

SLOVENSKI STANDARD oSIST prEN 17216:2023

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Določanje aktivnosti radija Ra-226, torija Th-232 in kalija K-40 z gama spektrometrijo

Construction products: Assessment of release of dangerous substances - Determination of radium-226, thorium-232 and potassium-40 activity using gamma-ray spectrometry

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Messung der spezifischen Aktivität von Radium-226, Thorium-232 und Kalium-40 mittels Halbleiter-Gammaspektrometrie

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17.240	Merjenje sevanja	Radiation measurements
91.100.01	Gradbeni materiali na splošno	Construction materials in general

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Construction products: Assessment of release of dangerous substances - Determination of radium-226, thorium-232 and potassium-40 activity using gamma-ray spectrometry

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Messung der spezifischen Aktivität von Radium-226, Thorium-232 und Kalium-40 mittels Halbleiter-Gammaspektrometrie

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 351.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 17216:2023) has been prepared by Technical Committee CEN/TC 351 "Construction products: Assessment of release of dangerous substances", the secretariat of which is held by NEN.

The document is currently submitted to the CEN Enquiry.

The document will supersede CEN/TS 17216:2018.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The main changes compared to the previous edition are as follows:

- Transfer of technical specification into European Standard;
- Addition of validation data from interlaboratory validation on repeatability and reproducibility (see Clause 1, Clause 9 and Annex F); [to be completed for 2nd CEN Enquiry]
- Updating of normative and informative cross-references.

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Introduction

This document is a European Standard developed under Mandate M/366 issued by the European Commission in the framework of the "Construction Products Directive" 89/106/EEC. This document addresses the part of Mandate M/366 which provides for the preparation of horizontal measurement/test methods for the determination of the activity of the radionuclides radium-226, thorium-232 and potassium-40 in construction products using gamma-ray spectrometry. Mandate M/366 is a complement to the product mandates issued by the European Commission to CEN under the Construction Products Directive (CPD). The harmonized product standards (hEN) developed in CEN under mandates (and ETAs developed in EOTA for products or kits) specify construction product(s) as placed on the market and address their intended conditions of use.

The information produced by applying this document can be used for purposes of CE marking and evaluation/attestation of conformity. Product specification, standardization of representative sampling and procedures for any product-specific laboratory sample preparation are the responsibility of product TCs and are not covered in this document.

This document supports existing regulations and standardized practices and is based on methods described in standards such as EN ISO 10703, EN ISO 18589-2, EN ISO 18589-3 and NEN 5697. In summary, this document describes the following:

- sampling, sub-sampling and test specimen preparation;
- measurement by gamma-ray spectrometry;
- background subtraction, energy and efficiency calibration, spectrum analysis;
- calculation of activities with associated uncertainties;

reporting of results.
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Determination of the activities is done by gamma-ray spectrometry. Procedures for all stages of the analytical process are provided in this document. Although the tested sample rarely reflects a product's form under its intended conditions of use, the measured activity concentration is an intrinsic property of the product. It does not vary with the construction product's form. Consequently, the test results reflect the radiological content of the product under its intended use. The document is intended to be non-product-specific in scope. However, there are some limited elements related to the sample preparation that are product-specific.

1 Scope

This document describes a test method for the determination of the activity of the radionuclides radium-226, thorium-232 and potassium-40 in construction products using semiconductor gamma-ray spectrometry.

This document describes sampling from a laboratory sample, sample preparation, and the sample measurement by semiconductor gamma-ray spectrometry. It includes background subtraction, energy and efficiency calibration, spectrum analysis, activity calculation with the associated uncertainties or the decision threshold and detection limit calculation, and the reporting of results.

The scope of this document is not product-specific. However, there are a limited number of productspecific components, such as the preparation of the laboratory sample and drying of the test portion. The method is applicable to samples from products consisting of single or multiple constituents.

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.

