INTERNATIONAL STANDARD

ISO 13356

Second edition
2008-06-01

Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)

Implants chirurgicaux — Produits céramiques à base de zircone tétragonal stabilisé à l'oxyde d'yttrium (Y-TZP)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 13356 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 13356:1997) which has been technically revised.

Introduction

No known surgical implant material has ever been shown to cause absolutely no adverse reactions in the human body. However, long-term clinical experience of the use of the material referred to in this International Standard has shown that an acceptable level of biological response can be expected when the material is used in appropriate applications.

Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)

1 Scope

This International Standard specifies the characteristics of, and corresponding test methods for, a biocompatible and biostable ceramic bone-substitute material based on yttria-stabilized tetragonal zirconia (yttria tetragonal zirconia polycrystal, Y-TZP) for use as material for surgical implants.

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 3611, *Micrometer callipers for external measurement*

ISO 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

ISO 18754, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity*

EN 623-2, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 2: Determination of density and porosity*

EN 623-3, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 3: Determination of grain size and size distribution (characterized by the Linear Intercept Method)*

ASTM C1499, *Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature*

ASTM E112-96, *Standard Test Methods for Determining Average Grain Size*

ASTM G136-03, *Standard Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction*

ASTM F1873-98 [1)], *Standard Specification for High-Purity Dense Yttria Tetragonal Zirconium Oxide Polycrystal (Y-TZP) for Surgical Implant Applications*

1) Standard since withdrawn.

3 Physical and chemical properties

The physical and chemical properties, when tested as specified in Clause 4, shall comply with the values specified in Table 1.

Table 1 — Limits for material properties

Property	Unit	Requirement	Test method according to subclause
Bulk density	g/cm^3	$\geqslant$ 6,00	4.1
Chemical composition: $ZrO_2 + HfO_2 + Y_2O_3$ Y_2O_3 HfO_2 Al_2O_3 Other oxides	percent mass fraction	 $\geqslant$ 99,0 $>$ 4,5 to $\leqslant$ 6,0 $\leqslant$ 5 $\leqslant$ 0,5 $\leqslant$ 0,5	4.2
Microstructure: grain size	µm	Intercept distance $\leqslant$ 0,4	4.3
Microstructure: amount of monoclinic phase		Standard deviation $<$ 0,18 $\leqslant$ 20 %	4.3.7
Strength [a]: biaxial flexure or 4-point bending	MPa	$\geqslant$ 500 $\geqslant$ 800	4.4 4.5
Cyclic fatigue limit stress at 10^6 cycles	MPa	$\geqslant$ 320	4.6
Radioactivity [b]	Bq/kg	$\leqslant$ 200	4.7
Accelerated aging: maximum amount of monoclinic phase after accelerated aging residual biaxial flexure strength residual 4-point bending strength		 $\leqslant$ 25 % $\geqslant$ 500 MPa, and decrease not more than 20 % $\geqslant$ 800 MPa, and decrease not more than 20 %	4.8

[a] Measured on a minimum of 10 samples.

[b] The radioactivity, defined as the sum of the mass activity of ^{238}U, ^{226}Ra, ^{232}Th and determined by gamma spectroscopy on the ready-to-use powder, should be equal or less than 200 Bq/kg. This value will be reviewed at the next revision of this International Standard and will be based upon the radioactivity data from implant ceramic manufacturers.

4 Test methods

4.1 Bulk density

The bulk density shall be determined in accordance with ISO 18754 or EN 623-2.

4.2 Chemical composition

The chemical compositions should be determined by ICP-OES (Inductively Coupled Plasma — Optical Emission Spectrometry), fluorescent X-ray, or atomic absorption spectrum analysis methods.

NOTE ISO 12677[1] can be used.

4.3 Microstructure

4.3.1 Principle

For describing the microstructure, the average grain size is determined by measuring the linear intercept size in accordance with EN 623-3 or ASTM E112.

4.3.2 Apparatus

The apparatus shall consist of the following items:

4.3.2.1 **Grinding and polishing devices**, for preparing plane and smooth surfaces.

