SLOVENSKI STANDARD
SIST ISO 12830:2013

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Nadomešča:
SIST ISO 1830:2011
SIST ISO 777:2011
SIST ISO 778:2011
SIST ISO 779:2011
SIST ISO 9668:1996

Papir, karton, lepenka in vlaknine - Določevanje v kislini topnega magnezija, kalcija, mangana, železa, bakra, natrija in kalija

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

Papiers, cartons et pâtes -- Détermination de la teneur en magnésium, calcium, manganèse, fer, cuivre, sodium et potassium soluble dans l'acide

Ta slovenski standard je istoveten z: **ISO 12830:2011**

ICS:

85.040	Vlaknine	Pulps
85.060	Papir, karton in lepenka	Paper and board

SIST ISO 12830:2013 **en**

INTERNATIONAL STANDARD

ISO 12830

First edition
2011-11-15

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

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

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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 12830 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This first edition of ISO 12830 cancels and replaces ISO 777:2005, ISO 778:2005, ISO 779:2005, ISO 1830:2005 and ISO 9668:1990.

Introduction

This International Standard 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 standard. The scope of this International Standard covers only the acid-soluble part of the elements.

ISO 17812 specifies the procedure for the determination of total magnesium, total calcium, total manganese, total iron and total copper by atomic absorption spectrometry or by plasma emission spectrometry.

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

WARNING — The method specified in this International Standard 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.

1 Scope

This International Standard specifies the procedure for the determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium by atomic absorption spectrometry or by plasma emission spectrometry. 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. In the case where the residue is completely soluble, the result obtained by the procedure specified in this International Standard is a measure of the total amount of each element in the sample.

This International Standard is applicable to all types of paper, board and pulps.

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

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 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 Principle

A test portion is incinerated at 525 °C and the residue is dissolved in 6 mol/l hydrochloric acid. The concentration of each element in the test solution is then determined by atomic absorption or plasma emission spectrometry, as specified in this International Standard.

4 Reagents and materials

4.1 General.

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

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

4.3 Nitric acid (HNO_3), concentrated (specific gravity 1,4 g/ml).

4.4 Lanthanum chloride ($LaCl_3$), solution, $\rho(La) = 50$ 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 (4.2) and dilute to 1 000 ml with water.

NOTE This lanthanum solution is used to eliminate chemical interference when determining calcium and magnesium in an air/acetylene flame. The solution is not required when the nitrous oxide/acetylene flames or inductively coupled plasma technique (ICP technique) is used.

4.5 Cesium chloride (CsCl), solution $\rho(Cs) = 50$ g/l. In a 1 000 ml volumetric flask, dissolve 63,5 g of ultra-pure cesium chloride (CsCl) in water and dilute to 1 000 ml with water.

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

4.6 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:

4.6.1 Magnesium, 1 000 mg/l standard solution. Dissolve 1,000 g of magnesium metal ribbon in 100 ml of 1:4 nitric acid (4.3) and dilute to 1 000 ml with water.

4.6.2 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 (4.3) and dilute to 1 000 ml with water.

4.6.3 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 (4.3) and dilute to 1 000 ml with water.

4.6.4 Iron, 1 000 mg/l standard solution. Dissolve 1,000 g of iron metal strip or wire in 20 ml of hydrochloric acid (4.2) and dilute to 1 000 ml with water.

4.6.5 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 (4.3) and dilute to 1 000 ml with water.

4.6.6 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.

4.6.7 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.

4.7 Acetylene gas and/or **nitrogen oxide gas**, of a grade suitable for atomic absorption spectrometry. Nitrous oxide is used only when measuring calcium.

WARNING — Acetylene gas forms explosive mixtures with air.

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

5 Apparatus and equipment

5.1 General.

Ordinary laboratory equipment is used. All glassware and plastic ware shall be rinsed with 0,1 mol/l hydrochloric acid prior to use.

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

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

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

5.5 Balance, of capacity 100 g, accurate to 0,1 mg.

5.6 Atomic absorption spectrometer (AAS), equipped with air/acetylene and nitrous oxide/acetylene burners and with hollow cathode lamps for Mg, Ca, Mn, Fe, Cu, Na and K.

NOTE Multi-element lamps can also be used.

5.7 Inductively coupled plasma/optical emission spectrometer (ICP/OES).

5.8 Disposable protective gloves.

6 Sampling and preparation of sample

If the analysis is being made to evaluate a lot of paper, board or pulp, 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 specimens so that they are representative of the sample received. A sufficient amount of sample shall be collected to allow for at least duplicate determinations. Avoid cut edges, punched holes and other parts where metallic contamination may have occurred. Disposable protective gloves (5.8) shall be worn when handling samples to avoid contamination.

Prepare a test specimen by tearing at least 30 g of small pieces from various parts of the sample. This amount is sufficient for the duplicate determinations described in Clause 7.

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

7 Procedure

7.1 General

Although dry ignition followed by acid treatment is described in this International Standard, other dissolution methods such as wet ignition or microwave digestion using various acid combinations can also be used, provided that the results have been validated.

WARNING — For samples with high silicon content, microwave digestion with nitric acid will give lower results for magnesium and for some other elements.

7.2 Incineration of the test portion

Carry out the procedure in duplicate.

Air-dry the specimen in the laboratory atmosphere until it reaches moisture equilibrium.

