

INTERNATIONAL STANDARD

ISO 1830

Second edition
2005-04-15

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

*Papiers, cartons et pâtes — Détermination du manganèse soluble dans
l'acide*



Reference number
ISO 1830:2005(E)

© ISO 2005

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Reagents and materials	2
6 Apparatus and equipment	2
7 Sampling and preparation of sample	3
8 Procedure	3
8.1 Ignition and dissolution of the residue	3
8.2 Blank	3
9 Preparation of calibration solutions	4
10 Determination	4
11 Calculation	4
12 Precision	4
12.1 Repeatability	5
12.2 Reproducibility	5
13 Test report	5
Bibliography	6

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 1830 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 1830:1982), which included the photometric procedure as well as the procedure based on atomic absorption spectroscopy. The photometric procedure has been deleted, as it is now seldom used. The scope has been enlarged to include paper and board, in addition to pulp.

ISO 1830 has been harmonized with ISO 777 ^[1], ISO 778 ^[2] and ISO 779 ^[3], in order to make it possible to perform the final measurement of all four elements on the same solution.

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

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 that the relevant precautions are observed.

1 Scope

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

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

This International Standard specifies a method to determine manganese in the acid-soluble part of the ignition residue, i.e. that part of the ignition residue remaining after ignition 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 manganese in the sample.

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 3696, *Water for analytical laboratory use — Specification and test methods*

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 manganese

amount of the element manganese, expressed as milligrams per kilogram of oven-dry sample, in the solution obtained after ignition of the specimen at 525 °C and treating the residue with 6 mol/l hydrochloric acid

4 Principle

A test portion is ignited 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 manganese is determined by one of the following procedures:

- measurement of the absorption of the 279,5 nm line emitted by a manganese hollow-cathode lamp; or
- measurement of the absorption of the 257,6 nm line emitted by plasma emission spectrometry.

5 Reagents and materials

Use only chemicals of recognized analytical grade.

5.1 Water: distilled or deionised water shall be used throughout the test. The conductivity of the water shall not exceed 0,1 mS/m after boiling for 1 h and cooling to between 20 °C and 25 °C. Instructions for the determination of conductivity are given in ISO 3696.

5.2 Hydrochloric acid, about 6 mol/l.

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

5.3 Manganese stock solution, 100 mg/l of Mn.

Weigh 0,308 g ± 0,001 g of manganese sulfate monohydrate (MnSO₄·H₂O), dried to constant mass at a temperature of 400 °C to 450 °C prior to use, into a 1 000 ml volumetric flask. Add 0,5 ml of hydrochloric acid (5.2), dilute with water to the mark and mix.

1 ml of this stock solution contains 0,10 mg of Mn.

5.4 Manganese standard solution, 10 mg/l of Mn.

Transfer 100 ml of the manganese stock solution (5.3) to a 1 000 ml volumetric flask. Add 200 ml of hydrochloric acid (5.2), dilute with water (5.1) to the mark and mix.

1 ml of this standard solution contains 0,01 mg of Mn.

Commercially available certified standard manganese solutions may be used.

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

WARNING — Acetylene gas forms explosive mixtures with air.

5.6 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 dinitrogen monoxide/acetylene or air/acetylene and with a hollow-cathode lamp for manganese.

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 specified in Clause 8. During handling, special precautions shall be taken to avoid any change in moisture content.

8 Procedure

8.1 Ignition 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 ignition.

Carry out ignition of the test portion as described in ISO 1762. A portion of 2 g to 5 g will be suitable for many materials. However, the exact mass of the portion used shall be adjusted as necessary, based on the manganese level in the sample and the analysis procedure used, that is, either flame atomic absorption spectrometry or plasma emission spectroscopy.

Carry out the dissolution of the ignition residue under a fume hood. To avoid splattering, carefully moisten the ignition residue with water (5.1) and add 5 ml of hydrochloric acid (5.2) to the dish. Evaporate to dryness on a boiling-water bath, a hotplate, a heating lamp or an 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 (5.2) to the 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 if necessary. Filter this last portion of acid into the main portion in the volumetric flask with the aid of some water (5.1). Fill up to the mark and mix. This is the test solution.

8.2 Blank

Run a blank with the same quantity of each of the chemicals as those added to the ignition residue, but without any residue.

9 Preparation of calibration solutions

It is important that the acid concentration is the same in the calibration solutions as in the test solution, since the acid concentration influences the signal.

From the manganese standard solution (5.4) prepare at least three calibration solutions, and in addition one zero solution, for the construction of the calibration graph. (The zero solution is similar to the calibration solutions, but contains no added manganese. Do not confuse it with the blank.)

NOTE Two calibration solutions are sufficient for plasma emission spectrometry.

10 Determination

Carry out the spectrometric measurement of the calibration solutions, including the zero solution, the test solution and the blank solution. Operate the instrument as instructed by the manufacturer.

The solutions may contain lanthanum or caesium, if the same solutions are used for the determination of calcium. These salts do not interfere with the determination of manganese, but the same amounts of salts shall be added to the calibration solutions.

If any value obtained exceeds the range covered by the calibration solutions, the measurement shall be repeated with a more dilute test solution. Add acid, caesium solution or lanthanum solution as required.

Most instruments have a system for automatic calculation of results. If the instrument has no automatic calculation system, construct the calibration graph by plotting the absorbance, corrected for the blank, against the concentration of manganese, in milligrams per litre. Then read the manganese concentration in the test solution from the calibration graph.

11 Calculation

Calculate the mass fraction of manganese from the expression

$$w_{\text{Mn}} = \frac{f \times \rho_{\text{Mn}} \times V}{m}$$

where

w_{Mn} is the mass fraction of manganese in the sample, in milligrams per kilogram;

ρ_{Mn} is the manganese concentration in the test solution, as obtained from the calibration plot corrected for the blank, in milligrams per litre;

V is the volume, in millilitres, of the original test solution (standard volume = 25 ml);

m is the mass of sample taken, oven-dry basis, in grams;

f is the dilution factor; $f = 1$, unless the original test solution has been diluted.

Calculate the mean with two significant figures.

12 Precision

A study made by SCAN-test in 1996 gave the following results.

12.1 Repeatability

A sample of uncoated paperboard was analysed in one laboratory nine times. The mean found was 33 mg/kg, and the coefficient of variation (CV) was 2 %.

NOTE As the above study comprises only one sample and one laboratory, the result is only a rough estimation of the repeatability.

12.2 Reproducibility

Four samples were analysed in eight laboratories. The results in Table 1 were obtained for the mean and the coefficient of variation (CV):

Table 1

Sample type	Mean mg/kg	CV %
Bleached pulp	0,2	19
Coated paperboard	7,4	18
Uncoated paperboard	32	10
Copy paper	19	15

13 Test report

The test report shall include the following information:

- a) reference to this International Standard;
- b) the date and place of testing;
- c) all information for complete identification of the sample;
- d) the result, expressed as indicated in Clause 11;
- e) any departure from the procedure described in this International Standard or any other circumstances which may have affected the result.

Bibliography

- [1] ISO 777, *Paper, board and pulp — Determination of acid-soluble calcium*
- [2] ISO 778, *Paper, board and pulp — Determination of acid-soluble copper*
- [3] ISO 779, *Paper, board and pulp — Determination of acid-soluble iron*

.....

ICS 85.040; 85.060

Price based on 6 pages