4.3.2.2 **Furnace**, capable of maintaining a temperature of 1 400 °C.

4.3.2.3 **Scanning electron microscope**.

4.3.3 Preparation of test piece

Test pieces shall be prepared in accordance with EN 623-3 and the following instructions.

a) Prepare test pieces of the zirconia ceramic using methods representative of the method of production of parts for surgery, using the same precursor powder, pressing technique, pressure and firing conditions.

b) Grind one surface plane, polish it until the percentage of interpretable area is at least 90 % and thermally etch in air at a low temperature of less than 200 °C from sintering temperature. The etching conditions shall be specifically determined for each zirconia material.

c) Coat the polished surface by sputtering with a thin conductive layer, for example, gold or carbon.

NOTE A gold or a gold-platinum alloy can be used.

d) Five extra test pieces shall be made from the chosen flexure test sample. These test pieces shall be made in the same way as described for each according to 4.4.3 for the biaxial test and 4.4.5 for the 4-point bend test. The samples for microstructural analysis shall be chosen randomly from the flexure test samples.

e) Each microstructure test sample shall be cut in half. If it is a disc it shall be cut diametrically, if a bar it shall be cut lengthwise through the centre as shown in Figure 1. The piece may be sectioned further to permit it to fit into the microscope (4.3.2.3), the positions for the micrographs being those determined for the full section.

f) The surface shall then be thermally etched to produce appropriate grain relief, using typically temperatures in the range 1 300 °C to 1 400 °C for 30 min to 60 min.

g) Use

— either a low vacuum electron microscope with optics capable of discerning the microstructure on a sufficiently fine scale or

— sputter a thin conducting coating of Au, Au-Pd or C sufficient to allow for conductivity of the beam from the site

while retaining adequate feature resolution on the sample to allow analysis.

4.3.4 Procedure

Carry out the test in accordance with EN 623-3 or ASTM E112 and the following instructions.

a) Observe the microstructure using the scanning electron microscope (4.3.2.3) at a magnification sufficient to clearly delineate grain boundaries. Using either lines drawn on photomicrographs or stage movement, follow the general procedure described in EN 623-3 or ASTM E112 to measure the linear intercept sizes of at least 250 grains in total over at least three fields of view on lines sufficiently long to encompass at least 20 grains, taking random orientations of measurement. Calibrate the magnification employed using a certified graticule or grid. The micrographs should be of sufficient magnification, approximately 10 000 ×.

NOTE Alternatively a calibrated stage micrometer can be used.

b) Align the cross section or cut fraction thereof so that the side of the bottom of the part when firing is at the bottom of the micrograph.

c) Take micrographs for each of the five positions shown on the appropriate cross-section below. The micrographs should be of sufficient magnification, approximately 10 000 ×.

d) The location of the micrographs on the disc cross section on the left of Figure 1 (using the lower left hand corner as the origin of an x-y-coordinate system) are as given in Table 2 (tolerance ± 0,05 mm).

Table 2

Point	1	2	3	4	5
x	6	12	18	24	30
y	0,33	0,66	1,00	1,33	1,66

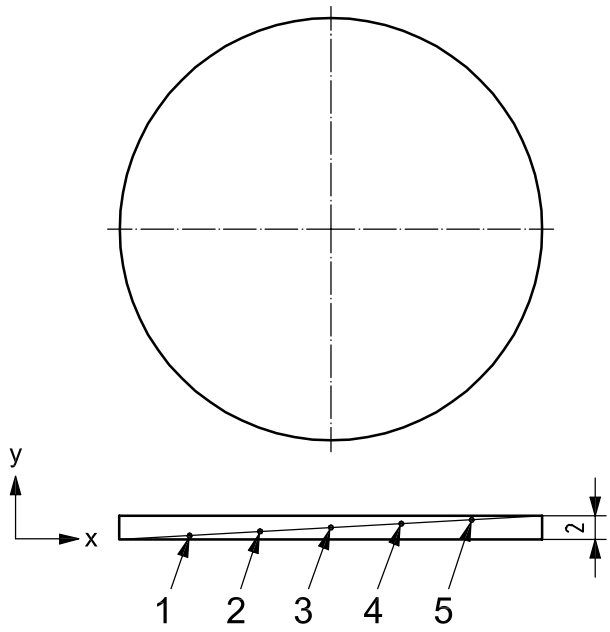
The location of the micrographs on the 4-point flexure cross section on the right of Figure 1 (using the lower left hand corner as the origin as the origin of a x-y-coordinate system) are as given in Table 3 (tolerance ± 0,05 mm).

