trade, and national criteria or WHO guidance[18] should be used to determine whether the food, milk or water is suitable for long term consumption after the emergency phase[16].

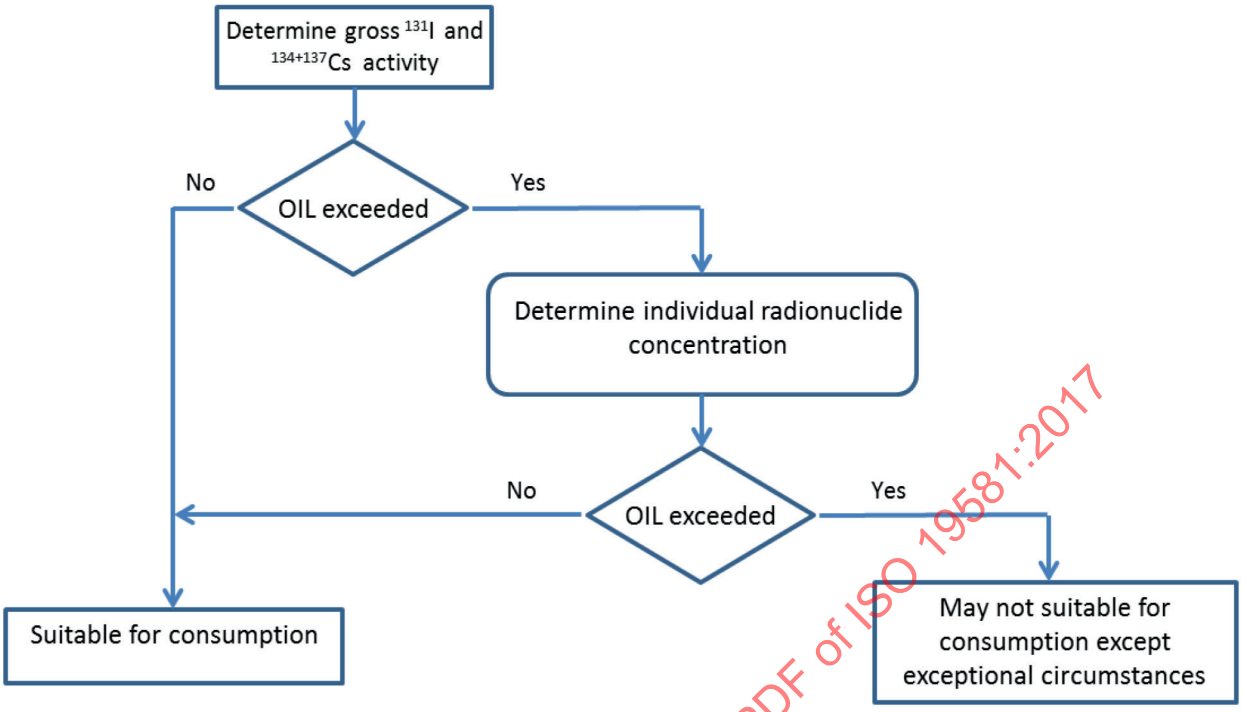

Figure 1 — Example of process of assessing radionuclide concentrations in food
(see explanations in the text and modified from Reference[16])

Laboratories shall make the necessary arrangements to be able to perform appropriate and reliable analyses of environmental and food/feed samples for the purposes of an emergency response. Thus, a screening approach is required, using a fast test method that rapidly provides test results to the decision maker in order to determine whether food and/or feed is suitable for human and/or animal consumption during the post-accident monitoring period and for international trade.

The main radioactive materials released into the atmosphere during a power plant nuclear accident are volatile elements including iodine isotopes (^{131}I, ^{132}I, ^{133}I), caesium isotopes (^{134}Cs, ^{136}Cs, ^{137}Cs) and tellurium (^{132}Te). Samples taken from the environment, the foodstuffs and feed may initially contain high activity concentrations of ^{131}I relative to the caesium isotopes[19]. Although often activity released is also dominated by noble gases, these cannot end up in food.

Therefore the accident monitoring that shall be implemented immediately following a nuclear accident requires a test method designed for the screening of ^{131}I activity concentration of environmental and food samples. When using a test method with a scintillation detector system incorporating a spectrometer (hereinafter referred to as scintillation spectrometer) or a portable gamma-ray detector (e.g. survey meter) with no radionuclide discrimination function, ^{131}I is not determined separately from other iodine isotopes and caesium isotopes. When using a test method with a scintillation spectrometer ^{131}I, ^{134}Cs and ^{137}Cs can be discriminated and potentially quantified with a test method using a scintillation spectrometer. However, using a multichannel analyzer (MCA) with a peak deconvolution program does not avoid the contributions in the energy region of interest of other radionuclides, including short-lived iodine isotopes, caesiums and naturally occurring radionuclides. The ^{131}I activity concentration is therefore overestimated but this is considered as acceptable during the immediate phase following a nuclear accident to rapidly assess the contamination.

