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Water quality — Radon-222 —

Part 2:
Test method using gamma-ray spectrometry

Qualité de l'eau — Radon 222 —

Partie 2: Méthode d'essai 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, www.iso.org/directives.

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The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 3, *Radioactivity measurements*.

ISO 13164 consists of the following parts, under the general title *Water quality — Radon-222*:

- *Part 1: General principles*
- *Part 2: Test method using gamma-ray spectrometry*
- *Part 3: Test method using emanometry*

The following part is under preparation:

- *Part 4: Test method using two-phase liquid scintillation counting*

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Introduction

Radioactivity from several naturally occurring and human-made sources is present throughout the environment. Thus, water bodies (surface waters, groundwaters, sea waters) can contain radionuclides of natural and human-made origin.

- Natural radionuclides, including potassium-40, and those of the thorium and uranium decay series, in particular radium-226, radium-228, uranium-234, uranium-238, lead-210, can be found in water for natural reasons (e.g. desorption from the soil and wash-off by rain water) or releases 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, curium), tritium, carbon-14, strontium-90 and gamma-emitting radionuclides can also be found in natural waters as they can be authorized to be routinely released into the environment in small quantities in the effluent discharged from nuclear fuel cycle facilities and following their used in unsealed form in medicine or industry. They are also found in water due to the past fallout of the explosion in the atmosphere of nuclear devices and the accidents at Chernobyl and Fukushima.

Drinking-water can thus contain radionuclides at activity concentration which could present a risk to human health. In order to assess the quality of drinking-water (including mineral waters and spring waters) with respect to its radionuclide content and to provide guidance on reducing health risks by taking measures to decrease radionuclide activity concentrations, water resources (groundwater, river, lake, sea, etc.) and drinking water are monitor for their radioactivity content as recommended by the World Health Organization (WHO).

Standard test methods for radon-222 activity concentrations in water samples are needed by test laboratories carrying out such measurements in fulfillment of national authority requirements. Laboratories may have to obtain a specific accreditation for radionuclide measurement in drinking water samples.

The radon activity concentration in surface water is very low, usually below 1 Bq l^{-1}. In groundwater, the activity concentration varies from 1 Bq l^{-1} up to 50 Bq l^{-1} in sedimentary rock aquifers, from 10 Bq l^{-1} up to 300 Bq l^{-1} in wells, and from 100 Bq l^{-1} up to 1 000 Bq l^{-1} in crystalline rocks. The highest activity concentrations are normally measured in rocks with high concentration of uranium (Reference [7]).

High variations in the activity concentrations of radon in aquifers have been observed. Even in a region with relatively uniform rock types, some well water may exhibit radon activity concentration greatly higher than the average value for the same region. Significant seasonal variations have also been recorded (see Annex A).

Water may dissolve chemical substances as it passes from the soil surface to an aquifer or spring waters. The water may pass through or remain for some time in rock, some formations of which may contain a high concentration of natural radionuclides. Under favourable geochemical conditions, the water may selectively dissolve some of these natural radionuclides.

Guidance on radon in drinking-water supplies provided by WHO in 2008 suggests that controls should be implemented if the radon concentration of drinking-water for public water supplies exceeds 100 Bq l^{-1}. It also recommended that any new, especially public, drinking-water supply using groundwater should be tested prior to being used for general consumption and that if the radon concentration exceeds 100 Bq l^{-1}, treatment of the water source should be undertaken to reduce the radon levels to well below that level (Reference [8]).

This International Standard is one of a series dealing with the measurement of the activity concentration of radionuclides in water samples.

The origin of radon-222 and its short-lived decay products in water and other measurement methods are described generally in ISO 13164-1.

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Water quality — Radon-222 —

Part 2:
Test method using gamma-ray spectrometry

WARNING — Persons using this document should be familiar with normal laboratory practice. This document 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 in accordance with this document be carried out by suitably qualified staff.

1 Scope

This part of ISO 13164 specifies a test method for the determination of radon-222 activity concentration in a sample of water following the measurement of its short-lived decay products by direct gamma-spectrometry of the water sample (see Annex A).