Table 3

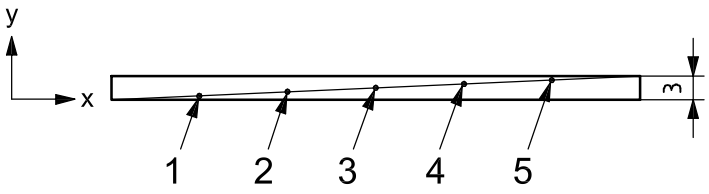
Point	1	2	3	4	5
x	7,5	15	22,5	30	37,5
y	0,5	1,0	1,5	2,0	2,5

e) For each micrograph strike no fewer than 12 lines, 4 horizontal to the diameter (bottom line), 4 at $+(60 \pm 5)$ degrees from the horizontal and 4 at $-(60 \pm 5)$ degrees from the horizontal (see Figure 1). These lines should be spread evenly across the micrograph so that as much as possible of the micrograph is represented without allowing any of the diagonal lines to be cut by the micrograph borders that are perpendicular to the diametral dimension.

f) Determine the linear intercept of each of these lines in accordance with ASTM E112 and record the intercept numbers.



a) diametral cut



b) lengthwise cut through the 45 × 4 mm surface

Figure 1 — Cuts for the cross sections of the biaxial disc (left) and 4-point flexure bar (right): bottom shows positions of micrographs to be taken

4.3.5 Calculation of results

The evaluation of the data shall be performed as follows. Determine the mean linear intercept size for each micrograph ("*micrograph mean*") in accordance with EN 623-3 or ASTM E112.

Calculate the average mean linear intercept size for test piece ("*test piece mean*") from the average of the micrograph means.

Calculate *mean value* and *standard deviation* from the 5 test piece means. These results shall be used for the test report.

4.3.6 Test report

The test report shall be established in accordance with EN 623-3.

The test report shall contain at least the following information:

a) identity of the ceramic material, details of batch number or other codes sufficient to identify the test pieces uniquely;

b) method of preparation of the test pieces, including details of the grinding and polishing procedure employed to prepare the test surfaces and of the etching procedure;

c) mean linear intercept size and its standard deviation, expressed in micrometres for each disc or bar and for the total 5 disc or bar samples;

d) at least one micrograph from the set taken to show the micro structural relief developed during the thermal etch; the micrograph need not be identified as to the sample or region from which it was taken;

e) reference to this International Standard, i.e. ISO 13356:2008.

4.3.7 Amount of monoclinic phase

The amount of monoclinic phase shall be determined using X-ray diffraction methods in accordance with the procedure set out on ASTM F1873-98, Section 4.4.

The X-ray measurement shall be conducted on the sample prepared as described in 4.3.3. b): the surface to be tested shall be in a polished state.

NOTE The method is also described in Garvie and Nicholson[3].

4.4 Biaxial flexural strength

4.4.1 Principle

A disc of the test material is placed between two coaxial rings of unequal diameter and a compressive force is applied. The force applied at fracture of the test disc and the location of the fracture are recorded and the fracture stress is calculated. This test method is standardized in ASTM C1499.

4.4.2 Apparatus

4.4.2.1 Mechanical testing machine, suitable for applying a compressive load of at least 5 kN at a nominal loading rate of (500 ± 100) N/s and equipped to record the peak force applied to an accuracy of better than 1 %. Calibration of the force-measuring device shall be performed in accordance with ISO 7500-1.