A few months after the accident, the short-lived radionuclides, including iodine isotopes, have decayed. Longer-lived radionuclides including ^{134}Cs and ^{137}Cs become predominant in environmental and food samples. During this later period, when using a test method with a portable gamma-ray detector (e.g. survey meter) with no radionuclide discrimination function, ^{134}Cs and ^{137}Cs activity concentration cannot be quantified separately and the test result is considered as the gross activity of both ^{134}Cs and ^{137}Cs. With a scintillation spectrometer individual nuclide activity concentrations can be determined.

However contributions from naturally occurring radionuclides might be unavoidable even during this later period.

NOTE In the later stage, the radionuclide composition is usually known, including the ratio of ^{134}Cs to ^{137}Cs; it allows to estimate the activity concentration of the individual caesium isotopes using the gross activity results.

Direct measurement without iodine retention treatment, evaporation or ashing can be used to measure the activity concentration rapidly during the accident and post-accident monitoring period. The test sample is measured directly without any preparation, preferably in a Marinelli beaker type container.

As the tests are performed to check the activity concentration of the sample against OILs set up by national authority, screening levels should be set in a range from half of these OILs to close to the OILs in order to avoid false-positives.

Upper limit of confidence interval of the best estimate of the true value for the screening level $C^{\triangleright}_{A,\mathrm{SL}}$ shall be below that for the operational intervention level $C_{A,\mathrm{RL}}$ with a 95 %[1] confidence level (α = 0,05, k = 2) as shown in the following equation:

$$C^{\triangleright}_{A,\mathrm{SL}} = C_{A,\mathrm{SL}} + t_{k-1,\alpha} \cdot u(C_{A,\mathrm{SL}}) < C_{A,\mathrm{RL}} \tag{1}$$

where

t	is the two sided t-distribution;
$u(C_{A,\mathrm{SL}})$	is the uncertainty of the best estimate of the true value for the screening level.

The probability distribution function associated with the test results can be obtained by repeating the tests on the same samples. $u(C_{A,\mathrm{SL}})$ should be determined by repeated measurement with the minimum counting time of a reference source or reference material with an activity close to the screening level of the radionuclide tested and containing no other radionuclide. $C^{\triangleright}_{A,\mathrm{SL}}$ should specify the number of repeated measurements which would enable the use of a t-distribution (see 8.3). This document recommends to repeat the measurements of the same sample a minimum of 4 times.

In order to ensure the reliability of the screening test, the decision threshold (see ISO 11929) is defined taking into consideration that the counting time shall be shortened to obtain the test results rapidly for a large number of samples. This document recommends that one-fourth of the OIL value is an appropriate value for the decision threshold.

6 Apparatus

Several types of scintillation spectrometers can be used for sample screening. Commercially available scintillation crystals that would be useful for the screening test are shown in Table 1.

Table 1 — Examples of commercially available scintillation crystals. Typical detector size and its resolution are also shown

Crystal	Detector size	Resolution at 662 keV
NaI(Tl)	2” ϕ × 2”	8 %
CsI(Tl)	2” ϕ × 2”	10 %
$LaBr_3$	2” ϕ × 2”	<3 %

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[1] α = 0,05 (k = 2) is often chosen by default in the international standards for radiological protection but another α value may be required by the national regulation authority. For example, screening level being set above half of the level of the reference value with α=0,02 were required by Japanese government authority for the radio-caesium screening test in food following the Fukushima accident.

Table 1 *(continued)*

Crystal	Detector size	Resolution at 662 keV
SrI(Eu)	1,5” ϕ × 1,5”	<4 %
$CeBr_3$	2” ϕ × 2”	<4 %
GAGG(Ce)	2” ϕ × 2”	<7 %
BGO	2” ϕ × 2”	10 %
CLYC	2” ϕ × 2”	<5 %

A spectrometry apparatus consists of two parts: the scintillation detector with plug-on or external electronics and multi-channel analyser and the device which handles stores and analyses the measured spectra. Digital signal processing electronics are commonly used. Portable scintillation spectrometers for *in situ* measurements are also available. For screening, a simple total spectrum counting system that counts all pulses above a low-energy threshold or single-channel analyser (SCA) counting system that counts all pulses between upper and lower energy boundaries can be also used.