The radon-222 activity concentrations, which can be measured by this test method utilizing currently available gamma-ray instruments, range from a few becquerels per litre to several hundred thousand becquerels per litre for a 1 l test sample.

This test method can be used successfully with drinking water samples. The laboratory is responsible for ensuring the validity of this test method for water samples of untested matrices.

Annex B gives indication on the necessary counting conditions to meet the required sensitivity for drinking water monitoring.

2 Normative references

The following referenced documents are indispensable for the application 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 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 10703, *Water quality — Determination of the activity concentration of radionuclides — Method by high resolution gamma-ray spectrometry*

ISO 13164-1, *Water quality — Radon-222 — Part 1: General principles*

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

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

IEC 60973, *Test procedures for germanium gamma-ray detectors*

IEC 61151, *Nuclear instrumentation — Amplifiers and preamplifiers used with detectors of ionizing radiation — Test procedures*

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IEC 61452, *Nuclear instrumentation — Measurement of gamma-ray emission rates of radionuclides — Calibration and use of germanium spectrometers*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-10 and ISO 13164-1 apply.

3.2 Symbols

For the purposes of this document, the symbols defined in ISO 80000-10, ISO 13164-1, and the following apply.

A	activity of each radionuclide in calibration source, at the calibration time, in becquerels
c_A	activity concentration of radon in water, in becquerels per litre
c_A^*	decision threshold, in becquerels per litre
$c_A^\#$	detection limit, in becquerels per litre
$c_A^{\triangleleft}$, $c_A^{\triangleright}$	lower and upper limits of the confidence interval, in becquerels per litre
f_d	correction factor for the decay of radon during time interval between sampling and measuring and for the decay of the radionuclide being measured (^{214}Bi or ^{214}Pb) over the counting time, dimensionless
k_p, k_q	quantiles of the standardized normal distribution for the probabilities, p, q, respectively
$n_{b,E}$, $n_{b0,E}$, $n_{bs,E}$	number of counts in the background of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{g,E}$, $n_{g0,E}$, $n_{gs,E}$	number of counts in the gross area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
$n_{N,E}$, $n_{N0,E}$, $n_{Ns,E}$	number of counts in the net area of the peak, at energy, E, in the sample spectrum, in the background spectrum and in the calibration spectrum, respectively
P_E	probability of the emission of a gamma-ray with energy, E, of each radionuclide, per decay
t_0	background spectrum counting time, in seconds
t_g	sample spectrum counting time, in seconds
t_s	calibration spectrum counting time, in seconds
U	expanded uncertainty calculated by $U = ku(c_A)$ with $k = 2$
$u(c_A)$	standard uncertainty associated with the measurement result
V_{H_2O}	volume of test sample, in litres

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α, β	probability of the error of the first and second kind, respectively
γ	probability for the confidence interval of the activity concentration
ε_E	efficiency of the detector at energy, E
$\lambda_{^{222}\mathrm{Rn}}$	decay constant of radon-222, in reciprocal seconds
Φ	distribution function of the standardized normal distribution

4 Principle

The determination of radon activity concentration in water using direct gamma-ray spectrometry is based on the:

- collection and storage in a suitable container of a representative sample of the water at time, t;
- detection and quantification of gamma-radiation emitted by the short-lived decay products of the radon present in the water sample (see Table 1).

Table 1 — Energies and emission probability of the main X- and gamma-ray emission of short-lived ^{222}Rn decay products (Reference [5])

Radionuclide	Half-life	Energy	Probability of emission
	min	keV	%
^{214}Pb	26,8	351,93	35,60
		295,22	18,50
^{214}Bi	19,9	609,31	45,49
		1 764,49	15,28
		1 120,29	14,907
		1 238,11	5,827

The activity concentration is computed from the decay product activity measured and the volume of the sample having the radon in equilibrium with its decay products.

5 Sampling

5.1 General requirement

The sample shall be representative of the environment to be analysed at a given time.

5.2 Sampling requirement

The sampling shall be carried out in compliance with the conditions and techniques specified in ISO 5667-1, ISO 5667-3, and ISO 13164-1.

The sample shall be transferred directly into the container to be used for the gamma-spectrometric analysis.

