SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17216:2018

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Določevanje koncentracije aktivnosti radija Ra-226, torija Th-232 in kalija K-40 v gradbenih proizvodih z gama spektrometrijo

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

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Messung von Aktivitätskonzentrationen von Gammastrahlung

Produits de construction - Evaluation de l'émission de substances dangereuses - Mesurage des concentrations de radioactivité des rayonnements gamma

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English Version

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

Produits de construction - Evaluation de l'émission de substances dangereuses - Détermination de l'activité du radium-226, du thorium-232 et du potassium-40 dans les produits de construction par spectrométrie gamma

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Messung von Aktivitätskonzentrationen von Gammastrahlung

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

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

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European foreword

This document (FprCEN/TS 17216:2018) 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.

This document is currently submitted to the Vote on TS.

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

Introduction

This document is a Technical Specification 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 concentrations 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 Technical Specification 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 Technical Specification.

This Technical Specification supports existing regulations and standardized practices, and is based on methods described in standards, such as ISO 10703 [1], ISO 18589-2 [2], ISO 18589-3 [3] and NEN 5697 [4]. In summary, the Technical Specification describes the following:

— sampling, sub-sampling and test specimen preparation;

— measurement using gamma-ray spectrometry;

— background subtraction, energy and efficiency calibration, analysis of the spectrum;

— calculation of activity concentrations with associated uncertainties;

— reporting of test results.

Determination of the activity concentration is based on the principles of gamma-spectrometry, and procedures for all stages of the testing are provided in this document. Although the tested material sample rarely reflects a product's form under its intended conditions of use, the measured activity concentration is an intrinsic property of the material, which does not vary with the construction product's form. Consequently, the test results reflect the radiation behaviour of the product under its intended use. In addition, the Technical Specification is intended to be non product-specific in scope, with only a limited number of product-specific elements.

1 Scope

This document describes a test method for the determination of the activity concentrations 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, analysis of the spectrum, calculation of the activity concentrations with the associated uncertainties, the decision threshold and detection limit, and reporting of the results. The preparation of the laboratory sample from the initial product sample lies outside its scope and is described in product standards.

This document is intended to be non product-specific in scope, however, there are a limited number of product-specific elements 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 material components.

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 16687:2015, *Construction products - Assessment of release of dangerous substances - Terminology*

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 IEC Guide 98-3, *Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM, 1995)*

3 Terms and definitions

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

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

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

3.1
blank
volume of demineralized or distilled water that corresponds to the volume and geometry of the test specimen

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 16687:2015, 4.4.2]

3.3
composite sample
sample that consist of two or more material components, put together in appropriate portions, from which the mean value of a desired characteristic may be obtained

[SOURCE: EN 16687:2015, 3.4.1; modified to read 'material components' instead of 'increments']

3.4
crushed material
sample material prepared by crushing a portion of the laboratory sample

[SOURCE: EN 16687:2015, 4.4.4]

3.5
dead time
time during which the measurement system is actually processing the signal and is not able to accept the next signal

[SOURCE: EN 16687:2015, 4.4.3]

3.6
laboratory sample
sample or sub-sample(s) sent to or received by the laboratory

[SOURCE: EN 166872015, 3.2.1]

Note 1 to entry: When the laboratory sample is further prepared by subdividing, cutting, sawing, coring, mixing, drying, grinding, and curing or by combinations of these operations, the result is the test sample. When no preparation of the laboratory sample is required, the laboratory sample is the test sample. A test portion is removed from the test sample for the performance of the test/ analysis or for the preparation of a test specimen.

Note 2 to entry: The laboratory sample is the final sample from the point of view of sample collection but it is the initial sample from the point of view of the laboratory.

3.7
product sample
construction product taken in whole or in part at the factory, on the market or on the construction site representative of the construction product

[SOURCE: EN 16687:2015, 3.1.4]

3.8
test portion
amount of the test sample taken for testing/ analysis purposes, usually of known weight or volume

[SOURCE: EN 16687:2015, 3.2.3]

3.9
test sample
sample, prepared from the laboratory sample from which test portions are removed for testing or for analysis

[SOURCE: EN 16687:2015, 3.2.2]

3.10
test specimen
test portion specially prepared for testing in a test facility in order to simulate the radiation behaviour of the product under intended conditions of use

[SOURCE: EN 16687:2015, 3.2.4]

3.11
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 16687:2015, 4.4.5]

