

SLOVENSKI STANDARD

SIST ISO 10775:2014

01-februar-2014

Nadomešča:
SIST ISO 10775:1996

Papir, karton, lepenka in vlaknine - Določanje vsebnosti kadmija - Metoda atomske absorpcijske spektrometrije

Paper, board and pulps -- Determination of cadmium content -- Atomic absorption spectrometric method

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Papier, carton et pâtes -- Détermination de la teneur en cadmium -- Méthode par spectrométrie d'absorption atomique [SIST ISO 10775:2014](https://standards.iteh.ai/catalog/standards/sist/42dcff26-b0e2-4b23-9105-29a7c0f50200/sist-iso-10775-2014)

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Ta slovenski standard je istoveten z: ISO 10775:2013

ICS:

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

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en

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INTERNATIONAL STANDARD

**ISO
10775**

Second edition
2013-08-01

Paper, board and pulps — Determination of cadmium content — Atomic absorption spectrometric method

*Papier, carton et pâtes — Détermination de la teneur en cadmium —
Méthode par spectrométrie d'absorption atomique*

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ISO 10775:2013(E)

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. www.iso.org/directives

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

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 10775:1995), of which it constitutes a minor revision.

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Paper, board and pulps — Determination of cadmium content — Atomic absorption spectrometric method

1 Scope

This International Standard specifies a method for the determination of traces of cadmium in all types of paper, board and pulp, including products containing recycled fibre, that can be wet-combusted in nitric acid as specified in this International Standard.

The lower limit of the determination depends on the equipment used and is normally about 10 µg/kg. Cadmium present in pigments and fillers that do not dissolve in nitric acid under the conditions of this test may not be determined quantitatively.

NOTE It has been claimed that the dissolution of cadmium from pigments other than calcium carbonate is incomplete by a few percent.

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.

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

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

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

ISO 7213, *Pulps — Sampling for testing*

3 Principle

The sample is treated with nitric acid in a closed vessel. The resulting solution is diluted and cadmium content determined by atomic absorption spectrometry using the graphite furnace technique.

Wet combustion may be done either in an autoclave or microwave oven.

4 Reagents

All reagents shall be of the highest possible purity. The quality normally designated “pro analysi” or “analytical reagent (AR)” is often not sufficiently pure. Use only freshly distilled and deionized water or water of equivalent purity.

NOTE Commercially available solutions may also be used.

4.1 Concentrated nitric acid, $c(\text{HNO}_3) = 15 \text{ mol/l}$

Use a quality specially made for use in the determination of trace metals.

4.2 Dilute nitric acid, $c(\text{HNO}_3) = 0,15 \text{ mol/l}$

Dilute with water 10 ml of concentrated nitric acid (4.1) to one litre.

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4.3 Cadmium nitrate standard solution, $p(\text{Cd}) = (1,000 \pm 0,002) \text{ g/l}$, made, for example, by dissolving 2,774 g of cadmium nitrate tetrahydrate, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, per litre of nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$.

4.4 Cadmium stock calibration solution, $\rho(\text{Cd}) = 1,00 \text{ mg/lp}$

Using a precision pipette dilute 1,00 ml of the cadmium standard solution (4.3) to 1 000 ml with dilute nitric acid (4.2) in a volumetric flask. Mix by shaking the flask.

The solution has a shelf life of several months if stored in a polyethylene bottle.

4.5 Matrix modifier solution

Several matrix modifier solutions are recommended in the literature. The three solutions given in 4.5.1 to 4.5.3 are in common use. The choice among them depends on their performance in each particular laboratory, which is evaluated by running a blank.

4.5.1 Palladium nitrate solution

Dissolve 2,0 g of $\text{Pd}(\text{NO}_3)_2$ in 10 ml of nitric acid (4.1) and dilute with water to 500 ml in a volumetric flask. (Alternatively, the equivalent amount of palladium metal, i.e. 0,924 g, is dissolved in nitric acid.) Prepare a working solution by diluting 5 ml of this stock solution with water to 100 ml.

or

4.5.2 Ammonium dihydrogenphosphate solution

Dissolve 2 g of $\text{NH}_4\text{H}_2\text{PO}_4$ in water and dilute to 100 ml.

or

4.5.3 Magnesium nitrate solution

Dissolve 0,5 g of $\text{Mg}(\text{NO}_3)_2$ in water and dilute to 100 ml.