The container is filled completely in order to avoid the presence of air in contact with the water sampled.

The container shall be filled in such a way as to avoid degassing the radon in the container sample. The sampling techniques to be used vary according to the actual situation.

When the analytical laboratory is not in charge of sampling, the laboratory shall supply the container for measurement and specify the sampling procedure to the person carrying out the sampling.

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The temperature of the water shall be measured during sampling.

It is recommended that the container and its cap be weighed before and after sampling in order to estimate the quantity of suspended material and particles in the container.

5.3 Sample volume

Experience shows that a sample volume of at least 1 l is needed for the sample to be representative of the environment to be analysed.

5.4 Container characteristics

The selection and preparation of a suitable container are important. The container shall be in accordance with the requirements of ISO 5667-3.

The container and cap used to contain the sample shall comply with the following requirements.

- They shall be made from inert materials with low gamma-ray absorption. They shall be impermeable to radon, non-hydrophobic, and conductive (in order not to adsorb radon and its decay products from the surrounding atmosphere).
- They shall not themselves emit gamma-radiation interfering with the measurements.
- They shall be shock-proof.

6 Transportation and storage

During transportation and storage, the sample shall be maintained at a temperature below that of the original water (but above 0 °C) until its measurement. The container shall be protected and tightly sealed. The container shall be packed in an appropriate manner in order to prevent any leakage.

The period of transportation and storage prior to analysis shall be as short as possible given the half-life of radon-222, the expected activity concentration and the detection limit of the measurement equipment to be used.

On arrival at the laboratory, the sample shall be maintained at a temperature below that of the original water (but above 0 °C) if it cannot be analysed immediately. The sample shall be analysed as soon as possible after radioactive equilibrium has been achieved.

Experience indicates that it is essential the time between sampling and analysis not exceed 48 h.

7 Detection

The activity concentration of the ^{222}Rn is determined from the gamma-ray emissions from ^{214}Bi and/or ^{214}Pb measured by direct gamma-spectrometry of the water sample.

The determination of the activity concentration of radon-222 in water samples shall be determined by high resolution gamma-ray spectrometry as specified in ISO 10703.

The measurement period shall start when the radon is at radioactive equilibrium with its decay products, i.e. at least 3 h after the end of the sampling.

The selection of the measurement equipment shall take into account the specifications given in IEC 60973 and IEC 61151.

The calibration shall follow the requirements of IEC 61452 using, for example, a radium-226 calibration source.

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8 Measurement procedure

The measurement procedure is as follows:

a) choice and location of the sampling point;

b) taking one or more water samples;

c) recording of the location, date, and time of the sampling;

d) establishment of a radioactive equilibrium between ^{222}Rn and its short-lived decay products (^{214}Bi, ^{214}Pb) in the sample by waiting, for an optimum count, 3 h after the sampling;

e) calibration of the spectrometer;

f) determinination of the background of the detector;

g) recording of a spectrum of the sample;

h) analysis of the spectrum and determination of the activity concentration by calculation.

9 Quality assurance and quality control programme

9.1 General

Quality control operations shall meet the requirements of ISO/IEC 17025.

9.2 Influence quantities

The determination of radon-222 activity concentration shall take into consideration the correction of its decay during the storage time prior to the measurement, as well as the duration of the measurement process.

Special precautions shall be taken to minimize the influence of quantities that can affect the measurement results.

During the sampling, it is recommended that the influence be minimized of the:

— water temperature;

— turbulence in the water;

— volume of air in the container.

During measurement, take into account the influence of the:

— ^{222}Rn present in the atmosphere at the site where the measurements are performed;

— ^{226}Ra present in the water to be analysed;

— presence of suspended material in the sample.

Wide temporal variations of the radon activity concentration can be observed in the atmosphere of the laboratory. The continuous monitoring of the ^{222}Rn activity concentration in the laboratory using a radon-measuring device is recommended, since even in well-ventilated rooms, considerable variations are possible in special weather conditions (see ISO 11665-1[1] and ISO 11665-5[2]).

The air shall be continuously renewed through an air-conditioning system to maintain a constant temperature and relative humidity.

