INTERNATIONAL STANDARD

ISO 12830

Second edition
2019-07

Paper, board, pulps and cellulose nanomaterials — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium

Papiers, cartons, pâtes et nanomatériaux à base de cellulose — Détermination de la teneur en magnésium, calcium, manganèse, fer, cuivre, sodium et potassium soluble dans l'acide

Reference number
ISO 12830:2019(E)

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Published in Switzerland

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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.

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 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 12830:2011), which has been technically revised. The main changes to the previous edition are as follows:

— the scope has been changed to include cellulose nanomaterials in addition to paper, board and pulps;

— a definition of cellulose nanomaterial has been incorporated, along with additional instructions for sampling, sample preparation, incineration and dissolution of the residue for cellulose nanomaterials;

— additional instructions are given on how to express results when an element is not detected.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document combines the determination of the acid-soluble part of magnesium (Mg), calcium (Ca), manganese (Mn), iron (Fe), copper (Cu), sodium (Na) and potassium (K) into a single document. The scope of this document covers only the acid-soluble part of the elements.

ISO 17812[1] specifies the procedure for the determination of total magnesium, total calcium, total manganese, total iron and total copper by atomic absorption spectrometry (AAS) or by inductively coupled plasma emission spectrometry (ICP/ES).

In the context of this document, “cellulose nanomaterial” refers specifically to cellulose nano-objects (see 3.1 to 3.3). Owing to their nanoscale dimensions, these cellulose nano-objects can have intrinsic properties, behaviours or functionalities that are distinct from those associated with paper, board and pulps.

Paper, board, pulps and cellulose nanomaterials — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium

WARNING — The method specified in this document involves the use of some hazardous chemicals and of gases that may form explosive mixtures with air. Care should be taken to ensure that the relevant precautions are observed.

WARNING — The method specified in this document involves the use of nanomaterials. Care should be taken to ensure observation of the relevant precautions and guidelines for nanotechnology laboratory safety and best practices.

1 Scope

This document specifies the procedure for the determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium by atomic absorption spectrometry (AAS) or by inductively coupled plasma emission spectrometry (ICP/ES). The acid-soluble element comprises the acid-soluble part of the incineration residue, i.e. that part of the ignition residue obtained after incineration which is soluble in hydrochloric acid or nitric acid. In cases where the residue is completely soluble, the result obtained by the procedure specified in this document is a measure of the total amount of each element in the sample.

This document is applicable to all types of paper, board, pulps and cellulose nanomaterials.

The limit of determination depends on the element and on the instrument used.

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.

ISO 186, *Paper and board — Sampling to determine average quality*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 1762, *Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C*

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

ISO 7213, *Pulps — Sampling for testing*

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:

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at http://www.electropedia.org/

3.1
cellulose nanomaterial
material composed predominantly of cellulose, with any external dimension between approximately 1 nm and 100 nm, or a material having internal structure or surface structure in the *nanoscale* (3.4), with the internal structure or surface structure composed predominantly of cellulose

Note 1 to entry: The terms nanocellulose and cellulosic nanomaterial are synonymous with cellulose nanomaterial.

Note 2 to entry: Some cellulose nanomaterials can be composed of chemically modified cellulose.

Note 3 to entry: This generic term is inclusive of cellulose nano-object and cellulose nanostructured material.

Note 4 to entry: See also definitions of cellulose, nanoscale, cellulose nano-object and cellulose nanostructured material in ISO/TS 20477:2017.

[SOURCE: ISO/TS 20477:2017, 3.3.1, modified — "1 nm to 100 nm" changed to "1 nm and 100 nm"; abbreviations deleted from Note 1 to entry; Note 4 to entry added.]

3.2
nano-object
discrete piece of material with one, two or three external dimensions in the *nanoscale* (3.4)

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-1:2015, 2.5]

3.3
cellulose nano-object
nano-object (3.2) composed predominantly of cellulose

[SOURCE: ISO/TS 20477:2017, 3.3.2]

3.4
nanoscale
length range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from larger sizes are predominantly exhibited in this length range.

[SOURCE: ISO/TS 80004-1:2015, 2.1]

4 Principle

A test specimen is incinerated at 525 °C and the residue is dissolved in hydrochloric acid or nitric acid. The concentration of each element in the test solution is then determined by AAS or ICP/ES. Techniques using other types of instrumentation, such as ICP-mass spectrometry (ICP/MS), may also be used provided that they give at least the same level of precision as AAS or ICP/ES, and that they have been properly validated. The use of any such instrumentation shall also be reported.

5 Reagents and materials

5.1 General

All chemicals shall be of reagent grade or better unless otherwise indicated. Water shall be distilled or deionized, of grade 2 or better in accordance with ISO 3696.

5.2 Hydrochloric acid (HCl), 6 mol/l, trace metal grade. Dilute 500 ml of concentrated hydrochloric acid (specific gravity 1,19 g/ml) to 1 000 ml with water.

5.3 Nitric acid (HNO_3), concentrated (specific gravity 1,4 g/ml), trace metal grade.

5.4 Lanthanum chloride ($LaCl_3$), solution, ρ(La) = 50 g/l. In a 1 000 ml volumetric flask, dissolve 59 g of lanthanum oxide (La_2O_3), in small portions, in 200 ml of hydrochloric acid (5.2) and dilute to 1 000 ml with water.

