SLOVENSKI STANDARD
SIST EN 16711-1:2016

01-januar-2016

Tekstilije - Določevanje kovin - 1. del: Določevanje kovin z uporabo mikrovalovnega razklopa

Textiles - Determination of metal content - Part 1: Determination of metals using microwave digestion

Textilien - Bestimmung von Metallen - Teil 1: Bestimmung von Metallen mittels Mikrowellenaufschluss

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Textiles - Détermination de la teneur en métaux - Partie 1: Dosage des métaux par minéralisation par micro-ondes

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ICS:

59.060.01	Tekstilna vlakna na splošno	Textile fibres in general

SIST EN 16711-1:2016 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16711-1

November 2015

ICS 59.060.01

English Version

Textiles - Determination of metal content - Part 1: Determination of metals using microwave digestion

Textiles - Détermination de la teneur en métaux - Partie 1: Dosage des métaux par minéralisation par micro-ondes

Textilien - Bestimmung von Metallen - Teil 1: Bestimmung von Metallen mittels Mikrowellenaufschluss

This European Standard was approved by CEN on 5 September 2015.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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

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European foreword

This document (EN 16711-1:2015) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

EN 16711, *Textiles — Determination of metal content* is composed of the following parts:

— *Part 1: Determination of metals using microwave digestion*;

— *Part 2: Determination of metals extracted by acidic artificial perspiration solution.*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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1 Scope

This European Standard specifies a procedure for determination of metals, in particular antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), mercury (Hg), nickel (Ni) in natural and man-made textiles, including coated fabrics and garment components (e.g. buttons, zips, etc.) after microwave digestion.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1233, *Water quality — Determination of chromium — Atomic absorption spectrometric methods*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885)*

EN ISO 12846, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846)*

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EN ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace (ISO 15586)*

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EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2)*

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EN ISO 17852, *Water quality — Determination of mercury — Method using atomic fluorescence spectrometry (ISO 17852)*

ISO 8288, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

3 Principle

The total metal content is determined after microwave digestion.

Analysis is performed with appropriate analytical techniques of atomic absorption, inductively coupled plasma and mass spectrometry (e.g. ICP-MS, ICP-OES, AAS, cold vapor atomic absorption spectrometry, etc.).

4 Apparatus

General laboratory equipment for analytical chemistry. In addition, the following is required:

4.1 Analytical balance, readability 0,000 1 g.

4.2 Microwave digestion system with appropriate high pressure vessels.

4.3 Appropriate digestion vessels, e.g. of polytetrafluoroethylene (PTFE).

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5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 Nitric acid, $\omega(HNO_3)$ = 65 %.

5.2 Sulphuric acid, $\omega(H_2SO_4)$ = 96 %.

5.3 Hydrofluoric acid, $\omega(HF)$ = 48 %.

5.4 Grade 2 water, complying with EN ISO 3696.

5.5 Hydrogen peroxide, $\omega(H_2O_2)$ = 30 %.

5.6 Metal standard solutions for calibration of the analytical systems complying with EN 1233, EN ISO 11885, EN ISO 12846, EN ISO 15586, EN ISO 17294-2, EN ISO 17852 or ISO 8288.

5.7 Dilution solution, 1,2 ml nitric acid (5.1) is made up to 100 ml with water (5.4).

6 Test specimen sampling

6.1 Natural and man-made textiles

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Cut from the textile specimen (with a sharp pair of scissors made of noncorrosive material, preferably of ceramics) a representative subsample of approximately 0,5 g and record the mass to the nearest 0,1 mg. If the textile article is composed of several parts of textile products, such as knitted cuffs or lining, all the different types of materials have to be tested as a representative composite specimen with equal parts of each component. Select the number of parts that can be collected as a composite specimen in relation to the detection and quantification limits of the instrumental equipment. Weigh out materials that cannot be separated as one specimen. Consider also different colours of the material for sampling. Avoid any contact with metal articles, especially in wet conditions or with corrosive surfaces.

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6.2 Garment components with painted and other similar surface coatings

For products coated with paint or a similar surface coating, remove and digest the coating separately from the substrate material. Care should be taken to remove as little of the substrate as possible. It may be necessary to add a few drops of a solvent, such as dichloromethane, to soften the paint and aid in its removal from the substrate. If used, such solvent shall be evaporated fully prior to analysis. The scraped paint shall be finely divided to help in the digestion. Scrape approximately 100 mg to 200 mg of paint from the product. If it is not possible to collect this much paint it may be necessary to combine more than one unit of such product to collect sufficient paint.

6.3 Metallic products

When preparing a sample, the laboratory shall make every effort to assure that the aliquot removed from a component part of a sample is representative of the component to be tested, and is free of contamination.

If the item is coated with paint or a similar surface coating, the coating shall be removed and analysed, separately from the base metal, for metal content as described in 6.2. Component parts of children's products, including metal jewellery items, generally weigh several grams or more, and an aliquot (with no paint or similar surface coating) will have to be obtained. Samples should be cut or ground into many small pieces to increase the rate of dissolution. If used, grinding apparatus (such as a rotary grinding tool with disposable grinding bits) must be thoroughly cleaned to prevent cross-contamination.

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6.4 Non-metal products

6.4.1 General

The general approach is to grind or cut any accessible component part of a sample into small pieces or a powder; digest an aliquot completely in nitric acid or, for siliceous products, in a combination of hot, concentrated nitric and hydrofluoric acids.