5 Apparatus

Ordinary laboratory equipment and the following.

5.1 Apparatus for wet combustion, either:

5.1.1 Autoclave, with an inner vessel of polytetrafluoroethylene (PTFE), capacity 250 ml, provided with a lid of PTFE, and a heating block that can maintain the autoclave at a temperature of $160 \pm 5 \text{ }^\circ\text{C}$.

The block shall be provided with an extra safety switch that prevents overheating.

NOTE 1 The PTFE vessels can be protected from corrosion from the outside by applying a film of silicone grease on all outside surfaces. The film is removed and renewed after each heating period. Vessels treated in this manner should not be used when silica has to be determined.

NOTE 2 The use of an oven instead of the heating block is not recommended because of the hazard involved in removing the hot autoclaves from the oven at the end of the heating period.

or

5.1.2 Laboratory microwave oven, with programming facilities, specially designed for wet combustion, with digestion vessels of PTFE, capacity at least 120 ml, having safety valves to release pressures over 830 kPa.

5.2 Atomic absorption spectrometer, equipped for the graphite furnace technique and with a so-called “L’vov” platform, and with a lamp for the determination of cadmium.

Preferably, the instrument should have background correction.

6 Sampling and sample preparation

Ensure that the sample is representative of the lot to be tested. If applicable, follow the instructions given in ISO 186 or in ISO 7213.

In order to avoid contamination, keep the samples wrapped in aluminium foil until required.

Tear from the sample enough pieces, about 10 mm x 10 mm in size, to provide the amount required, taking an approximately equal amount from each sample. Do not use a knife or any other metal tool. (Tools of plastics or ceramics may be used.) Split samples of pulp sheets or board to reduce their thickness.

Keep the sample near the balance for moisture equilibration for at least 20 min. Withdraw a specimen for the determination of dry matter content in accordance with ISO 287 or ISO 638, as applicable.

7 Wet combustion

Carry out wet combustion in duplicate; also run a blank (see [Clause 8](#)).

7.1 Test sample preparation

Weigh a test portion of 1 g to the nearest 1.0 mg and transfer it to the appropriate PTFE vessel ([5.1](#)). Add 10 ml of the concentrated nitric acid ([4.1](#)). Close the vessel with its lid and place it in the autoclave ([5.1.1](#)) or in the microwave oven ([5.1.2](#)), as applicable.

7.2 Combustion procedure

7.2.1 Autoclave procedure

If an autoclave is used, close the autoclave as instructed by the manufacturer and place it in the heating block ([5.1.1](#)). Heat it at 160 ± 5 °C for 16 ± 1 h. Allow the heating block and the autoclave to cool and, with caution, open the autoclave in a hood. Proceed to [7.3](#).

7.2.2 Microwave oven procedure

If a microwave oven is used, the test portion size shall be adjusted to the capacity of the digestion vessel. If this is 120 ml, the maximum test portion mass shall be 0,3 g. If more test portion is taken, the safety valve of the digestion vessel will activate.

Set the power regulator to the predetermined value and heat the closed digestion vessel ([5.1.2](#)), with its contents for 10 – 20 min typically, 30 min maximum, depending on the microwave power. Allow the vessel to cool and, with caution, open it in a hood.

The correct setting of the power regulator shall be determined separately for each oven. A typical setting is 40 % of the maximum power. Also see [5.1.2](#).

7.3 Dilution

Allow nitrous fumes to escape from the wet combustion vessel and dilute the remaining solution to a known volume with water. The dilution volume depends on the equipment used, and should be kept as small as possible.

The dilution can be made in a disposable graduated plastic vessel. Volumetric flasks thoroughly cleaned, may also be used.