INTERNATIONAL STANDARD

ISO 9198

Second edition
2001-06-01

Paper, board and pulp — Determination of water-soluble sulfates

Papier, carton et pâte — Détermination des sulfates solubles dans l'eau

Reference number
ISO 9198:2001(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 9198 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 9198:1989), of which it constitutes a technical revision.

There are two major differences between this and the previous edition of ISO 9198.

a) The extraction is now done at 23 °C, whereas boiling water was used in the previous edition.

b) Ion chromatography, a technique now widely used for the determination of anions because of its high sensitivity and selectivity, replaces the conductometric titration using barium chloride and lithium sulfate solutions in the former edition.

Paper, board and pulp — Determination of water-soluble sulfates

1 Scope

This International Standard specifies a method for the determination of water-soluble sulfates in all types of pulp, paper and board. The lower limit of the determination is 20 mg of sulfate ion per kilogram of dry 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 186:1994, *Paper and board — Sampling to determine average quality.*

ISO 287:1985, *Paper and board — Determination of moisture content — Oven-drying method.*

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

ISO 7213:1981, *Pulps — Sampling for testing.*

3 Term and definition

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

3.1
water-soluble sulfates
⟨in pulp, paper and board⟩ amount of sulfate ion that is extracted with water at 23 °C and determined under the conditions specified

4 Principle

Pieces of the sample are extracted with water at 23 °C in a disintegrator. An aliquot of the resulting suspension is used for determination of the sulfate ion content by ion chromatography.

5 Reagents

Use only reagents of recognized analytical quality and only water as specified in 5.1.

5.1 **Distilled water** or **deionized water,** of conductivity less than 0,1 mS/m at 25 °C.

5.2 Sulfate stock solution, $\rho(SO_4^{-2})$ = 1 000 mg/l.

Dry a portion of potassium sulfate (K_2SO_4) at 140 °C. Transfer 181,5 mg ± 2 mg thereof to a 100 ml volumetric flask, dissolve the salt and make up to the mark with water (5.1).

Commercially available standard solutions can be used.

5.3 Sulfate matching solution

Dilute the sulfate stock solution (5.2) with water (5.1) to a sulfate ion concentration of, for example, $\rho(SO_4^{-2})$ = 10 mg/l. Do not use sulfate matching solutions that are more than 1 week old.

5.4 Other solutions, as specified in the instructions for the ion chromatograph.

6 Apparatus

6.1 Wet disintegrator, a high-speed mixer, capable of disintegrating the sample completely.

6.2 Ion chromatograph, having a pump, an injector loop of known volume, a column system suitable for the determination of sulfates and a conductivity detector.

6.3 Syringe, of a quality suitable for ion chromatography, of capacity 5 ml and having a filter of about 0,2 µm pore width.

6.4 Tea strainer or **similar device with a fine mesh screen,** of stainless steel, to prevent fibres from clogging the syringe.

7 Sampling and preparation of sample

The procedure to be followed when sampling depends on the particular circumstances in each case. If the analysis is being made to evaluate a lot or a consignment of pulp, paper or board, the sample shall be taken in accordance with ISO 7213 or ISO 186, as relevant. If the analysis is made on another type of sample, report the origin of the sample and, if possible, the sampling procedure.

Since the amount of sulfates in the sample can be very low, take care not to contaminate it during sampling. Wear clean gloves at all times when handling the sample.

Keep the sample protected, wrapped in aluminium foil or in plastic bags, until required for analysis.

Select the sample portion for the analysis so that it is representative of the sample received.

8 Procedure

Carry out the procedure in duplicate. A blank shall also be carried through the entire procedure.

Weigh, to the nearest 0,01 g, a test portion, generally of between 2 g and 5 g. Split thick board and pulp sheets into thinner pieces to facilitate soaking.

The size of the test portion should be selected so that the sulfate ion content of the extract is within the optimum range of the ion chromatograph.

At the same time, determine the dry matter content on a separate portion using the procedure specified in ISO 287 or ISO 638, as relevant.

Transfer the weighed test portion to the disintegrator (6.1) and add 250 ml ± 2 ml of water (5.1) at 23 °C ± 2 °C. Disintegrate the sample portion until it is completely disintegrated, but no longer. After disintegration, let the sample portion soak for about 2 h.

Withdraw an aliquot for analysis of the suspension, using the syringe (6.3). If this operation is hampered by the presence of fibres or fibre bundles, use the tea strainer or similar device (6.4) to prevent fibrous material from clogging the syringe. Place the device over the fibres and withdraw the aliquot from the solution above this device. It is essential that the withdrawn aliquot be free from suspended material.

Since the operation of the ion chromatograph (6.2) depends on its design, no detailed instructions can be given in this International Standard. Operate the apparatus as instructed by the manufacturer.

For calibration, prepare from the sulfate matching solution (5.3) a series of five calibration solutions, covering about one decade of concentrations, for example, from 1 mg/l to 10 mg/l.

Run the calibration solutions and the sample solution (the aliquot) on the chromatograph as instructed by the manufacturer of the apparatus.

Plot the readings for the calibration solutions against their sulfate ion concentrations. The five points for the calibration solutions should fall on a straight line. If they fail to do so, repeat the calibration with another set of calibration solutions, covering a lower concentration range. However, the range of calibration solutions shall always bracket the sulfate concentration obtained from the aliquot.

Check the calibration daily and whenever a new set of calibration solutions is taken into use.

Read the sulfate peak of the extract (the aliquot). From the calibration graph, read the sulfate ion concentration of the extract, ρ, and of the blank, ρ_0.

9 Calculation

Calculate the mass fraction of water-soluble sulfates in the sample from the expression

$$\omega = 100 \frac{(\rho - \rho_0)V}{m\omega_\mathrm{d}}$$

where

ω is the mass fraction of sulfate ions in the sample, in milligrams per kilogram;

ρ is the sulfate ion concentration of the filtered extract, in milligrams per litre;

ρ_0 is the sulfate ion concentration of the blank solution, in milligrams per litre;

V is the volume of water (5.1) used in the disintegration (the volume specified is 250 ml);

m is the mass of sample taken, in grams;

ω_d is the dry matter content of the sample, expressed as a percentage.

Calculate the mean and report the result to the nearest 10 mg/kg. Report values below 20 mg/kg as "less than 20 mg/kg".

10 Precision

Five pulp and paper samples were extracted several times in a laboratory. From every extract, two aliquots were analysed. The results are shown in Table 1.

Table 1 — Results obtained when five samples were analysed several times, mg/kg

	Bleached pulp, soft wood	**Unbleached wrapping paper**	**Writing paper**	**Bleached pulp, hard wood**	**Paper** (40 g/m^2)
1st extraction	36,5 37,0	65,5 68,2	155,0 155,3	335,5 336,0	840 833
2nd extraction	38,2 38,4	60,8 61,1	153,3 154,3	323,2 320,0	824 830
3rd extraction	36,9 37,8	69,6 67,1	— —	— —	— —
Mean value	37,5	65,4	154,5	328,7	832,0
Coefficient of variation (% of all values)	2,1	5,6	0,9	2,5	6,3

11 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 9;

e) any departure from the procedure described in this International Standard or any other circumstances which may have affected the result.