EN ISO 20042:2021, Measurement of radioactivity - Gamma-ray emitting radionuclides - Generic test method using gamma-ray spectrometry (ISO 20042:2019)

3 Terms and definitions **STANDARD PREVEW**

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

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

- IEC Electropedia: available at https://www.electropedia.org/3
 - https://standards.iteh.ai/catalog/standards/sist/1cd33224-c2c3-4351-abf2-
- ISO Online browsing platform: available at <u>https://www.iso.org/obp</u>

3.1

blank

test result obtained by carrying out the test procedure with an equivalent volume of demineralised or distilled water instead of the test portion

[SOURCE: EN 16687:2023¹, 3.3.4.1]

3.2

calibration source

sample with known radioactivity concentration and material properties that corresponds to the volume and geometry of the test specimen

[SOURCE: EN 16687:2023¹, 3.3.4.2]

3.3

crushed material

sample material prepared by crushing (a part) of the laboratory sample

[SOURCE: EN 16687:2023¹, 3.3.4.4]

¹ Under preparation. Stage at the time of publication: FprEN 16687:2023.

3.4

dead time

time during spectrum acquisition (real time) during which pulses are not recorded or processed

Note 1 to entry: Dead time is given by real time minus live time.

Note 2 to entry: The time is given in seconds.

[SOURCE: EN ISO 20042:2021, 3.5]

3.5

laboratory sample

sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: EN 16687:2023¹, 3.2.2.1 – modified, Notes to entry removed]

3.6

live time

time during which pulses are processed during an acquisition (real) time

Note 1 to entry: The time is given in seconds.

Note 2 to entry: Live time is the counting time corrected for the dead time.

[SOURCE: EN ISO 20042:2021, 3.12]

3.7

test portion

amount of the test sample taken for testing/analysis purposes, usually of known dimension, mass or volume

[SOURCE: EN 16687:2023¹, 3.2.2.3 – modified, Examples removed]

3.8

test sample

sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: EN 16687:2023¹, 3.2.2.2]

Note 1 to entry: From the test sample a test portion is removed for determining the correction factor for dry mass, and one or multiple test specimen(s) are removed for radiation testing.

3.9

test specimen

test portion specially prepared for testing in a test facility in order to determine the radiation behaviour of the product under intended conditions of use

[SOURCE: EN 16687:2023¹, 3.2.2.4 – modified to read 'determine' instead of 'simulate'; Examples removed]

3.10

test specimen container

holder shaped like a beaker or a vessel that can be sealed and that is used to make determinations on the test specimen

[SOURCE: EN 16687:2023¹, 3.3.4.5]

4 Symbols and abbreviations

For the purposes of this document, the following symbols, names of quantities and units apply

Symbol	Name of quantity	Unit
$A_{i;j}$	activity of the standardized calibration source j at energy E_i	Bq
a _i	massic activity of radionuclide <i>i</i>	Bq/kg
a _{i;l}	massic activity of radionuclide <i>i</i> in the test specimen <i>l</i>	Bq/kg
$a_{i;l}^{\#}$	detection limit of radionuclide <i>i</i> of test specimen <i>l</i>	Bq/kg
$a_{i;l}^*$	decision threshold of radionuclide <i>i</i> of test specimen <i>l</i>	Bq/kg
E_i	energy used for determining radionuclide <i>i</i>	keV
f_1	attenuation correction factor	-
f_2	true-summing correction factor and site h.ai	-
f_3	dead-time correction factor	-
f_4	decay correction factor <u>oSIST prEN 17216:2023</u>	_
f5	sample versus reference source positioning/height correction	_
f_j	is the mass fraction of the constituent <i>j</i> ;	-
i, j, l	ordinals to indicate radionuclides, materials and samples	-
k	uncertainty coverage factor	-
<i>k</i> _{1-α}	uncertainty coverage factor with a default value of 1,65 at α = 0,05	_
k_{1-eta}	uncertainty coverage factor with a default value of 1,65 at β = 0,05	_
m _d	dry mass of the test portion	kg
<i>m</i> _j	mass of the material constituent <i>j</i> per m ³ of the construction product	kg
<i>m_{j;mat}</i>	mass of matrix material <i>j</i>	kg
<i>m_{j;l;stand}</i>	mass of sub sample <i>l</i> of standardized material <i>j</i>	kg
m_l	mass of the test specimen <i>l</i>	kg
m _w	fresh mass of the test portion	kg
N	corrected number of pulses in the photopeak	_
N_q	number of pulses collected in channel q	-
$\overline{N}_{\rm b}$	average number of pulses per channel before the peak	-