A portable gamma-ray detector (e.g. dose-rate meter that cannot provide spectrum information) which has sufficient sensitivity can also be used for screening as a simple total spectrum counting system that counts all pulses above a low-energy threshold instead of a spectrometer. For screening measurements, a portable gamma dose-rate meter may be simpler to use than spectrometers if a sufficiently low decision threshold is achievable within an appropriate counting time. However when low OILs are implemented, a radiation shield and/or large scintillator may be required to reduce the background level and to have sufficient detection efficiency, respectively.

7 Sample container

Sample containers that are suited to gamma spectrometry have the recommended characteristics:

- be made of materials with low absorption of gamma radiation;
- be made of transparent or semi-transparent material to see the level of content;
- have volumes and geometries adapted to the shape of the detector to ensure maximum detection efficiency;
- be watertight and not react with the test sample constituents;
- have a wide-necked opening and a close-fitting lid to facilitate filling;
- highly resistant to breakage.

In order to verify easily that the content of the container conforms to the standard counting geometry, a transparent container with a clearly discernible mark to indicate the fill level should be used.

To avoid contamination of the container or in order to reuse it, the sample can be placed in a plastic bag in the container.

8 Procedure

8.1 Packaging of samples for measuring purposes

Test samples submitted to screening by gamma spectrometry measurement are usually rough test samples without any preparation.

The procedure shall be carried out as follows:

a) Choose the container type that is best suited to the largest quantity of the test sample and determine the mass of the empty container (tare).

b) Fill the container to the level of the filling mark and measure the gross mass of the container. Visually check the surface level of the test sample in the container and ensure that it is horizontal before measuring.

c) Measure and note the test sample mass or volume. This information is needed to calculate the activity concentration (Bq·kg^{-1} or Bq·l^{-1}).

d) Hermetically seal the container if volatile radionuclides are being measured.

e) Clean the outside of the container to remove potential contamination due to the filling process.

8.2 Calibration

8.2.1 General

The calibration procedure is presented for two types of apparatus:

a) gross-count type apparatus.

An apparatus that counts all the signals with pulse heights higher than a threshold.

b) single-channel or multi-channel pulse height analysing type apparatus.

An apparatus that analyses pulse heights of signals using a single-channel or multi-channel pulse height analysing function.

All apparatus shall be operated in accordance with the manufacturer's instructions.

8.2.2 Reference source

Use of commercially available solid sealed sources of ^{137}Cs, ^{133}Ba (Mock ^{131}I) of appropriate geometry are recommended. These solid sources may be more useful when working in the field condition during an emergency situation.

A liquid reference solution can also be used for the calibration.

The activity content of reference sources, solid or in solutions shall be traceable to the national or international standards of radioactivity.

8.2.3 Check source

A check source is used for the instrument operability check, as well as for carrying out the tests to verify/evaluate the stability and the influence of ambient factors. Solid sealed sources of long-lived gamma-emitting radionuclide (e.g.^{137}Cs, ^{133}Ba, Mock ^{131}I, ^{40}K) of appropriate geometry can be used and are commercially available.

8.2.4 Energy calibration

8.2.4.1 General

Since the energy calibration of a scintillation spectrometer is temperature dependent, the temperature should be recorded before the test sample measurements and the energy calibration should be corrected by temperature variations. When the temperature is not measured, the energy calibration should be carried out immediately before the test sample measurements. Energy calibration shall be carried out in accordance with IEC 61453 or other appropriate method (see 8.2.4.2 and 8.2.4.3). The energy calibration of the spectrometer shall be established over the desired energy region at a fixed gain. Energy region ranging from 100 keV to 1 500 keV would be sufficient to perform the tests in accordance with this document.

Temperature compensating equipment is also commercially available, which might not be required test before each use. In this case energy calibration should be performed as a test before first use and a periodic test.

8.2.4.2 Single channel analyser counting

Perform the energy calibration of the system over the defined energy region at a fixed gain. Using reference or check sources of known energy, determine the relationship between the gamma-ray energies and the corresponding settings of the discriminator.

Measure the count rate as a function of the lower level discriminator setting at increments of no more than 2 % of the energy range of interest.

The window width should be constant and approximately equal to the increments of the lower level discriminator setting. The centre of the window width corresponding to the highest count rate may be assumed to be the centre of the total absorption peak. Fitting a function around the count rate maximum value can help in assessing the centre of the total absorption peak.