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, b, c	free parameters used in an e-power formula	–
$A_{i;j}$	the activity of the standardized calibration source *j* at energy E_i	Bq
b	ordinal	–
$\overline{C}_i$	the average activity concentration of radionuclide *i*	Bq/kg
$C_{i;k}$	the activity concentration of radionuclide *i* in the test specimen *k*	Bq/kg
$C^{\#}_{i;k}$	is the detection limit of radionuclide *i* of test specimen *k*	Bq/kg
$C^{*}_{i;k}$	is the decision threshold of radionuclide *i* of test specimen *k*	Bq/kg
E_i	energy used for determining radionuclide *i*	keV
f	ordinal	–
i	ordinal	–
j	ordinal	–
k	ordinal	–
k	the uncertainty coverage factor	–
$k_{1-\alpha}$	the uncertainty coverage factor with a default value of 1,65 at α = 0,05	–
$k_{1-\beta}$	the uncertainty coverage factor with a default value of 1,65 at β = 0,05	–
$m_{j;\mathrm{mat}}$	the mass of matrix material *j*	kg
$m_{j;k;\mathrm{stand}}$	the mass of sub sample *k* of standardized material *j*	kg
m_k	the mass of the test specimen *k*	kg
n	number of test specimens	–
N	the corrected number of pulses in the photopeak	–
N_q	the number of pulses collected in channel *q*	–
$\overline{N}_\mathrm{b}$	the average number of pulses per channel before the peak	–

Symbol	Name of quantity	Unit
$\overline{N}_{\mathrm{f}}$	the average number of pulses per channel after the peak	–
$N_{i;j}$	the net number of pulses in the photopeak that corresponds to energy E_i of standardized calibration source *j*	–
$N_{\mathrm{cont};k}$	the number of pulses that is collected in the continuum under the photopeak when counting the test specimen *k*	–
$N_{\mathrm{cont};i;j;\mathrm{mat}}$	the number of pulses that is collected in the continuum under the photopeak with energy E_i of matrix material *j*	–
$N_{\mathrm{cont};i;j;k;\mathrm{stand}}$	the number of pulses that is collected in the continuum under the photopeak with energy E_i of sub sample *k* of standardized material *j*	–
$N_{\mathrm{tot};i;j;\mathrm{mat}}$	the total number of pulses that is collected in the channels belonging to the photopeak with energy E_i of matrix material *j*	–
$N_{\mathrm{tot};i;j;k;\mathrm{stand}}$	the 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*	–
$N_{\mathrm{tot};k}$	the total number of pulses that is collected in the channels belonging to the photopeak when counting the test specimen *k*	–
p	ordinal	–
q	ordinal	–
$R_{\mathrm{cont};0}$	the counting rate of the blank that is determined from the number of pulses that is collected in the continuum under the photopeak	s^{-1}
$R_{\mathrm{cont};k}$	the counting rate of the test specimen *k* that is determined from the number of pulses that is collected in the continuum under the photopeak	s^{-1}
$R_{\mathrm{cont};w}$	the counting rate in spectrum *w* that is determined from the number of pulses that is collected in the continuum under the photopeak	s^{-1}
$R_{\mathrm{cor};0}$	the corrected counting rate of the blank	s^{-1}
$R_{\mathrm{cor};i;j;\mathrm{spec;mat}}$	the corrected specific counting rate of matrix material *j* at energy E_i	$(s·kg)^{-1}$
$R_{\mathrm{cor};i;j;\mathrm{spec;stand}}$	the average specific corrected counting rate of all subsamples *k* of standardized material *j* at energy E_i	$(s·kg)^{-1}$
$R_{\mathrm{cor};i;j;k;\mathrm{spec;stand}}$	the corrected specific counting rate of sub sample *k* of standardized material *j*at energy E_i	$(s·kg)^{-1}$
$R_{\mathrm{cor};k}$	the corrected counting rate of the test specimen *k* that is determined for the photopeak	s^{-1}
$R_{\mathrm{cor};w}$	the corrected counting rate in spectrum *w* that is determined for the photopeak	s^{-1}
$R_{\mathrm{tot};0}$	the 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}
$R_{\mathrm{tot};k}$	the total counting rate of the test specimen *k* that is determined from the total number of pulses that is collected in the channels belonging to the photopeak	s^{-1}
$R_{\mathrm{tot};w}$	the total counting rate in spectrum *w* that is determined from the total number of pulses that is collected under the photopeak	s^{-1}
S	the total radon production in the building material	Bq/s
t	the counting time	s