Symbol	Name of quantity	Unit
$\overline{N}_{\mathrm{f}}$	average number of pulses per channel after the peak	-
N _{i;j}	net number of pulses in the photopeak that corresponds to energy E_i of standardized calibration source j	_
N _{cont;1}	number of pulses that is collected in the continuum under the photopeak when counting the test specimen <i>l</i>	_
N _{cont;i;j;mat}	number of pulses that is collected in the continuum under the photopeak with energy E_i of matrix material j	_
N _{cont;i;j;k;stand}	number of pulses that is collected in the continuum under the photopeak with energy E_i of sub sample k of standardized material j	-
N _{tot;<i>i;j;</i>mat}	total number of pulses that is collected in the channels belonging to the photopeak with energy E_i of matrix material j	-
$N_{{ m tot};i;j;k;{ m stand}}$	total number of pulses that is collected in the channels belonging to the photopeak with energy E_i of sub sample k of standardized material j	-
N _{tot;1}	total number of pulses that is collected in the channels belonging to the photopeak when counting the test specimen <i>l</i>	-
n	number of test specimens	-
P _i	gamma-ray-emission probability at energy <i>E</i> _i	-
<i>p</i> ₁ , <i>p</i> ₂ , <i>p</i> ₃	free parameters used in an e-power formula	-
R _{cont;0}	counting rate of the blank that is determined from the number of pulses that is collected in the continuum under the photopeak	S ^{−1}
R _{cont;l} http:	counting rate of the test specimen <i>l</i> that is determined from the number of pulses that is collected in the continuum under the photopeak	S⁻¹
R _{cor;0}	corrected counting rate of the blank	S ^{−1}
R _{cor;i;j;spec;mat}	corrected specific counting rate of matrix material j at energy E_i	(s•kg)-1
R _{cor;i;j;spec;stand}	average specific corrected counting rate of all subsamples k of standardized material j at energy E_i	(s∙kg)-1
R _{cor;i;j;l;} spec;stand	corrected specific counting rate of sub sample <i>l</i> of standardized material <i>j</i> at energy E_i	(s∙kg)-1
R _{cor;1}	corrected counting rate of the test specimen <i>l</i> that is determined for the photopeak	S ^{−1}
R _{cor;j;v}	corrected counting rate in spectrum <i>v</i> of the tested container <i>j</i>	S ^{−1}
R _{tot;0}	total counting rate of the blank that is determined from the total number of pulses that is collected in the channels belonging to the photopeak	S ⁻¹
R _{tot;l}	total counting rate of the test specimen <i>l</i> that is determined from the total number of pulses that is collected in the channels belonging to the photopeak	S ⁻¹
S	total radon production in the building material	Bq/s
t	live time	s
t_0	live time of the blank	S