4.4.2.2 Test jig, comprising unequal diameter loading rings and having a geometry typically as shown in Figure 2. The jig shall have a support ring diameter of (30 ± 0,1) mm at the diameter of contact with the test piece, and a loading ring mean diameter of (12 ± 0,1) mm at the diameter of contact with the test piece. The radius of curvature of the test piece contact surface of the rings shall be (2,0 ± 0,2) mm. The jig shall have a means of centring the loading and support rings and the test piece on a common axis to within ± 0,2 mm. Preferably the rings should be made of hardened steel (> HV 500 or > HRC 40) in order to minimize damage or roughness caused by fracture of the test pieces.

In order to accommodate slight departures from surface flatness of the test pieces, a (0,6 ± 0,1) mm thick rubber plate with a Shore hardness of 60 ± 5 shall be placed between the support ring and the test piece, and a piece of paper shall be placed between the test piece and the loading ring.

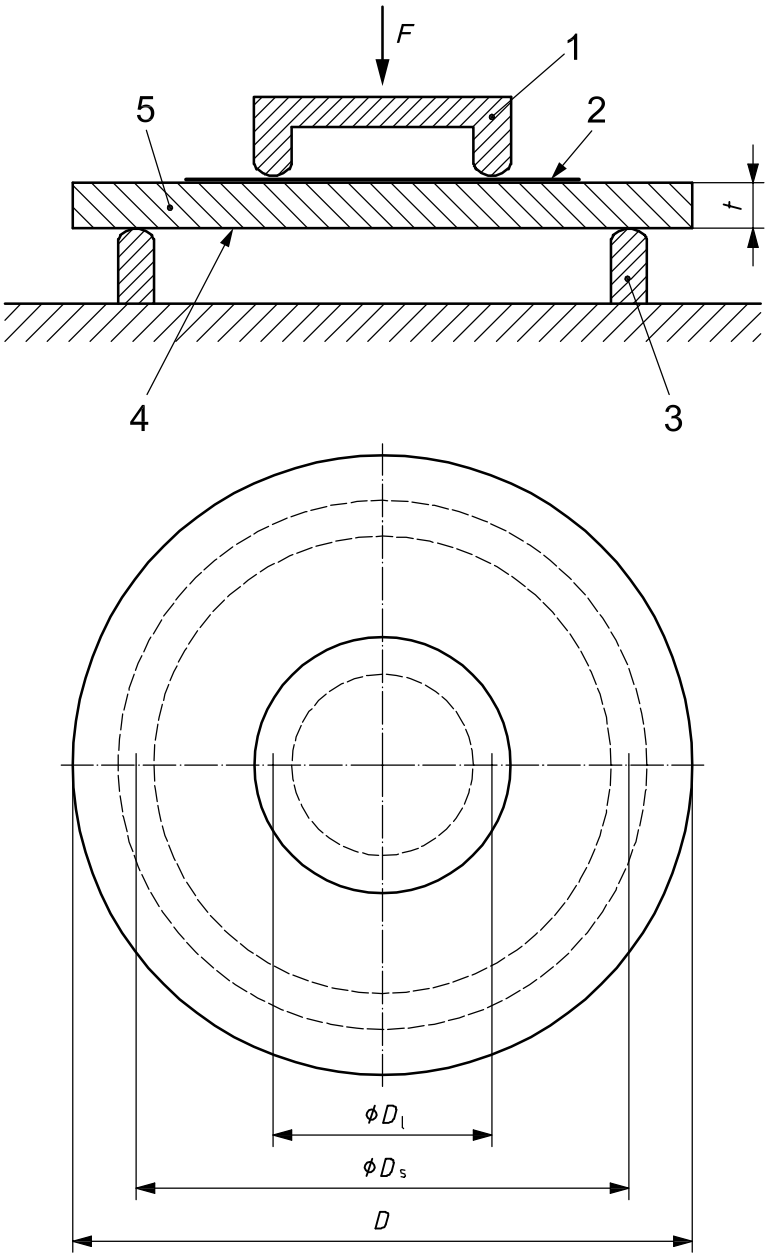
4.4.2.3 Micrometer, in accordance with ISO 3611, capable of measuring to an accuracy of ± 0,01 mm.

