INTERNATIONAL STANDARD

ISO 9668

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Pulps — Determination of magnesium content — Flame atomic absorption spectrometric method

Pâtes — Détermination de la teneur en magnésium — Méthode par spectrométrie d'absorption atomique dans une flamme

Reference number
ISO 9668:1990(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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9668 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

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Pulps — Determination of magnesium content — Flame atomic absorption spectrometric method

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 shall be taken to ensure that the relevant safety precautions are observed.

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the magnesium content of pulp. The method is applicable to all kinds of pulp, except pulps containing talc.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 638:1978, *Pulps — Determination of dry matter content.*

ISO 1762:1974, *Pulps — Determination of ash.*

3 Principle

Ashing of the pulp and dissolution of the ash in hydrochloric acid. Aspiration of the ash solution into an acetylene/nitrous oxide or acetylene/air flame, after addition of lanthanum ions to suppress certain interferences. Measurement of the absorption of the 285,2 nm line emitted by a magnesium hollow-cathode lamp.

4 Reagents

During the analysis, unless otherwise indicated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

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

4.2 Lanthanum chloride ($LaCl_3$), solution containing 20 g of La^{3+} per litre.

To a 1 000 ml one-mark volumetric flask add 24 g of lanthanum oxide (La_2O_3) of atomic absorption spectrometric grade. Slowly and cautiously add 50 ml of concentrated hydrochloric acid, (ρ = 1,18 g/ml) while agitating to dissolve the lanthanum oxide. Dilute to the mark with water.

4.3 Magnesium, standard solution containing 500 mg of Mg^{2+} per litre.

Dissolve 500 mg of magnesium metal ribbon in 50 ml of hydrochloric acid solution (4.1), dilute to the mark with water in a 1 000 ml one-mark volumetric flask and mix.

1 ml of this standard solution contains 0,500 mg of Mg^{2+}.

NOTE 1 Commercially available spectrographic grade standard solutions of equivalent quality and dilution may be used.

4.4 Magnesium, standard solution containing 10 mg of Mg^{2+} per litre.

Transfer 20 ml of the magnesium standard solution (4.3) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,010 mg of Mg^{2+}.

NOTE 2 This solution is not stable and should be freshly prepared each day.

5 Apparatus

Ordinary laboratory apparatus and

Atomic absorption spectrometer, fitted with either an acetylene/nitrous oxide (N_2O) burner or an acetylene/air burner and a magnesium hollow-cathode lamp.

6 Preparation of the sample

Tear not less than 40 g of the air-dried sample into pieces of suitable size. Use protective cotton gloves. Do not use cut or punched edges, or other parts where metallic contamination may have occurred.

NOTE 3 Low magnesium-containing pulps (e.g. dissolving pulps) may require larger test portions.

7 Procedure

7.1 Test portions

Weigh two test portions of pulp of about 10 g, to the nearest 0,01 g, and carry out the determination in duplicate. Weigh two other test portions of about 10 g, to the nearest 0,001 g, for determination of the dry matter content as described in ISO 638.

7.2 Ashing of the test portion

Carefully clean a dish of fused silica or platinum. Remove any impurities in the platinum dish by rubbing with fine sand. Ash the test portion as specified in ISO 1762.

7.3 Dissolving the ash

To the ash obtained in 7.2, add 5 ml of the hydrochloric acid (4.1) and evaporate to dryness on a steam bath. Repeat this once, and treat the residue with another portion of 5 ml of the hydrochloric acid solution and heat for 5 min on the steam bath. Wash the contents of the dish into a 100 ml one-mark volumetric flask with distilled water. To ensure complete transfer and solution, add a further 5 ml of the hydrochloric acid solution to the residue in the dish, and heat on the steam bath. Wash this last portion into the main quantity in the volumetric flask, dilute to the mark and mix.