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Symbol	Name of quantity	Unit
t _j	live time of the calibration source <i>j</i>	s
<i>t_{j;mat}</i>	live time of matrix material <i>j</i>	s
<i>t_{j;l;stand}</i>	live time of sub sample <i>l</i> of standardized material <i>j</i>	s
t _l	live time of the test specimen <i>l</i>	s
$t_{ m L}$	live time when measuring the radon leakage of the beaker	s
t_w	live time of the gas sample in spectrum <i>w</i>	S
u _{p1}	uncertainty in the free parameter p_1 of the radon leakage test	S ⁻¹
<i>u_{i;ext}</i>	external uncertainty of the mass activity of radionuclide <i>i</i>	Bq/kg
<i>u_{i;int}</i>	internal uncertainty of the mass activity of radionuclide <i>i</i>	Bq/kg
<i>U_{i;l;R}</i>	uncertainty of the massic activity of radionuclide <i>i</i> from counting of the test specimen <i>l</i>	Bq/kg
<i>u</i> _{<i>i</i>;R}	uncertainty of the massic activity of radionuclide <i>i</i> from counting of the test specimen(s)	Bq/kg
U _{i,tot}	total uncertainty of the massic activity of radionuclide <i>i</i>	Bq/kg
V	sample volume	m ³
v	spectrum number, ascending from 1 to 10	-
Wi;l	calibration factor for energy E_i for the conditions used in testing the test specimen l	Bq∙s/kg
α	probability of a first order error with a default value of 0,05	-
β	probability of a second order error with a default value of 0,05	-
$\mathcal{E}_{i;j}$	radionuclide-specific counting efficiency for energy E_i and the standardized calibration source j	(Bq∙s)-1
$\mathcal{E}_{i;l}$	radionuclide-specific counting efficiency for energy E_i and a counting sample with mass m_i	(Bq∙s)-1
η	correction factor for dry mass of the test specimen	-
$\lambda_{ m L}$	radon leakage rate (of the test specimen container)	S ^{−1}
$\lambda_{ m Rn}$	decay constant of radon-222 NOTE $\lambda_{Rn} = 2,1 \times 10^{-6} \text{ s}^{-1}.$	S ⁻¹
$v_{i;j; ext{ext}}$	relative external uncertainty of the corrected specific counting rate of standardized material j at energy E_i	-
$oldsymbol{ u}_{i;j; ext{int}}$	relative internal uncertainty of the corrected specific counting rate of standardized material j at energy E_i	_
$ u_{ m L}$	relative uncertainty due to radon-222 leakage from the test specimen container	_
$v_{i;w}$	relative uncertainty of the activity of radionuclide <i>i</i> associated with the term <i>w</i>	_
$v_{i;\epsilon}$	relative uncertainty in the counting efficiency of radionuclide <i>i</i>	_

5 Principles of the test method

The activity of the gamma-ray emitting radionuclides in construction products are determined using gamma-ray spectrometry. Activity content is a material property independent from the physical form of a construction product.

The activity of gamma-ray emitting radionuclides present in the test specimen is based on the analysis of the energies and the peak areas obtained from the full-energy peaks of the gamma-ray lines in the spectrum.

The test method requires accurate energy and efficiency calibration. Calibration methods presently used in the laboratories can be applied, these shall be accompanied with a determination of the uncertainty. Selected gamma-ray lines are specified to determine the relevant radionuclides.

The activity is determined by measuring a sample held in a container with a known geometry. This determination, requiring as it does a test specimen of granular material, will only rarely reflect a product's form under its intended conditions of use. Nevertheless, as the massic activity is an intrinsic material property, it will reflect the massic activity under its intended conditions of use.

For radium-226 and thorium-232 the activity is determined using a progeny nuclide, while for potassium-40 the activity is determined directly. In case the activity is determined using a progeny nuclide, a secular equilibrium between the progeny nuclide and its originating nuclide shall be established. To reach equilibrium between radium-226 and its progenies, the test specimen is stored in a radon-tight container for a period of at least three weeks.

Despite the required waiting time of three weeks a disequilibrium in the thorium-232 decay chain can be present. Such disequilibrium is caused by different dissolution ratios between thorium and radium, in combination with its particular hydrogeological history or industrial processing. In case of such disequilibrium the thorium-232 activity is approximated by the activity of radium-228 or thorium-228.

NOTE 1 Thorium-232 with a half-life of $1,41 \times 10^{10}$ years is the parent nuclide of the thorium decay chain. Thorium-232 emits a 63,81 keV gamma ray with an emission probability of 0,263 %. It overlaps with the 63,28 keV gamma ray emitted by thorium-234 with an emission probability of 4,1 %. Thus thorium-232 cannot be determined directly by gamma-ray spectrometry. Determination through its progeny radionuclides actinium-228, lead-212 and thallium-208 can be performed correctly only if these radionuclides are in radioactive equilibrium with thorium-232.

NOTE 2 Where the activity of thorium-228 and radium-228 is significantly different, alternative measurement techniques or procedures to determine the thorium-232 activity more accurately are available but are outside the scope of this document.

6 Sampling and sample preparation

6.1 Sampling scheme

A flowchart of the sampling is presented in Figure 1 in support of the relevant definitions given in Clause 3.