4.4.3 Preparation of test piece

Prepare billets or discs of the zirconia ceramic using methods representative of the method of production of parts for surgery, using the same precursor powder, pressing technique and pressure and firing conditions.

Test pieces (see Figure 2) shall be circular plates of diameter (36,0 ± 1,0) mm and thickness (2,0 ± 0,1) mm and shall be flat on the test face to closer than 0,3 mm. The surface to be tested shall be in the as-fired state.

At least 10 test pieces shall be prepared for determination of mean strength, or at least 30 test pieces if a Weibull statistical analysis is required.



Key

1	loading ring	3	support ring	5	specimen
2	paper	4	rubber plate		

Figure 2 — Schematic diagram of the biaxial flexure testing device employing concentric loading and support rings

4.4.4 Procedure

4.4.4.1 Measure the diameter of the test piece to the nearest 0,1 mm and the thickness to the nearest 0,01 mm, each in at least three random positions. Calculate the mean diameter and mean thickness.

4.4.4.2 Place the rubber sheet on the support ring of the test jig. Place the test piece on the rubber sheet, with the surface to be tested in contact with the rubber, and centre it. Place a paper disc on the top of the test piece, place the loading ring on the paper and centre it relative to the test piece and support ring.

4.4.4.3 Apply a steadily increasing compressive force, F, to the jig at a loading rate of (500 ± 100) N/s until the test piece fractures. Record the load at fracture and its location.

4.4.4.4 Inspect the fragments for evidence of the failure origin. If this is more than 0,5 mm outside the inner loading ring, note this fact in the test report (see 4.4.6). For the purposes of calculation of the fracture stress, assume failure within the inner loading ring. Do not discard the result in calculating the mean strength of the test batch.

4.4.4.5 Repeat the procedure for each test piece in the batch.

4.4.5 Calculation of results

For each test piece, calculate the nominal fracture stress, σ, in megapascals, as:

$$\sigma = \frac{3F}{2\pi \times t^2}\left[(1+\nu)\times \ln\left(\frac{D_\mathrm{s}}{D_\mathrm{l}}\right) + (1-\nu)\times\left(\frac{D_\mathrm{s}^2 - D_l^2}{2D^2}\right)\right]$$

where

F is the force applied at fracture, in newtons;

t is the mean test piece thickness, in millimetres;

D_l is the support ring mean (contact) diameter in millimetres;

D_s is the loading ring mean (contact) diameter, in millimetres;

D is the test piece diameter, in millimetres;

ν is Poisson's ratio for zirconia (assume to be 0,3).

Calculate the mean fracture stress and the standard deviation for the batch of test pieces.

4.4.6 Test report

The test report shall contain at least the following information.

a) The identity of the ceramic material, details of batch number or other codes sufficient to uniquely identify the test pieces.

b) Method of preparing the test pieces.

c) Mean value and standard deviation of the fracture stresses. If appropriate, the individual fracture stresses of the series of test pieces as well as Weibull statistical data may be given. The position of failure of test pieces shall be reported if this appears to fall outside the loading ring diameter (see 4.4.4).

d) Reference to this International Standard, i.e. ISO 13356:2008.

4.5 Four-point bending strength

The four-point bending strength shall be determined in accordance with ISO 14704 using 4-point (20 × 40) mm spans.

Calculate the individual nominal flexural strengths, the mean value and the standard deviation.

The test pieces shall be 45 mm in length (4,0 ± 0,2) mm in width and (3,0 ± 0,2) mm in thickness. The test piece is supported by two parallel rollers of diameter (5,0 ± 0,2) mm. The two rollers shall be positioned symmetrically with respect to the length of the test piece with their centres (40 ± 0,5) mm apart (outer span).