This lanthanum chloride solution is used to eliminate chemical interference when determining calcium and magnesium in an air/acetylene flame. The solution is not required for use with the nitrous oxide/acetylene flame or when the ICP/ES technique is used.

5.5 Cesium chloride (CsCl), solution, ρ(Cs) = 50 g/l. In a 1 000 ml volumetric flask, dissolve 63,5 g of ultrapure cesium chloride (CsCl) in water and dilute to 1 000 ml with water.

This cesium chloride solution is used to suppress ionization of sodium and potassium in an air/acetylene flame. It is also used to suppress ionization of calcium in a nitrous oxide/acetylene flame. The solution is not required when the ICP/ES technique is used.

5.6 Standard stock solutions of each element, commercially available certified atomic absorption or atomic emission standard solutions can be used. Standard stock solutions can also be prepared as follows:

5.6.1 Magnesium, 1 000 mg/l standard solution. Dissolve 1,000 g of magnesium metal ribbon in 100 ml of 1:4 nitric acid (5.3) and dilute to 1 000 ml with water.

5.6.2 Calcium, 1 000 mg/l standard solution. Dissolve 2,497 g of primary standard calcium carbonate ($CaCO_3$) in a minimum volume of 1:4 nitric acid (5.3) and dilute to 1 000 ml with water.

5.6.3 Manganese, 1 000 mg/l standard solution. Dissolve 1,000 g of manganese metal strip or wire in a minimum volume of 1:1 nitric acid (5.3) and dilute to 1 000 ml with water.

5.6.4 Iron, 1 000 mg/l standard solution. Dissolve 1,000 g of iron metal strip or wire in 20 ml of hydrochloric acid (5.2) and dilute to 1 000 ml with water.

5.6.5 Copper, 1 000 mg/l standard solution. Dissolve 1,000 g of copper metal strip or wire in a minimum volume of 1:1 nitric acid (5.3) and dilute to 1 000 ml with water.

5.6.6 Sodium, 1 000 mg/l standard solution. Ignite a portion of anhydrous sodium sulfate (Na_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 3,089 g of dried sodium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

5.6.7 Potassium, 1 000 mg/l standard solution. Ignite a portion of anhydrous potassium sulfate (K_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 2,228 g of dried potassium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

5.7 Acetylene gas and/or **nitrogen oxide gas**, of a grade suitable for AAS. Nitrous oxide is used only when measuring calcium.

WARNING — Acetylene gas forms explosive mixtures with air.

5.8 Carrier gas, appropriate gas for the inductively coupled plasma emission spectrometer. Argon is usually recommended as a carrier gas.

6 Apparatus and equipment

6.1 General

Ordinary laboratory equipment is used. All glassware and plastic ware shall be cleaned thoroughly and rinsed with 0,1 mol/l hydrochloric acid or 10 % nitric acid, followed by reagent grade water, prior to use.

6.2 Filter paper, ash free, particle retention 20 µm to 25 µm.

6.3 Crucibles, platinum or fused silica, of minimum capacity 50 ml.

6.4 Muffle furnace, capable of maintaining a temperature of 525 °C ± 25 °C.

6.5 Balance, of capacity 100 g, with a scale division (readability) of 0,1 mg or better.

6.6 Atomic absorption spectrometer, equipped with air/acetylene and nitrous oxide/acetylene burners and with hollow cathode lamps for Mg, Ca, Mn, Fe, Cu, Na and K. Multi-element lamps can also be used.

6.7 Inductively coupled plasma/emission spectrometer.

6.8 Disposable protective gloves.

7 Sampling

7.1 General considerations

If the analysis is being made to evaluate a lot of paper, board, pulp or cellulose nanomaterial, the sample shall be selected in accordance with ISO 186 or ISO 7213, as relevant. If the analysis is made on another type of sample, report the source of the sample and, if possible, the sampling procedure. Select the material to be tested so that it is representative of the sample received. A sufficient amount of material shall be collected from the sample to allow for at least duplicate determinations. Avoid cut edges, punched holes and other parts where metallic contamination may have occurred.

Disposable protective gloves (6.8) shall be worn when handling samples to avoid contamination.

Since iron tends to have a non-homogeneous distribution, it is recommended that a composite sample be used.

7.2 Paper, board and pulp sampling

In the case of paper, board and pulp, tear or remove at least 30 g of small pieces from various parts of the sample. This amount is sufficient for the duplicate determinations described in Clause 8.

7.3 Cellulose nanomaterial sampling

In some cases, it may not be practical or possible to obtain large quantities of solid material from a cellulose nanomaterial sample. In the case of solid cellulose nanomaterials, tear or remove sufficient material for duplicate determinations as described in Clause 8, in the form of small pieces, dry powder or flakes from various parts of the sample. If the sample is in aqueous suspension form, remove sufficient material for duplicate determinations as described in Clause 8 (calculated as oven-dry, i.e. water- and moisture-free) from various portions of the aqueous suspension, and dry to give a pre-dried sample in the form of flakes, powder or other solid, which shall be mixed to homogeneity, after which the test specimen shall be obtained from the pre-dried sample. Filtration to concentrate dilute samples prior to drying is not recommended as it may result in loss of dissolved material.