INTERNATIONAL STANDARD

ISO 779

Second edition
2001-06-01

Paper, board and pulp — Determination of iron

Papier, carton et pâte — Détermination de la teneur en fer

Reference number
ISO 779:2001(E)

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 779 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 779:1982), of which it constitutes a technical revision.

The previous edition of this International Standard 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.

Introduction

This International Standard corresponds to ISO 777[1] and ISO 778[2] in order to make it possible to perform the final measurement of all three elements on the same solution.

Paper, board and pulp — Determination of iron

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 iron 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 which 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 iron in the sample.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
mass fraction of iron
amount of the element iron 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.

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 iron is determined by one of the following procedures:

— measurement of the absorption of the 248,3 nm line emitted by an iron hollow-cathode lamp; or

— measurement of the absorption of the 248,3 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 Iron stock solution, 100 mg/l of Fe.

Dissolve 100 mg of pure iron wire in the smallest quantity possible of hydrochloric acid (density 1,19 g/ml) in a 1 000 ml volumetric flask. Dilute with water to the mark and mix.

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

5.3 Iron standard solution, 10 mg/l of Fe.

Transfer 100 ml of the iron stock solution (5.2) to a 1 000 ml volumetric flask and add 200 ml of hydrochloric acid (5.1). Dilute with water to the mark and mix.

1 ml of this standard solution contains 0,01 mg of Fe. The solution is not stable.

Commercially available, certified standard iron solutions may be used.

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

WARNING — Acetylene gas forms explosive mixtures with air.

5.5 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 iron.

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 as prescribed 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.

For samples with a high carbonate content, more than 10 ml of acid (2 × 5 ml) may be needed, for example 20 ml (2 × 10 ml).

Add 2,5 ml of the hydrochloric acid (5.1) in order to dissolve 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. Filter this last portion of acid into the main portion in the volumetric flask with the aid of some water. 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 incineration residue but without any residue.

9 Preparation of calibration solutions

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

From the iron standard solution (5.3), 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 iron. Do not confuse it with the blank.)

NOTE Not more than two calibration solutions are needed 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 iron, 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 evaluation of results. If the instrument has no automatic evaluation system, construct the calibration graph by plotting the absorbance, corrected for the blank, against the concentration of iron in milligrams per litre.Then read the iron concentration in the test solution from the calibration graph.

11 Calculation

Calculate the mass fraction of iron from the expression

$$\omega_{\mathrm{Fe}} = \frac{f \cdot \rho_{\mathrm{Fe}} \cdot V}{m}$$

where

ω_{Fe} is the mass fraction of iron in the sample, in milligrams per kilogram;

ρ_{Fe} is the iron 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

12.1 General

A study[1] gave the following results.

12.2 Repeatability

A sample of uncoated paperboard was analysed in one laboratory nine times. The mean found was 13 mg/kg, and the coefficient of variation (CV) was 8 %.

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

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1) Study made by SCAN-test in 1996.

12.3 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	3,6	45
Coated paperboard	430	19
Uncoated paperboard	13	28
Copy paper	180	4
NOTE The high CV-values are possibly due to an uneven distribution of iron in the sample.		

13 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) date and place of testing;

c) complete identification of the sample tested;

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.