The energy calibration shall be determined for each amplifier gain and photomultiplier high-voltage setting used.

Energy calibration is performed at the energy gamma ray(s) of the radionuclides for which tests are performed or using radionuclides with gamma rays that cover the energy region of interest. It is recommended to use reference sources with single-line or double-line emitting radionuclides for the energy calibration.

8.2.4.3 Multichannel analyser counting

Perform the energy calibration of the system over the defined energy region at a fixed gain. Using reference or check sources, record the spectrum containing the total absorption peaks covering the gamma-ray energy region of interest.

Determine the channel numbers which correspond to two gamma-ray energies that are near the extremes of the energy region of interest.

The energy calibration shall be determined for each amplifier gain and photomultiplier high-voltage setting used.

Determine the slope and the intercept of the energy calibration curve.

For most applications, such a linear energy calibration curve is adequate, except when dealing with the low-energy region.

8.2.5 Detection efficiency calibration

8.2.5.1 General

The detection efficiency calibration for a sample with a known radionuclide shall be conducted using the calibration source described in 8.2.2. To avoid errors due to pulse pile-up, the activity of the calibration source and the geometry shall be selected to avoid that the counting time of the measurement differs by more than 10 % from the live counting time (i.e. the dead time shall be less than 10 %). A pulse pile-up rejection circuit may also be used.

As another approach calculation of detection efficiency by Monte Carlo simulation or other modelling technique is also available. Such numerical models are sensitive to input parameters such as the detector dimensions, and therefore shall be checked using at least one reference material which emit gammas covering the energy range of interest. If a discrepancy is found between efficiency calculated using the model and the reference source, the discrepancy shall be investigated and corrections applied to the numerical model.

The check source reading shall be recorded after each calibration. This reading can be later used when routinely checking the operability of the instrument. ^{137}Cs solid sealed sources of appropriate geometry mentioned in 8.2.3 can be used.

A source jig should be provided to hold the source at a fixed position relative to the detector.

Type test information for detection efficiencies from manufacturers would be useful to start the tests immediately in the emergency situation. The detection efficiency calibration also should be performed by users as periodic tests to confirm continued good operation. Function checks that are performed before each use using check source should be carried out by users.

8.2.5.2 Total spectrum counting / single channel analyser counting

Set the lower-level discriminator to a value that ensures that the following conditions are satisfied:

1) the gamma rays from 50 keV to 1 500 keV are being counted for total spectrum counting, or entire energy range of the gamma rays (365 keV for ^{131}I, 605 keV and 796 keV for ^{134}Cs, and 662 keV for ^{137}Cs) to be analysed are being counted for SCA counting.

2) the system response is insensitive to small changes in discriminator setting, or upper and lower limits setting;

3) any significant electronic noise is below the counting threshold and the upper-limit discriminator is set to the highest possible setting for total spectrum counting.

Accumulate counts using a solution or solid calibration source with a reference activity A_i of each radionuclide, i, in the selected counting geometry. Marinelli beakers are commonly used as they usually provide good positional reproducibility. At least 10 000 net counts should be accumulated if the background is relatively insignificant.

The instrument response to the check source shall be recorded following the calibration.

The response R_i for the specific radionuclide, i, is calculated as follows:

$$R_i = \frac{n_{Ns,E} / t_s}{A_i} \tag{2}$$

where A_i is the activity of the specific nuclide, i, in the reference source.

The number of net counts $n_{Ns,E}$ is calculated using Formula (3):

$$n_{Ns,E} = n_{gs,E} - n_{gb,E} \tag{3}$$

8.2.5.3 Multichannel analyser counting

8.2.5.3.1 Counting efficiency calibration

Measure gamma-ray spectra using reference sources in the selected geometry. At least 10 000 net counts should be accumulated in each total absorption gamma-ray peak of interest.

Record the live counting time.

For each radionuclide of the reference source, determine the net counts in the total absorption gamma-ray peaks of interest. When using MCA systems, the baseline of background under each peak in the calibration spectrum shall be subtracted to determine the net peak areas. The background peaks areas (normalized to equal the live counting time) are subtracted from the gross peak areas, with proper consideration of uncertainties, in order to get the net peak areas of the reference source.

Correct the reference source gamma-ray emission rate for decay from the time of calibration to the time at which the reference source is measured.