7.4 Preparation of the test solution

Select a volume, V, of the solution prepared in 7.3, so that, when diluted to 50 ml, the magnesium content will fall within the range (0,01 mg to 0,10 mg/100 ml) given in table 1. If the magnesium content is not known, run a preliminary test.

With a pipette, transfer this volume of the ash solution into a 50 ml one-mark volumetric flask.

Add 10 ml of the lanthanum chloride solution (4.2), 5 ml of the hydrochloric acid solution (4.1) and dilute to the mark with water. If the solution now contains suspended matter, allow this to settle. Use the clear part of the solution to carry out the spectrometric measurement.

7.5 Calibration

7.5.1 Preparation of the set of calibrating solutions

Into each of a series of five 100 ml one-mark volumetric flasks, place 20 ml of the lanthanum chloride solution (4.2), 10 ml of the hydrochloric acid solution (4.1) and a volume of the standard magnesium solution (4.4) as shown in table 1. Dilute the contents of each volumetric flask to the mark with water and mix.

7.5.2 Spectrometric measurements

Operate the atomic absorption spectrometer (clause 5) for the determination of magnesium according to the manufacturer's instructions.

WARNING — To avoid explosion, it is necessary to light the burner with the acetylene/air mixture, before switching to the acetylene/nitrous oxide mixture.

Carry out a blank test, employing the prescribed spectrometric procedure, using the quantities of reagents given in 7.5.1, but omitting the standard magnesium solution (4.4) (table 1, term zero). Adjust the instrument to zero absorbance with the blank test solution.

Table 1 — Set of calibrating solutions

Standard magnesium solution (4.4) ml	Mass of Mg^{2+} mg
0[1)]	0
1,0	0,01
2,0	0,02
5,0	0,05
10,0	0,10

1) Blank test on reagents for calibration graph.

Aspirate the set of calibrating solutions (7.5.1) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph. Spray water through the burner after each measurement.

7.5.3 Plotting calibration graph

Plot a graph having, for example, the mass, in milligrams of Mg^{2+} contained in 100 ml of each of the standard magnesium solutions as abscissae, and the corresponding values of the measured absorbance as ordinates.

NOTE 4 Some instruments have a system for automatic evaluation of data. For such instruments the instruction given above may be disregarded.

7.6 Determination

7.6.1 Blank test

Carry out a blank test according to the instructions given in 7.5.2, following the same procedure and using the same quantities of all reagents used in the test solution but omitting the test specimen obtained in 7.3. Adjust the instrument to zero absorbance with the blank test solution.

7.6.2 Spectrometric measurement

Carry out the spectrometric measurement on the test solution (7.4) according to the instructions of 7.5.2.

8 Expression of results

8.1 By means of the calibration graph (7.5.3), determine the mass, in milligrams, of Mg^{2+} corresponding to the value of the absorbance measured on the test solution in 7.6.2.

Calculate the magnesium content, expressed in milligrams of Mg per kilogram, from the formula

$$5 \times 10^4 \times \frac{m_1}{V \cdot m_2}$$

where

m_1 is the mass, in milligrams, of Mg^{2+} in 100 ml of the test solution (7.4) obtained from the calibration graph (7.5.3);

m_2 is the mass, in grams, of the test portion (7.1), calculated on an oven-dry basis in accordance with ISO 638;

V is the volume, in millilitres, of the ash solution (7.3) used to prepare the test solution (7.4).

8.2 Take as the result the mean of the values obtained from the two simultaneous determinations, and round as specified in table 2.

Table 2 — Rule for the rounding of the mean

Mean of values obtained mg/kg	In the test report round to the nearest mg/kg
< 100	1
> 100	5

9 Test report

The test report shall include the following particulars:

a) all the information necessary for complete identification of the sample;

b) reference to this International Standard;

c) any variation from standard procedure, if applied;

d) the magnesium content in milligrams per kilogram of o.d. pulp;

e) any unusual features observed in the course of the test;

f) any operation not specified in this International Standard or in the International Standards to which reference is made, which might have affected the results.

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