The walls of the laboratory should be painted in a suitable material in order to limit the release of radon from material within the walls (e.g. epoxy paints) (see Reference [6]).

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A case may be added around the detector and filled with a radon-free gas (e.g. N_2, air passed through a granulated activated charcoal filter) in order to prevent any accumulation of radon in the vicinity of the detector.

When the presence of ^{226}Ra is suspected, take a second measurement of the same sample after a period equal to 10 times the half-life of ^{222}Rn (38 days). If the radon activity concentration is insignificant relative to the initial measurement result, the contribution of ^{226}Ra is considered negligible. If this is not the case, determine the activity concentration of ^{226}Ra present in the water sample.

9.3 Instrument verification

Major instrument parameters (energy calibration, efficiency calibration as a function of energy, and background spectrum) shall be periodically checked within a quality assurance programme established by the laboratory and following the manufacturer's instructions (see ISO 10703).

9.4 Method verification

Periodically verify the accuracy of the method by:

- participating in intercomparison exercises;
- analysing reference materials.

Method repeatability shall also be checked, e.g. by replicate measurements.

The acceptance limits of the tests mentioned in the preceding shall be defined.

9.5 Demonstration of analyst capability

If an analyst has not used this procedure before, a precision and bias test shall be performed by running a duplicate measurement of a reference or spiked material. Acceptance limits shall be defined by the laboratory.

A similar test shall be performed by analysts routinely using this procedure with a periodicity defined by the laboratory. Acceptance limits shall be defined.

10 Expression of results

10.1 Activity concentration

The activity concentration of radon in the water, c_A, expressed at the date and time of sampling can be obtained using Formula (1):

$$c_A = \frac{n_{\mathrm{N},E}/t_\mathrm{g}}{P_E \varepsilon_E V_{\mathrm{H_2O}} f_d} = \frac{n_{\mathrm{g},E} - n_{\mathrm{b},E}}{P_E \varepsilon_E V_{\mathrm{H_2O}} f_d t_\mathrm{g}} = \left(n_{\mathrm{g},E} - n_{\mathrm{b},E}\right)\frac{\omega}{t_\mathrm{g}} \quad (1)$$

where

$$\omega = \frac{1}{P_E \varepsilon_E V_{\mathrm{H_2O}} f_d} \quad (2)$$

The efficiency for energy, E, shall be calculated by:

$$\varepsilon_E = \frac{n_{\mathrm{Ns},E}/t_s}{P_E A} = \frac{n_{\mathrm{gs},E} - n_{\mathrm{bs},E}}{P_E A t_s} \quad (3)$$

Due to the half-life of radon, the specific activity shall be corrected by f_d in order to take into account the:

- radioactive decay of the radionuclide being measured (^{214}Bi or ^{214}Pb) during the counting time;

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— radioactive decay of radon-222 during the time between the reference instant, t = 0, and the measuring instant, $t = t_i$.

Formula (4) is used to calculate f_d:

$$f_\mathrm{d}^{-1} = \exp\left(\lambda_{222_\mathrm{Rn}} t_i\right)\left[\frac{\lambda_i t_\mathrm{g}}{1-\exp\left(-\lambda_i t_\mathrm{g}\right)}\right] \tag{4}$$

10.2 Standard uncertainty of the activity concentration

According to ISO/IEC Guide 98-3,[4] the standard uncertainty of c_A is calculated as given in Formula (5):

$$u(c_A) = \sqrt{\left(\frac{\omega}{t_\mathrm{g}}\right)^2 \left[u^2\left(n_{\mathrm{g},E}\right) + u^2\left(n_{\mathrm{b},E}\right)\right] + \left(c_A\right)^2 u_\mathrm{rel}^2(\omega)} \tag{5}$$

where the standard uncertainty of the counting time is neglected and the relative standard uncertainty of ω is calculated by:

$$u_\mathrm{rel}^2(\omega) = u_\mathrm{rel}^2\left(P_E\right) + u_\mathrm{rel}^2\left(V_{\mathrm{H_2O}}\right) + u_\mathrm{rel}^2\left(\varepsilon_E\right)$$