Calculate the total absorption peak efficiency (ε_E) at gamma-ray energy, E, as follows:

$$\varepsilon_E = \frac{n_{\mathrm{Ns},E} / t_{\mathrm{s}}}{A \cdot P_E} \tag{4}$$

Determine the full energy peak efficiency calibration data at energies for which reference sources are not available, by plotting and fitting an appropriate mathematical function to the values for the full energy peak efficiency versus gamma-ray energy.

8.2.5.3.2 Radionuclide-specific counting efficiency calibration

The efficiency for each specific radionuclide, i, at energy, E, is determined using Formula (5):

$$\varepsilon_{i,E} = \frac{n_{\mathrm{Ns},E} / t_{\mathrm{s}}}{A_i} \tag{5}$$

where A_i is activity of the specified nuclide in the reference source.

8.3 Validation of the screening level

A reference source having an activity equal to half of the OIL value or more is used.

Place the source in the spectrometer and record a spectrum or accumulate counts. The counting time shall be set at the same count time selected for the sample tests. The measurement should be repeated at least five times.

The areas of the peaks in the reference sources shall be determined using the same algorithm used for test samples.

Calculate the activity concentration c_{SL} (Bq·kg^{-1}) in the sample:

$$c_{A,\mathrm{SL}} = \frac{\left(\frac{n_{\mathrm{gSL},E}}{t_{\mathrm{SL}}} - \frac{n_{\mathrm{gb},E}}{t_{\mathrm{b}}} \right)}{R_i \cdot m} \tag{6}$$

In case of the use of peak processing with a MCA system, the following formula shall be used

$$c_{A,\mathrm{SL}} = \frac{\frac{n_{N,\mathrm{SL},E}}{t_{\mathrm{SL}}}}{P_E \times \varepsilon_E \times m} \text{ or } \frac{\frac{n_{N,\mathrm{SL},E}}{t_{\mathrm{SL}}}}{\varepsilon_{i,E} \times m} \tag{7}$$

Determine the upper limit of $c_{A,\mathrm{SL}}$ according to Formula (8) and compare with $c_{A,\mathrm{RL}}$.

$$c^{\triangleright}_{A,\mathrm{SL}} = c_{A,\mathrm{SL}} + t_{k-1,\alpha} \cdot u(c_{A,\mathrm{SL}}) \tag{8}$$

If $C^{\triangleright}_{A,\mathrm{SL}} < C_{A,\mathrm{RL}}$, the activity concentration $c_{A,\mathrm{SL}}$ can be warranted as the screening level under the stated measurement conditions (count time, sample size, container, instrument, etc.). An example is shown in Annex A.

8.4 Screening procedure

8.4.1 Total spectrum counting / Single channel analyser counting

Using the apparatus settings according to 8.2.5.2, place a blank sample in the same counting geometry used for the calibration and the test sample measurement.

A blank sample of water shall be used when measuring liquid foodstuff samples such as water, milk, juices, wine. For solid test samples, a solid blank sample with a similar density shall be used. For low density test samples, there is no need for a blank sample.

Accumulate enough counts to obtain the statistical level of accuracy requested at Clause 5. The background and the test sample shall be measured using the same count time.

In accordance with ISO 11929, the decision threshold and detection limit are determined using the background activity at a known or pre-set counting time. The detection limit shall not exceed the screening value.

Obtain the net count rate for the test sample by subtracting the background level count rate from the total count rate of the test sample.

Calculate the activity concentration C_A (Bq·kg^{-1}) in the test sample using the following Formula (9):

$$C_A = \frac{n_{N,E} / t_g}{R_i \cdot m} \tag{9}$$

where R_i is the response obtained by Formula (2).

NOTE When measuring water, “volume in litres” is an alternative for “mass in kilograms”.

The measurement result c_A cannot be directly interpreted as an activity concentration of a specific radionuclide because it may contain contributions from other radionuclides as mentioned in Clause 4. Therefore the activity concentration c_A is considered as the gross activity concentration and thus may overestimate the radionuclide activity concentration.

8.4.2 Multichannel analyser counting

Using the instrument settings described in 8.2.5.3 place the test sample in the same counting geometry used for the efficiency calibration reference source. Accumulate enough counts in the gamma-ray spectrum to obtain the statistical level of accuracy desired as specified in Clause 5. Record the live time counting interval. Search and identify the gamma-ray peaks present by the use of the energy calibration data obtained according to 8.2.4.3.

Obtain the net count rate in each total absorption gamma-ray peak of interest by dividing the net counts by the live counting time.

