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Paper, board and pulps — Determination of water-soluble sulfates — Titrimetric method

*Papiers, cartons et pâtes — Détermination des sulfates solubles dans l'eau —
Méthode titrimétrique*



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Foreword

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Paper, board and pulps.

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International Organization for Standardization

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Paper, board and pulps — Determination of water-soluble sulfates — Titrimetric method

1 Scope

This international Standard specifies a titrimetric method for the determination of water-soluble sulfates in paper, board and pulps.

The lower limit of the method is 20 mg of sulfate ion per kilogram of material.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

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

3 Definition

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

water-soluble sulfates (in paper, board and pulps) : The amount of sulfate ion extracted and determined under the conditions specified in this International Standard.

4 Principle

Pieces (at least 4 g) of the sample are extracted with hot water (100 ml) for 1 h. The extract is filtered and its sulfate ions are precipitated by an excess of barium ions. The excess of barium ions is determined by conductometric titration with lithium sulfate.

5 Reagents

During the analysis, use only reagents of recognized analytical quality and water as specified in 5.1.

5.1 Distilled or deionized water, conductivity less than 1 mS/m.

5.2 Ethanol (C_2H_5OH), 99,5 % (V/V).

5.3 Barium chloride, solution, $c(BaCl_2 \cdot 2H_2O) \approx 5$ mmol/l.

Dissolve 1,25 g of barium chloride dihydrate in water (5.1) and dilute to 1 litre.

5.4 Hydrochloric acid, $c(HCl) \approx 1$ mmol/l.

5.5 Lithium sulfate, standard volumetric solution, $c(Li_2SO_4 \cdot H_2O) = 5$ mmol/l.

Dissolve exactly 0,640 g of dry lithium sulfate monohydrate in water (5.1) and dilute to 1 000 ml in a volumetric flask.

6 Apparatus

Ordinary laboratory equipment, and

6.1 Conductometer, with a measuring cell for conductometric titration capable of measuring conductivity changes of 1 mS/m.

6.2 Microburette, capacity 10 ml, graduated in 0,02 ml.

6.3 Water bath, capable of maintaining a temperature of $25\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$. Other temperatures close to room temperature may be chosen and kept constant throughout the titration.

It is essential for the precision of the result that the temperature of the solution is kept constant during the entire titration.

NOTE — If available, an automatic titrator, having a motor-driven burette and chart recorder, may be used.

7 Preparation of sample

Since the amount of sulfates in the sample may be very low, take care not to contaminate it during sampling. Clean protective gloves should be worn at all times when handling the sample and the test pieces prepared from it.

Cut or tear the sample into pieces approximately 5 mm × 5 mm in size, and mix them thoroughly. Keep them protected from dust and fumes.

Determine the dry matter content separately in accordance with ISO 287 (for paper and board) or ISO 638 (for pulps).

8 Procedure

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

Weigh, to the nearest 0,01 g, not less than 4 g of sample in a 250 ml flask having a standard tapered joint. Thick board shall be split into plies before extraction.

With a pipette, add 100 ml of water (5.1). Attach a reflux condenser, fitting the flask, and boil gently for 1 h on an electric heater. Avoid any loss of water in the form of steam during this step.

Allow the extract to cool and filter it under suction, using an ashless paper filter and a Büchner funnel. Collect the filtrate in a flask having a ground glass stopper.

With a pipette, transfer 50,0 ml of the extract to a titration vessel and place this in the water bath (see 6.3). Add 100 ml of ethanol (5.2), 10 ml of hydrochloric acid (5.4) and 2 ml of barium chloride solution (5.3).

It is important that exactly the same volume of barium chloride solution be added to the sample and blank solutions. It is less critical if the volume deviates somewhat from 2 ml.

Insert the electrodes of the conductometer (6.1) and stir the solution at a constant speed with a magnetic stirrer or equivalent device for 5 min. Allow the solution to attain the temperature of the water bath.

Read the conductivity. Add from the microburette (6.2) portions of the lithium sulfate solution (5.5). After each addition allow the conductivity reading to reach a constant value before recording it. Continue until the total volume of lithium sulfate solution added is 4 ml.

If an automatic titrator is used, the titrant shall be added at a rate of about 0,2 ml/min.

Plot the conductivity readings against the volume of lithium sulfate solution added. Draw straight lines through the points, so that they form a "V". Read off the equivalent consumption of lithium sulfate at the point of intersection of the two lines.

IMPORTANT NOTE — To ensure complete precipitation of barium sulfate, it is important that a sufficient excess of barium ions is present at the beginning of the titration. Therefore, if the equivalent consumption of lithium sulfate solution is less than 1 ml, repeat the determination with a smaller volume of extract, i.e. 20 ml, adding water (5.1) to bring the total volume to 50 ml.

9 Expression of results

Calculate the content of water-soluble sulfates, in milligrams per kilogram, from the formula

$$\frac{96,1 V_3}{V_2} \times \frac{c(V_0 - V_1)}{m}$$

where

c is the actual concentration, in millimoles of $\text{LiSO}_4 \cdot \text{H}_2\text{O}$ per litre, of the lithium sulfate solution (standard 5 mmol/l);

V_0 is the volume, in millilitres, of lithium sulfate solution consumed in the titration of the blank;

V_1 is the volume, in millilitres, of lithium sulfate solution consumed in the titration of the test solution;

V_2 is the volume, in millilitres, of extract taken for titration (standard 50 ml);

V_3 is the total volume, in millilitres, of water added to the test portion (standard 100 ml);

m is the mass, in grams, of the oven-dry sample.

When substituting the standard values for c , V_2 and V_3 , the formula simplifies to

$$\frac{961 (V_0 - V_1)}{m}$$

Take the mean result of the two determinations as the content of water-soluble sulfates. Express the result in milligrams per kilogram of oven-dry sample and round off the result to the nearest integer.

10 Test report

The test report shall include the following particulars:

- reference to this International Standard;
- date and place of testing;
- identification of the material tested;
- results;
- any departure from the specified procedure, or other circumstances that may have affected the results.

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