
**Paper, board and pulps — Determination
of total magnesium, total calcium, total
manganese, total iron and total copper**

*Papier, carton et pâtes — Détermination de la teneur en magnésium
total, en calcium total, en manganèse total, en fer total et en cuivre total*

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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 17812 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

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Introduction

This International Standard combines the determination of magnesium (Mg), calcium (Ca), manganese (Mn), iron (Fe) and copper (Cu) into one single standard. The scope of this International Standard includes the acid-soluble part of the elements plus that part of the elements that may remain insoluble after dissolution of the incineration residue with hydrochloric acid. This International Standard is complementary to the following International Standards, which measure only the acid-soluble part of the element.

ISO 777, *Paper, board and pulp — Determination of acid-soluble calcium*

ISO 778, *Paper, board and pulp — Determination of acid-soluble copper*

ISO 779, *Paper, board and pulp — Determination of acid-soluble iron*

ISO 1830, *Paper, board and pulps — Determination of acid-soluble manganese*

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Paper, board and pulps — Determination of total magnesium, total calcium, total manganese, total iron and total copper

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 should be taken to ensure the relevant precautions are observed.

1 Scope

This International Standard 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. The total content comprises the acid-soluble part of the element plus the acid-insoluble part of the element.

It is applicable to all types of paper, board and pulps.

2 Normative references

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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 of a lot — Oven drying method*

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 7213, *Pulps — Sampling for testing*

3 Principle

A test specimen is incinerated at 525 °C and the residue is dissolved in 6 mol/l hydrochloric acid. If the resulting solution contains insoluble material, originating, for example, from fillers and coating formulations, these shall be destroyed. Two alternative methods for destroying the insoluble material are included in this International Standard: hydrofluoric acid digestion (7.3.2) and fusion with alkali metal salts (7.3.3). This insoluble part should be combined with the original solution. This is to ensure complete recovery of elements that may be occluded in the residue or combined with silicates. 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 with a maximum conductivity of 1,0 $\mu\text{S}/\text{cm}$ at 25 °C.

4.2 Hydrochloric acid (HCl), about 6 mol/l.

Dilute 500 ml of concentrated hydrochloric acid (specific gravity 1,19 g/ml) to 1 000 ml with water.

4.3 Hydrofluoric acid (HF), 48 % to 52 % solution.

4.4 Sulfuric acid (H₂SO₄), concentrated (specific gravity 1,84 g/ml).

4.5 Lanthanum chloride (LaCl₃), solution, $c(\text{La}) = 50 \text{ g/l}$. In a volumetric flask, dissolve 59 g of lanthanum oxide (La₂O₃) 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 magnesium in an air/acetylene flame. The solution is not required when the nitrous oxide/acetylene flame or inductively coupled plasma (ICP) technique is used.

4.6 Nitric acid (HNO₃), concentrated (specific gravity 1,4 g/ml).

4.7 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:

- **Magnesium**, 1 000 mg/l standard solution. Dissolve 1,000 g of magnesium metal ribbon in 100 ml of 1:4 nitric acid (4.6) and dilute to 1 000 ml with water.
- **Calcium**, 1 000 mg/l standard solution. Dissolve 2,497 g of primary standard calcium carbonate (CaCO₃) in a minimum volume of 1:4 nitric acid (4.6) and dilute to 1 000 ml with water.
- **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 (4.6) and dilute to 1 000 ml with water.
- **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.
- **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 (4.6) and dilute to 1 000 ml with water.

4.8 Acetylene gas and/or nitrous 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.

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

4.10 Alkali metal salts, mix sodium carbonate (Na₂CO₃) and boric acid (H₃BO₃) in the required amounts in the weight portions 3:1.

NOTE Other alkali metal salts can also be used [for example lithium metaborate (LiBO₂)]

4.11 Cesium chloride (CsCl), solution, $\rho(\text{Cs}) = 50 \text{ g/l}$. In a 1 000 ml volumetric flask, dissolve 63,5 g of ultra-pure cesium chloride (CsCl) in water and fill up to the mark with water.

NOTE This cesium solution is used to suppress ionization of calcium in a nitrous oxide/acetylene flame. The solution is not required when an air/acetylene flame or ICP technique is used.

5 Apparatus and equipment

5.1 General

Ordinary laboratory equipment is used. All glassware and plasticware 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.

NOTE If treatment with hydrofluoric acid is required, only platinum crucibles can be used, since fused silica is attacked by HF.

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, equipped with air/acetylene and/or nitrous oxide/acetylene burners and with hollow cathode lamps for Mg, Ca, Mn, Fe and Cu.

NOTE Multi-element lamps can also be used.

5.7 Inductively coupled plasma (ICP) spectrometer.

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. Protective disposable 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 as specified in Clause 7.

Since iron tends to be nonhomogeneous 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 a 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.