From the identified gamma-ray energies and other information available, identify the radionuclides present in the test sample. When calculating the activity concentration c_A (Bq·kg^{-1}) of a specific nuclide, the number of gamma-rays emitted per decay is required for each energy, the test sample mass and other corrections:

$$C_A = \frac{n_{N,E} / t_g}{\varepsilon_E \cdot P_E \cdot m \cdot f_E} \tag{10}$$

or

$$C_A = \frac{n_{N,E} / t_g}{\varepsilon_{i,E} \cdot m \cdot f_E} \tag{11}$$

where f_E is the correction factor considering all necessary corrections.

NOTE When measuring water, “volume in litres” is an alternative for “mass in kilograms”.

The measurement result C_A could be directly considered as the activity of a specific radionuclide provided that external background contributions are properly determined and considered. If such consideration has been omitted due to unavoidable contribution from natural nuclides or still remained short-lived nuclide, C_A is considered to be the gross activity concentration and may be an overestimate of the radionuclide activity concentration.

8.4.3 Effect of sample density

As the density of the sample increases, self-shielding of gamma-rays within a large volume sample increases and the peak detection efficiency decreases. A correction for this effect can be applied using Monte Carlo simulation, a mathematical model, experimental verification or an empirical correction

Most reference sources have a density of approximately 1 g·cm^{-3} and most foodstuffs have a density less than 1 g·cm^{-3}. If the correction for self-shielding is omitted in this case, the results from the measurements over-estimate the activity in the samples; this is an acceptable approach for screening (see Table 2). However, total efficiency for total spectrum counting increases as the sample density increases (due to detection of scattered radiation). This situation can result in underestimating the activity content of low density samples and a correction for self-shielding may be required. However, effects in efficiency variation for integral count rate could be neglected as compared to that for the total peak, if the difference between the densities is small.

Table 2 — Examples of the density of food samples

Food	Density g/l	Food	Density g/l
Apple	1000	Meat (beef)	1060
Asparagus	850	Milk	1030
Buckwheat flour	660	Persimmon	1000
Bread	730	Pomegranate fruit	1000
Cabbage	900	Pumpkin	1000
Carrot	750	Radish	950
Chestnut (Marron)	870	Rice	870
Chinese mushroom	840	Soybean	790
Fig	1000	Strawberry	820
Honey	1200	Sweet potato	800
Kiwi	900	Wheat flour	700

NOTE Simple machining technique was employed for sample preparation. Samples were shredded and then compressed into a 1 l Marinelli beaker.

9 Test report

The test report shall conform to ISO/IEC 17025 requirements. It shall contain the following information:

a) reference to this document, i.e. ISO 19581:2017;

b) all information necessary for the complete identification of the test sample;

c) nuclide and activity concentration in which the measured results are expressed;

d) the test result(s) obtained.

NOTE 1 Above or below screening level, which need to be defined, as many organisations will run on the sum of quotients approach, where the contribution from each nuclide, divided by its screening level, is summed. If the result is less than 1, then it can be eaten. If more than 1, it cannot.

e) mention of any relevant information likely to affect and/or explaining the results.

Depending on the customer requirements, there are different ways to present the result:

- As recommended by ISO 11929, the test result should be compared with the decision threshold. When the activity concentration c_A is below or equal to the decision threshold, a contribution from the sample has not been observed and the result of the measurement should be expressed as $\leq c_A^*$.
- If the customer requests that the activity concentration c_A is to be compared with the detection limit, then the result of the measurement can be expressed as $\leq C_A^\#$ when the results is below or equal to the detection limit.
- If the detection limit exceeds the guideline value or screening level, it shall be documented that the method is not suitable for the measurement purpose.
- When the activity concentration c_A is compared with the screening level, the result of the measurement should be expressed as $\leq c_{A,\mathrm{SL}}$ when the result is less than or equal to the screening level. The activity of the specified nuclide in a sample can be stated as less than the the guideline value.
- When activity concentration c_A is compared with the screening level, the result of the measurement should be expressed as $> c_{A,\mathrm{SL}}$ when the result exceeds the screening level. In this case the activity of specified nuclide in a sample cannot be defined below the guideline value even if the value of c_A obtained is below the reference level. In addition these results alone do not define “unacceptable” levels of activities in a sample even if c_A exceeds the reference level. Generally, when apparent activity concentration exceeds the screening level, further study (e.g. gamma-ray spectrometry using a germanium detector according to ISO 10703) is warranted.

NOTE 2 For example all operating details not specified in this document, or regarded as optional, together with details of any incidents which have influenced the test result(s) are intended to be stated.