Symbol	Name of quantity	Unit
t_0	the counting time of the blank corrected for the dead time	s
t_j	the counting time of the calibration source *j* corrected for the dead time	s
$t_{j;\mathrm{mat}}$	the counting time of matrix material *j* corrected for the dead time	s
$t_{j;k;\mathrm{stand}}$	the counting time of sub sample *k* of standardized material *j* corrected for the dead time	s
t_k	the counting time of the test specimen *k* corrected for the dead time	s
t_w	the counting time of the gas sample in spectrum *w* corrected for the dead time	s
u_a	the uncertainty is the uncertainty in the free parameter *a* of the radon-tightness test	s^{-1}
$u_{i;ext}$	the external uncertainty of the mass activity of radionuclide *i*	Bq/kg
$u_{i;ie}$	is the internal or external uncertainty of the activity of radionuclide *i*	Bq/kg
$u_{i;\mathrm{int}}$	the internal uncertainty of the mass activity of radionuclide *i*	Bq/kg
$u_{i;k}$	the uncertainty of radionuclide *i* of the test specimen *k*	Bq/kg
$u_{i,tot}$	the total uncertainty of the activity of radionuclide *i*	Bq/kg
$u_{(R\mathrm{cor};w)}$	the uncertainty of the corrected counting rate $R_{\mathrm{cor};w}$	Bq/kg
V	the volume	m^3
w	ordinal	–
α	the probability of a first order error with a default value of 0,05	–
β	the probability of a second order error with a default value of 0,05	–
$\varepsilon_{i;j}$	the radionuclide-specific counting efficiency for energy E_i and the standardized calibration source *j*	$(Bq \cdot s)^{-1}$
$\varepsilon_{i;k}$	the radionuclide-specific counting efficiency for energy E_i and a counting sample with mass m_k	$(Bq \cdot s)^{-1}$
η_k	the correction factor for dry mass of the test specimen *k*	–
λ_l	the tightness of a test specimen container	s^{-1}
λ_Rn	the decay constant of radon-222	s^{-1}
$\nu_{i;j;ext}$	the coefficient of variation of the corrected specific counting rate of sub sample *k* of standardized material *j* at energy E_i	–
$\nu_{\mathrm{i;l}}$	the coefficient of variation due to radon-222 leakage from the test specimen container	–
$\nu_{i,\mathrm{tot}}$	the total relative uncertainty of the activity of radionuclide *i*	–
$\nu_{i;\varepsilon}$	the relative uncertainty in the counting efficiency of radionuclide *i*	–
$\nu_{i;\rho}$	the relative uncertainty in the density correction of radionuclide *i*	–

5 Principles of the test method

The activity concentrations of the gamma-emitting radionuclides in construction products are determined using gamma-ray spectrometry. Activity concentration is a material-property and not a function of the physical form of a construction product.

The activity of gamma-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 lines that allow the identification and the quantification of the radionuclides.

The test method requires accurate energy and efficiency calibrations. Such calibration is performed using a calibration material with a known activity source that is similar in chemical composition and density to the materials that are to be tested. The calibration is based on a pre-selected set of photopeaks used for the determination of the activity concentration. Selected photopeaks are either emitted by the radionuclide itself or by one of its progeny nuclides.

The activity concentration is measured using a homogeneous, mostly granular, test specimen held in a container with a predefined geometry. This determination, requiring as it does a test specimen of granular material to be presented to the spectrometer, will only rarely reflect a product's form under its intended conditions of use. Nevertheless, because the activity concentration is an intrinsic material property, the test results will simulate the radiation behaviour of the product under its intended conditions of use.

For radium-226 and thorium-232 the activity concentration is determined using a progeny nuclide, while for potassium-40 the concentration is based on the photopeak from the nuclide itself. In those cases where the activity is determined using a progeny nuclide, a secular equilibrium between the progeny nuclide and its originating nuclide is necessary. To reach such equilibrium the test specimen is stored in a radon-tight container for a period of at least three weeks in order to ensure there is no degradation in the equilibrium due to a leakage of radon activity.

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, particular hydrogeological history and effects of industrial processes. In case of such disequilibrium the thorium-232 activity is approximated.

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 has a line at 63,81 keV with a very low emission probability of 0,263 % which overlaps a line of thorium-234 at 63,28 keV with a higher emission probability of 4,1 %, so that thorium-232 cannot be determined directly by gamma spectrometry. Determination through its decay radionuclides actinium-228, lead-212 and thallium-208 can be performed only if one assumes that these radionuclides are in radioactive equilibrium with thorium-232.

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

6 Sampling and sample preparation

6.1 Sampling hierarchy

A diagram of the sampling hierarchy (Figure 1) is presented and followed by a sketch with a physical description of the samples (Figure 2) in support of the relevant definitions given in Clause 3.

NOTE The test method described in this Technical Specification starts with a laboratory sample received by the laboratory. The preparation of a product sample lies outside the scope of this Technical Specification and is described in product standards.