



SLOVENSKI STANDARD
SIST ISO 777:2011

01-maj-2011

Papir, karton, lepenka in vlaknine - Določevanje kalcija, topnega v kislini

Paper, board and pulp -- Determination of acid-soluble calcium

Papier, carton et pâte -- Détermination de la teneur en calcium soluble dans l'acide

Ta slovenski standard je istoveten z: ISO 777:2005

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ICS:

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

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INTERNATIONAL
STANDARD

ISO
777

Third edition
2005-05-01

**Paper, board and pulp — Determination
of acid-soluble calcium**

*Papier, carton et pâte — Détermination de la teneur en calcium 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 777 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 777:2001), of which it constitutes a minor revision. Apart from minor editorial modifications, only the title has been changed, to be consistent with ISO 1830:2005, and to make a distinction with ISO 17812 (to be published) which specifies the method to determine the total mass fraction of calcium, manganese, iron and copper.

The first edition of this International Standard included the titrimetric procedure as well as the procedure based on atomic absorption spectroscopy. In the second edition, the titrimetric procedure was deleted, as it is now seldom used, and the scope was enlarged to include paper and board in addition to pulp.

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Introduction

This International Standard corresponds to ISO 778 ^[1] and ISO 779 ^[2] in order to make it possible to perform the final measurement of all three elements on the same solution.

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Paper, board and pulp — Determination of acid-soluble calcium

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

1 Scope

This International Standard specifies the procedure for the determination of acid-soluble calcium by atomic absorption spectrometry or by plasma emission spectrometry.

It is applicable to all kinds of paper, board and pulp.

It specifies a method to determine the acid-soluble part of the incineration residue, i.e. that part of the ignition residue obtained after incineration that is soluble in hydrochloric acid. If the residue is completely soluble, the result obtained by the procedure specified in this International Standard is taken as the total amount of calcium in the sample.

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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 287, *Paper and board — Determination of moisture content — Oven-drying method*

ISO 638, *Pulps — Determination of dry matter content*

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

ISO 7213, *Pulps — Sampling for testing*

3 Terms and definitions

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

3.1

mass fraction of calcium

amount of the element calcium in the solution obtained after incineration of the specimen at 525 °C and treating the residue with 6 mol/l hydrochloric acid, as specified in this International Standard

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4 Principle

A test portion is incinerated at 525 °C and the residue is treated with 6 mol/l hydrochloric acid. The test solution is aspirated into an acetylene/dinitrogen monoxide or acetylene/air flame and the mass fraction of calcium is determined by one of the following procedures:

- measurement of the absorption of the 422,7 nm line emitted by a calcium hollow-cathode lamp, or
- measurement of the absorption of the 422,7 nm line emitted by plasma emission spectrometry.

5 Reagents and materials

Use only chemicals of recognized analytical grade and only distilled or deionized water.

5.1 Hydrochloric acid, about 6 mol/l.

Dilute 500 ml of hydrochloric acid (density 1,19 g/ml) in 500 ml of water.

5.2 Calcium stock solution, 500 mg/l of Ca.

Weigh $1,249 \text{ g} \pm 0,001 \text{ g}$ of calcium carbonate, dried to constant mass at a temperature not exceeding 200 °C, into a 1 000 ml volumetric flask. Add 50 ml of water. Add a minimum volume of hydrochloric acid (5.1) (approximately 10 ml), drop by drop, to effect the complete dissolution of the calcium carbonate. Dilute with water to the mark and mix.

1 ml of this stock solution contains 0,500 mg of Ca.

5.3 Calcium standard solution, 50 mg/l of Ca.

Transfer 100 ml of the stock solution (5.2) to a 1 000 ml volumetric flask, dilute with water to the mark and mix.

1 ml of this standard solution contains 0,05 mg of Ca.

Commercially available, certified standard solutions may be used.

5.4 Lanthanum solution, 50 g/l of La.

Moisten 59 g of lanthanum oxide (La_2O_3) with water. Slowly and cautiously, add 250 ml of hydrochloric acid (density 1,19 g/ml) to dissolve the lanthanum oxide. Dilute to the mark in a 1 000 ml volumetric flask and mix.

This solution is used to eliminate interference from phosphate when determining calcium in an air/acetylene flame. It is normally not required when the dinitrogen oxide/acetylene flame is used.

5.5 Caesium solution, 50 g/l of Cs.

In a 1 000 ml volumetric flask, dissolve 63,5 g of ultra-pure caesium chloride (CsCl) in water and fill up to the mark with water.

This solution is used to suppress ionization of calcium in the dinitrogen monoxide/acetylene flame. It is normally not required when an air-acetylene flame is used.

NOTE Caesium chloride may be replaced by the corresponding amount of potassium chloride, unless potassium is to be determined in the same solution.

5.6 Acetylene gas and/or dinitrogen monoxide gas, of a grade suitable for atomic absorption spectrometry.

WARNING — Acetylene gas forms explosive mixtures with air.

5.7 Appropriate gas for the plasma spectrometer (6.4). Argon is usually recommended as a carrier gas.

6 Apparatus and equipment

Ordinary laboratory equipment. Clean all equipment in 0,1 mol/l hydrochloric acid.

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

6.2 Dishes, of platinum or quartz.

6.3 Atomic absorption spectrometer, with a burner for nitrogen monoxide/acetylene or air/acetylene and with a hollow-cathode lamp for calcium.

NOTE A multi-element lamp may be used.

6.4 Inductively coupled plasma spectrometer.

7 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.

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 mentioned in Clause 8.

8 Procedure

8.1 Incineration and dissolution of the residue

Carry out the procedure in duplicate.

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

Determine the moisture content on a separate air-dried portion as specified in ISO 287 or ISO 638, as relevant. Weigh this portion at the same time as the test portion used for incineration.

Carry out ashing of the test portion, as described in ISO 1762. A portion of 2 g to 5 g is usually sufficient.

Carry out the dissolution of the ash under a fume hood. To avoid splattering, carefully moisten the ash with water and add 5 ml of hydrochloric acid (5.1) to the dish. Evaporate to dryness on a boiling-water bath or equivalent device. Repeat this procedure twice.

For samples with a high carbonate content, more than 10 ml of acid (2 × 5 ml) may be needed.

Add 2,5 ml of the hydrochloric acid (5.1) to the dry residue. If necessary, heat the dish covered by a watch glass for a few minutes.

Using the filter paper (6.1), filter the contents of the dish into a 25 ml volumetric flask. To ensure that the transfer is complete, add another portion of 2,5 ml of acid to the dish and heat again. Filter this last portion of