The two loading rollers shall be symmetrically located with respect to the outer roller and shall have a span of (20 ± 0,2) mm. The surface to be tested shall be in contact with the outer rollers.

4.6 Cyclic fatigue

4.6.1 Principle

The cyclic fatigue shall be determined by a four-point bend cycling fatigue test under simulated physiological conditions.

4.6.2 Apparatus

4.6.2.1 Mechanical testing machine, suitable for applying a cycling sinusoidal load. The testing machine shall be in accordance with ISO 7500-1:2004, Class 1 with an accuracy of 1 % of the maximum load or better. The machine shall have instrumentation to monitor the maximum and minimum loads, and to record the number of cycles or the elapsed time of the test.

4.6.2.2 Testing jig, in accordance with ISO 14704. The test piece is supported by two parallel rollers. Each roller shall be positioned symmetrically with respect to the length of the test piece with their centres 40,0 ± 0,1 mm apart for the outer span. The two loading rollers shall be symmetrically located with respect to the outer rollers and shall have a span of 20,0 ± 0,05 mm.

4.6.3 Preparation of test pieces

Preparation of the test pieces and the specimen geometry shall be in accordance with ISO 14704. A sample number of 5 shall be used.

4.6.4 Procedure

The test shall be performed at a cyclic rate of between 5 Hz and 20 Hz. The test shall be conducted in a physiological saline solution and at 18 °C to 40 °C. The duration of the test shall be until the specimen fractures or to not less than one million (10^6) cycles. The test shall be conducted at a peak stress of σ_{max} = 320 MPa using a peak force computed from the relationship

$$F_{max} = \frac{2bh^2\sigma_{max}}{3(S_1 - S_2)}$$

where

b is the width;

h is the height of the test piece;

S_1 is the outer span of the jig;

S_2 is the inner span of the jig.

An R-ratio of 0,1 shall be used. No test piece shall fail in less than 10^6 cycles.

4.7 Radioactivity

4.7.1 Principle

This method describes the use of gamma spectrometry for the measurement of gamma photons emitted from radio nuclides in the sample without the need to separate the radio nuclides from the sample matrix. The simultaneous detection of several gamma emitters in the sample material is carried out with a high resolution single germanium semi-conductor detector, connected to a multichannel analyser (MCA). Automatic processing of the collected data can be conveniently controlled by a computer system with selected software recommended for processing data.

4.7.2 Apparatus

4.7.2.1 Martinelli beakers, one litre size with lids.

4.7.2.2 Ball Mill.

4.7.2.3 Sieve, complying with ASTM G136-03 test.

4.7.2.4 Gamma spectrometry system, comprising low background vertical high purity germanium detector (HPGe), efficiency 20 % to 25 %; energy range 10 keV to 10 MeV; resolution (FWHM) at 1,33 MeV-1,8 keV to 2 keV. Peak to peak Compton ratio $>$ 46:1. Detector pre-amplifier contained on the detector capsule; biased high voltage power supply; linear amplifier.

4.7.2.5 Lead detector shield, with cavity adequate to accommodate the Martinelli beaker (4.7.2.1), shield wall 5 cm to 10 cm thickness lined with cadmium (1,6 mm) and copper (0,4 mm) layers.

4.7.2.6 Multichannel analyser, with at least 4 096 channels, connected to a keyboard and display screen for input and output of data and computer interaction.

4.7.2.7 Appropriate software, to provide automatic peak search, evaluation of the peak position in energy, identification of radio nuclides by use of a nuclide library, calculation of the net peak area, calculation of activity concentrations in selected units, calculation of detection limits for specific nuclides, clear description of the computing algorithms used.

4.7.3 Sample preparation

The sample shall be in the form of powder with fill density in the range 1,1 g/cm^3 to 1,3 g/cm^3 and particle size distribution in the range 0,1 µm −3 µm according to ASTM G136. If necessary crush the sample. The Martinelli beaker (4.7.2.1) shall be filled with the sample to one litre volume without any settlement.