Taking into account Formula (3), the relative standard uncertainty of ε_E is calculated by:

$$u_\mathrm{rel}^2\left(\varepsilon_E\right) = u_\mathrm{rel}^2\left(n_{\mathrm{Ns},E}\right) + u_\mathrm{rel}^2(A) + u_\mathrm{rel}^2\left(P_E\right) = u_\mathrm{rel}^2\left(n_{\mathrm{gs},E} - n_{\mathrm{bs},E}\right) + u_\mathrm{rel}^2(A) + u_\mathrm{rel}^2\left(P_E\right) \tag{6}$$

where $u_\mathrm{rel}(A)$ includes all the uncertainties related to the calibration source: calibration certificate of the standard solution, and preparation of the calibration source and the addition of this calibration source.

The calculation of the characteristic limits according to ISO 11929[3] requires an equation for $\tilde{u}\left(\tilde{c}_A\right)$, i.e. the standard uncertainty of c_A as a function of its true value.

For a true value, $\tilde{c}_A$, it can be assumed that:

$$\frac{n_{\mathrm{g},E}}{t_\mathrm{g}} = \frac{\tilde{c}_A}{\omega} + \frac{n_{\mathrm{b},E}}{t_\mathrm{g}}$$

and with $u^2(n_\mathrm{g}) = n_\mathrm{g}$ is obtained:

$$\tilde{u}\left(\tilde{c}_A\right) = \sqrt{\omega^2\left[\frac{\left(\tilde{c}_A/\omega + n_{\mathrm{b},E}/t_\mathrm{g}\right)}{t_\mathrm{g}} + \frac{u^2\left(n_{\mathrm{b},E}\right)}{t_\mathrm{g}^2}\right] + \left(\tilde{c}_A\right)^2 u_\mathrm{rel}^2(\omega)} \tag{7}$$

10.3 Decision threshold

As specified in ISO 11929,[3] the decision threshold, c_A^*, is obtained from Formula (7) for $\tilde{c}_A = 0$. It follows that:

$$c_A^* = k_{1-\alpha}\tilde{u}(0) = k_{1-\alpha}\left(\frac{\omega}{t_\mathrm{g}}\right)\sqrt{n_{\mathrm{b},E} + u^2\left(n_{\mathrm{b},E}\right)} \tag{8}$$

α = 0,05 where $k_{1-\alpha}$ = 1,65 is often assumed by default.

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10.4 Detection limit

As specified in ISO 11929,[3] the detection limit, $c_A^{\#}$, is given by:

$$c_A^{\#} = c_A^{*} + k_{1-\beta}\tilde{u}\left(c_A^{\#}\right)$$

$$c_A^{\#} = c_A^{*} + k_{1-\beta}\sqrt{\omega^2\left[\left(\frac{c_A^{\#}}{\omega} + \frac{n_{\mathrm{b},E}}{t_\mathrm{g}}\right)/t_\mathrm{g} + \frac{u^2\left(n_{\mathrm{b},E}\right)}{t_\mathrm{g}^2}\right] + \left(c_A^{\#}\right)^2 u_{\mathrm{rel}}^2(\omega)} \qquad (9)$$

$\beta = 0{,}05$ where $k_{1-\beta} = 1{,}65$ is often assumed by default.

The detection limit may be calculated by solving Formula (9) for $c_A^{\#}$ or, more simply, by iteration using an initial approximation of $c_A^{\#} = 2c_A^{*}$.

Taking $\alpha = \beta$ gives $k_{1-\alpha} = k_{1-\beta} = k$ and the solution of Formula (9) is given by Formula (10):

$$c_A^{\#} = \frac{2c_A^{*} + \left(k^2 w\right)/t_\mathrm{g}}{1 - k^2 u_{\mathrm{rel}}^2(\omega)} \qquad (10)$$

10.5 Confidence limits

The lower, $c_A^{\triangleleft}$, and upper, $c_A^{\triangleright}$, confidence limits are calculated using Formulae (11) and (12) (see ISO 11929[3]):

$$c_A^{\triangleleft} = c_A - k_p u\left(c_A\right) \quad \text{with} \quad p = \omega\left(1 - \frac{\gamma}{2}\right) \qquad (11)$$

$$c_A^{\triangleright} = c_A + k_q u\left(c_A\right) \quad \text{with} \quad q = 1 - \frac{\omega\gamma}{2} \qquad (12)$$

where

$\omega = \Phi[y/u(y)]$ in which Φ is the distribution function of the standardized normal distribution;

$1 - \gamma$ is the probability for the confidence interval of the measurand;

ω can be assumed to be 1 if $c_A \geq 4u(c_A)$.

