TC 6

INTERNATIONAL STANDARD

ISO 9197-1

First edition
1989-07-15

Paper, board and pulps — Determination of water-soluble chlorides —

Part 1 : General method

Papiers, cartons et pâtes — Détermination des chlorures solubles dans l'eau — Partie 1 : Méthode générale

Reference number
ISO 9197-1 : 1989 (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 9197-1 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps.*

ISO 9197 consists of the following parts, under the general title *Paper, board and pulps — Determination of water-soluble chlorides*:

— *Part 1: General method*

— *Part 2: Method for high-purity products*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Paper, board and pulps — Determination of water-soluble chlorides —

Part 1: General method

1 Scope

This part of ISO 9197 specifies a general method for the determination of water-soluble chlorides in paper, board and pulps.

For electrical grade papers and pulps, the procedure in method 2, section 14 of IEC 554-2 : 1977, *Specification for cellulosic papers for electrical purposes — Part 2: Methods of test*, is recommended. For high purity products other than electrical grades, a method will be specified in ISO 9197-2.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9197. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9197 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 part of ISO 9197, the following definition applies.

water-soluble chlorides (in paper, board and pulps): The amount of chloride ion extracted and determined under the conditions specified in this part of ISO 9197.

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 chloride ion content is determined by potentiometric titration with silver nitrate solution in the presence of acetone. Copper(II) acetate is added to reduce possible interference from reducing carbohydrates.

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 0,5 mS/m.

5.2 Nitric acid, $c(HNO_3) \approx 1{,}5$ mol/l.

Dilute 100 ml of nitric acid, ϱ 1,40 g/ml, to 1 litre with water (5.1).

5.3 Acetone (CH_3COCH_3), free from chlorides.

5.4 Copper(II) acetate, saturated solution.

Dissolve 13 g of copper(II) acetate monohydrate [$(CH_3COO)_2Cu \cdot H_2O$)] in 200 ml of a 1 % (m/m) solution of acetic acid. Heat gently to facilitate dissolution.

The solution is stable for at least 1 month.

5.5 Silver nitrate, standard volumetric solution, $c(AgNO_3) = 2$ mmol/l.

Accurately weigh approximately 1,70 g of dry silver nitrate ($AgNO_3$), dissolve in water (5.1) in a 100 ml volumetric flask, and dilute to the mark. From this stock solution, pipette 20,0 ml and dilute to 1 000 ml in a volumetric flask. The concentration, c, of this reagent, expressed in millimoles of $AgNO_3$ per litre is then calculated from the formula:

$$c = \frac{2m}{1{,}70}$$

where m is the actual mass in grams, of silver nitrate used for the preparation of the stock solution.

Keep the solution away from light.

6 Apparatus

Glassware and other apparatus shall be scrupulously clean. Boil flasks, beakers and funnels in distilled water. Forceps and scissors for sample preparation shall be kept clean in the same way.

Ordinary laboratory equipment, and

6.1 Potentiometer or other measuring device (for example a pH meter), capable of measuring d.c. voltage in the range 0 to 300 mV with an accuracy of 2 mV. Use a silver wire indicator electrode and a glass electrode or a mercury(I) sulfate electrode as the reference electrode.

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

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

7 Preparation of sample

Since the amount of chlorides 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 by 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.

NOTE — A smaller sample may be taken if the content of soluble chlorides is high.

With a pipette, add 100 ml of water (5.1). Attach a reflux condenser, fitting the flask, and boil gently for 1 h ± 5 min on an electric heater. Avoid any loss of water in the form of steam during this step.

Cool the extract quickly and filter it immediately 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. Add 200 ml of acetone (5.3), 5 ml of nitric acid (5.2) and 2 ml of copper(II) acetate solution (5.4).

Insert the electrodes of the potentiometer (6.1) and stir the solution at a constant speed with a magnetic stirrer for 5 min.

Read the voltage on the potentiometer. Add from the microburette (6.2) portions of the silver nitrate solution (5.5). Read the voltage after each addition. When the voltage starts to change rapidly, add the titrant in smaller portions until the changes level off again.

Plot the voltage readings against the volume of silver nitrate solution added.

If an automatic titrator is used, the titrant shall be added at a rate of 0,1 ml/min to 0,2 ml/min.

From the graph, determine the point of maximum slope and read off the corresponding volume of titrant. Take this as the equivalent of silver nitrate solution.

NOTE — Some automatic titrators allow the first or second derivative of the titration curve to be plotted. If desired, these may be used for the determination of the point of maximum slope.

9 Expression of results

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

$$\frac{35{,}46\ V_3}{V_2} \times \frac{c(V_1 - V_0)}{m}$$

where

c is the actual concentration, in millimoles of $AgNO_3$ per litre, of the silver nitrate solution (standard 2 mmol/l);

V_0 is the volume, in millilitres, of silver nitrate solution consumed in the titration of the blank;

V_1 is the volume, in millilitres, of silver nitrate 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 standard values for c, V_1 and V_3, the formula simplifies to

$$\frac{141{,}8\ (V_1 - V_0)}{m}$$

Take the mean result of the two determinations as the content of water-soluble chlorides. 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:

a) reference to this part of ISO 9197;

b) date and place of testing;

c) identification of the material tested;

d) the results;

e) any departure from the specified procedure, or other circumstances that may have affected the results.