4.7.4 Isotope identification

4.7.4.1 Energy calibration

An energy calibration of the germanium detector system shall be made by measuring mixed standards sources of known radio nuclides with well-defined energies within the range of interest. Table 4 gives a list of suitable radio nuclides. The energy calibration source shall contain at least four different gamma ray energies one of which shall be due to Cs^{137}.

The gain of the system is adjusted to position the 662 keV photopeak of Cs^{137} at about one-third full-scale. The gain of the system is adjusted to 0,5 keV/channel.

The energy calibration source is counted for sufficient time to produce a well-defined photopeak and then the system software calibration procedure is followed and the slope and intercept of the resulting line are calculated. Stability of the slope and intercept shall be checked daily using at least two different gamma energies.

Table 4 — Radionuclides suitable for energy and efficiency calibration

Nuclide	Half life	E (keV)	Photons/decay
Na^{22}	950,4 d	511,00 1 274,54	1,807 0,999 4
Sc^{46}	83,80 d	889,28 1 120,55	0,999 84 0,999 87
Cr^{51}	27,71 d	320,08	0,098 5
Mn^{54}	312,5 d	834,84	0,999 75
Co^{57}	271,84 d	122,06 136,47	0,855 9 0,105 8
Co^{60}	1 925,5 d	1 173,24 1 332,50	0,999 0 0,999 824
Cd^{109}	436 d	88,03	0,036 8
Cs^{137}	30,0 a	661,66	0,850
Ce^{139}	137,65 d	165,853	0,800
Ce^{141}	32,50 d	145,44	0,489
Hg^{203}	46,612 d	279,20	0,813
Am^{241}	420,0 a	59,54	0,360

4.7.5 Quantitative analysis

4.7.5.1 Efficiency calibration

The one litre plastic Martinelli beaker is used for this purpose. The efficiency calibration is determined by use of an appropriate certified radionuclide standard. Calibration points shall be taken every 50 keV from 60 keV to 300 keV, approximately every 200 keV from 300 keV to 1 400 keV and at least one point between 1 400 keV and 2 000 keV. The concentration of activity of various radio nuclides in the standards used for calibration shall be known to at least 3 % of total error. Time of counting shall be of sufficient duration to ensure that well-defined peaks are obtained.

4.7.5.2 Sample analysis

The spectrum of the sample contained in the Martinelli beaker filled to one litre volume is obtained by counting for sufficient duration to ensure that well-defined peaks are obtained. The spectrum of the background counting is acquired for the same period. The software analysis program is used to remove the interference from the background and use the software to determine the analytical result.

4.7.6 Expression of results

Results are expressed in becquerel per kilogram.

4.7.7 Test report

The final report shall include:

a) identification and quantification of all major nuclides present;

b) the degree of uncertainty associated with the determination;

c) the lowest detectable activity for the nuclides that exhibit statistically insignificant peaks;

d) reference to this International Standard, i.e. ISO 13356:2008.

4.8 Accelerated Aging Test

4.8.1 General

This test describes the stability of the tetragonal phase vs. hydrothermal aging. After accelerated aging, monoclinic phase content and residual strength shall be determined according to 4.3.7 and 4.4/4.5, respectively.

4.8.2 Procedure

Specimens equivalent to those described in 4.3.7 for monoclinic phase content and 4.4 or 4.5 for strength shall be used. The specimens are placed in a suitable autoclave and exposed to steam at (134 $\pm$ 2) °C under a pressure of 0,2 MPa for a period of 5 h. After this period, cool the autoclave and remove and dry the test pieces.

4.8.3 Evaluation of accelerated aging outcome

Measure monoclinic phase content as described in 4.3.7. Apply the same strength test as selected before. Determine the strength according to either 4.4 or 4.5.

Bibliography

[1] ISO 12677, *Chemical analysis of refractory products by XRF — Fused cast bead method*

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[3] GARVIE and NICHOLSON, *J. Amer. Ceram. Soc.*, Vol. **55**(6), pp. 303-305, 1972

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