6.4.2 Ceramics, glass and crystal and other siliceous materials

Ceramic items generally weigh several grams or more, and consist of the base ceramic with a glaze and decoration fired on. The metals in ceramics are generally in the glaze or decoration. When analysing ceramics or glass, the entire item including the glaze, decoration, and ceramic base material should be ground in a cryogenic mill and 100 mg to 200 mg of the ground ceramic/glass powder weighed in an appropriate microwave vessel. If used, the grinding apparatus must be thoroughly cleaned to prevent cross-contamination.

6.4.3 Plastics, polymers (includes prints and coatings other than 6.2) and other non- siliceous materials

Cut the test specimen into small pieces. Hard to digest plastics may need to be cryomilled to get a finer powder. Ensure the milling apparatus is thoroughly clean between test specimens to avoid cross-contamination.

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7 Procedure

7.1 Digestion of natural and man-made textiles, and coated fabrics

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Perform a microwave digestion using a mixture of nitric acid and hydrogen peroxide, examples are given in Annex A. Other digestion methods may be used if the same results are obtained. Weigh out 0,1 g to 0,5 g of the finely cut specimen (record the mass to the nearest 0,1 mg) and put into a clean PTFE vessel. Add 4 ml of water (5.4) for surface wetting and subsequently 10 ml nitric acid (5.1) and 4 ml hydrogen peroxide (5.5) (see Table A.3) or 8 ml nitric acid (5.1) and 2,5 ml hydrogen peroxide (5.5) (see Table A.4). Care shall be taken to ensure that the specimen is wetted with acid, since otherwise burning of the sample material may occur. The filled volume of the PTFE vessels should not exceed 30 % to 40 % of the total volume.

Residues resulting from pigments or admixtures of titanium oxide in polyester and polyamide fibres have to be removed by filtration.

7.2 Digestion of paint and other similar surface coatings

Weigh 0,1 g to 0,2 g (record the mass to the nearest 0,1 mg) of the paint into a clean liner of the microwave digestion vessel. Carefully add at least 5 ml of concentrated nitric acid or extraction solution (made with 60 ml HNO_3 conc and 180 ml HCl conc / 1 l water grade 2.

Prepare also a digestion blank with the samples. Use a certified reference material if available (e.g. such as NIST SRM 2581 or 2582 for lead determination) to control the digestion. The result should be between ± 15 % of the true value.

Seal the vessels and load into the microwave oven according to the manufacturer's instructions. Programme the microwave digestion system using a programme with enough temperature or power to digest the sample (check this with the certified reference material).

At the end of the digestion period, remove the vessels from the microwave oven, place them in a fume hood, and allow the solutions to cool to room temperature.

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Carefully open the vessels (pay attention to the gas pressure that may be present inside the vessels). Quantitatively transfer the liquid contents of each vessel to a 50 ml volumetric flask (the size may be adjusted and will depend on the required limit). Carefully rinse each vessel with grade 2 water into the volumetric flask and bring to volume with water (be sure that the liquid temperature is near room temperature before making the volume up to the mark) . Seal each volumetric flask with a stopper and mix thoroughly.

7.3 Digestion of metallic products

Weigh out 0,1 g to 0,2 g (record the mass to the nearest 0,1 mg) piece of metal item into an appropriate microwave vessel, equipped with a controlled pressure relief mechanism.

Add 9 ml of concentrated nitric acid, and 3 ml of concentrated hydrochloric acid to each vessel. Wait for the initial reaction of the acid and sample at room temperature to be complete (to the point of no obvious fuming or bubbling) before sealing the vessels. Seal vessels in accordance with the manufacturer's instructions.

Prepare also a digestion blank with the samples. Use a certified reference material if available (e.g. such as NIST SRM 54d, 1728 for lead determination) to control the digestion. The result should be between +/- 15 % of the true value.

Seal the vessels and load into the microwave oven according to the manufacturer's instructions. Programme the microwave digestion system using a programme with enough temperature or power to digest the sample (check this with the certified reference material).

At the end of the digestion period, remove the vessels from the microwave oven, place them in a fume hood, and allow the solutions to cool to room temperature.

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Carefully open the vessels (pay attention to the gas pressure that may be present inside the vessels). Quantitatively transfer the liquid contents of each vessel to a 50 ml volumetric flask (the size may be adjusted and will depend on the required limit). Carefully rinse each vessel with grade 2 water into the volumetric flask and bring to volume with water (be sure that the liquid temperature is near room temperature before making the volume up to the mark) . Seal each volumetric flask with a stopper and mix thoroughly.

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7.4 Digestion of non-metal products

7.4.1 Ceramics, glass and crystal and other siliceous materials

Weigh out a 0,1 g to 0,5 g (record the mass to the nearest 0,1 mg) piece of ceramic, glass or crystal item into an appropriate microwave vessel equipped with a controlled pressure relief mechanism.

Add 6 ml of concentrated nitric acid and 2 ml of concentrated hydrofluoric acid to each vessel. Wait for completion of the initial reaction of the acid and the sample before sealing the vessels. Seal the vessels in accordance with the manufacturer's instructions.

Prepare also a digestion blank with the samples. Use a certified reference material if available (e.g. such as NIST SRM 89 and 610, leaded glass, for lead determination) to control the digestion. The result should be between +/- 15 % of the true value.

Programme the microwave digestion system using a programme with enough temperature or power to digest the sample (check this with the certified reference material).

At the end of the digestion period, remove the vessels from the microwave oven, place them in a fume hood, and allow the solutions to cool to room temperature.

Carefully open the vessels (pay attention to the gas pressure that may be present inside the vessels). If the analysis is to be carried out using ICP equipment, add 30 ml of 4 % (w/w) boric acid to each vessel to complex with the fluoride to protect the ICP quartz plasma torch. Quantitatively transfer the sample