In this case:

$$c_A^{\triangleleft\triangleright} = c_A \pm k_{1-\gamma/2} u\left(c_A\right) \qquad (13)$$

$\gamma = 0{,}05$ with $k_{1-\gamma/2} = 1{,}96$ are often chosen by default.

11 Test report

The test report shall be in accordance with ISO/IEC 17025 requirements and shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 13164 (ISO 13164-2:2013);

b) identification of the sample;

c) measuring date and time;

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d) units in which the results are expressed;

e) test result, $c_A \pm u(c_A)$ or $c_A \pm U$, with the associated k value.

Complementary information can be provided such as the following:

f) sampling date and time;

g) sampling location;

h) probabilities α, β, and $(1 - \gamma)$;

i) the decision threshold and the detection limit — depending on the needs of the customer, there are different ways to present the result:

— when the activity concentration is compared with the decision threshold (see ISO 11929[3]), the result of the measurement shall be expressed as $\le c_A^*$ when the result is below the decision threshold,

— when the activity concentration is compared with the detection limit, the result of the measurement can be expressed as $\le c_A^\#$ when the result is below the detection limit — if the detection limit exceeds the guideline value, it shall be documented that the method is not suitable for the measurement purpose;

j) activity concentration of ^{226}Ra, if its presence is detected in the water sample;

k) mention of any relevant information likely to have affected the results.

The results are expressed in a similar format to that shown in ISO 13164-1 (see Annex B).

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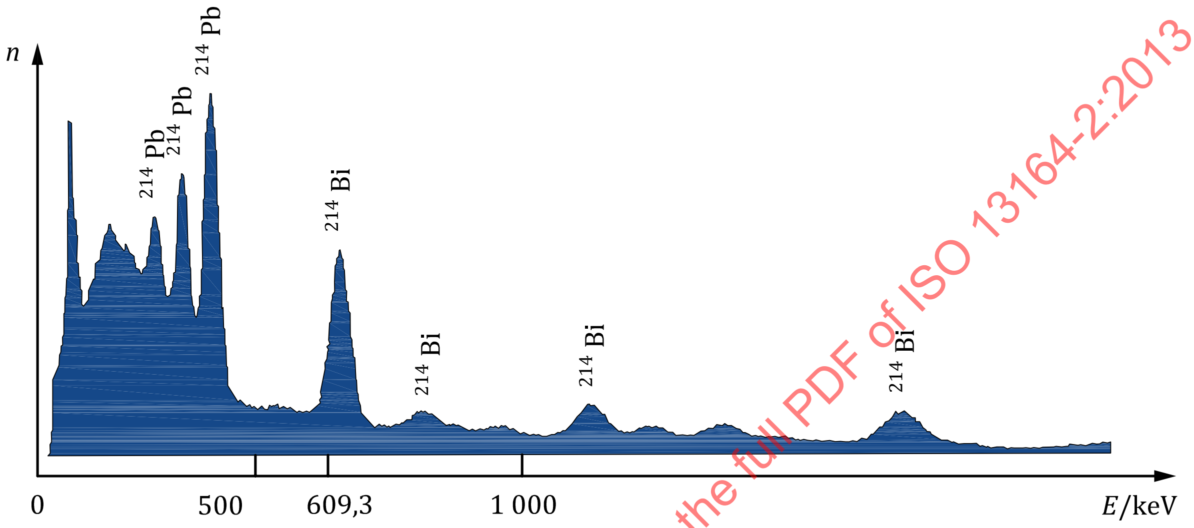
Annex A
(informative)

Sample spectra

Examples of spectra are given in Figure A.1 (sodium iodide detector) and Figure A.2 (germanium detector).



Key

n	number of counts
E	energy

Figure A.1 — Spectrum with sodium iodide detector

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