INTERNATIONAL STANDARD

ISO 9197

Third edition
2016-08-15

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

Papier, carton et pâtes — Détermination des chlorures solubles dans l'eau

Reference number
ISO 9197:2016(E)

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 9197:2006), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— the text in 5.4 on the preparation of nitric acid has been corrected;

— the precision statement has been moved to Annex A.

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

1 Scope

This International Standard specifies a method for the determination of water-soluble chlorides in all types of paper, board and pulp. The lower limit of the determination is 20 mg of chloride ion per kilogram of dry sample.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 of a lot — Oven-drying method*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 7213, *Pulps — Sampling for testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
water soluble chlorides
amount of chloride ion that is extracted with cold water and determined under the conditions specified

4 Principle

Pieces of the sample under test are extracted with water at room temperature in a disintegrator. The resulting suspension is filtered and an aliquot is used for determination of the chloride ion content by ion chromatography.

5 Reagents

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

5.1 Distilled or **demineralized water**, conductivity less than 0,2 mS/m.

5.2 Chloride stock solution, c(Cl) = 1 000 mg/l. Dry a portion of potassium chloride, (KCl), at 140 °C. Transfer 210,2 mg thereof to a 100 ml volumetric flask, dissolve the KCl and dilute to the mark with water (5.1). Commercially available standard solutions may be used.

5.3 Chloride matching solution. Dilute the chloride stock solution (5.2) to a mass fraction of chloride ion of, for example, c(Cl) = 10 mg/l. Do not use chloride matching solutions that are more than one week old.

5.4 Nitric acid, $c(HNO_3)$ = 1,3 mol/l. Add with caution 82 ml of concentrated nitric acid, $c(HNO_3)$ = 15,8 mol/l (about 70 % HNO_3), to 500 ml of water (5.1) and dilute to 1 l.

5.5 Additional solutions, as specified in the instructions for the ion chromatograph.

6 Apparatus

Glassware and other apparatus used for this analysis shall be scrupulously clean. Soak all glassware for 5 min to 10 min in the nitric acid (5.4) and then rinse thoroughly with water (5.1). Clean, in water, forceps, scissors and the disintegrator used for sample preparation.

6.1 Wet disintegrator, a high-speed mixer, capable of disintegrating the sample completely with minimum damage to the fibres.

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

6.3 Syringe, Class A, of capacity 5 ml and having a prefilter of about 0,2 µm pore width.

6.4 Tea-strainer or **similar device**, of stainless steel, for removing fibres from a suspension.

7 Sampling and preparation of sample

If the test is being made to evaluate a pulp lot, the sample shall be selected in accordance with ISO 7213. If the test is made on another type of sample, report the source of the sample and, if possible, the sampling procedure used. From the sample received, select specimens so that they are representative of the whole sample.

The procedure to be followed when sampling depends on the particular circumstances in each case. For sampling from lots of pulp, paper or board, the instructions in ISO 7213 or ISO 186, as relevant, are recommended.

Since the amount of chlorides in the sample can be very low, take care not to contaminate it during sampling. Wear clean protective gloves at all times when handling the sample and the test pieces prepared from it.

The laboratory where the analysis is made shall be free from dust and fumes from chlorine-containing substances, such as hydrochloric acid or chlorinated solvents. Particular care should be taken in mill-site laboratories if the mill uses chlorine or chlorine dioxide as a bleaching agent.

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

Analyse specimens as soon as possible after sampling.

Determine the dry matter content on a separate specimen using the procedure specified in ISO 287 (for paper and board) or in ISO 638 (for pulps).

8 Procedure

Carry out the procedure in duplicate. A blank test shall also be carried out in parallel with the entire determination.

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

Select the size of the test piece so that the mass fraction of chloride ion of the extract is within the optimum range of the ion chromatograph.

Transfer the weighed test piece to the disintegrator (6.1) and add 250 ml ± 2 ml of water (5.1) at 23 °C ± 2 °C.

Disintegrate the test piece until it is completely disintegrated, but no longer.

After disintegration, soak the test piece for about 1 h while stirring gently to ensure complete extraction of chloride. Immediately after stopping the gentle stirring, withdraw a portion 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 remove fibrous material. It is essential that the test piece solution be free from suspended material.

Since the operation of the ion chromatograph (6.2) depends on its design, no detailed instructions may be given here. Operate the apparatus as instructed by the manufacturer (see also Annex A).

For calibration, prepare from the chloride 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 test piece solution on the chromatograph as instructed by the manufacturer of the apparatus.

Plot the readings for the calibration solutions against their chloride 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 higher or lower concentration range, as relevant.

Check the calibration several times daily and whenever a new set of calibration solutions is used.

9 Expression of results

Read the chloride ion concentration of the sample solution from the calibration graph. Calculate the mass fraction of chloride ion in the sample from Formula (1):

$$w_{Cl} = \frac{100V\left(\rho_{Cl} - \rho_{Cl,0}\right)}{mX} \quad (1)$$

where

w_{Cl} is the mass fraction of chloride ion, in milligrams per kilogram, in the sample;

ρ_{Cl} is the chloride ion concentration, in milligrams per litre, of the filtered sample solution;

$\rho_{Cl,0}$ is the chloride ion concentration, in milligrams per litre, of the blank solution;

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

m is the mass, in grams, of sample taken;

X is the mass fraction of dry matter, expressed as a percentage, in the sample.

Calculate the mean of the duplicates and report results below 20 mg/kg as “less than 20 mg/kg”, and results of 20 mg/kg or more to the nearest 10 mg/kg.

10 Test report

The report shall include the following information:

a) a reference to this International Standard, ISO 9197:2016;

b) the date and place of testing;

c) the 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 can have affected the result.

Annex A
(informative)

Precision

A.1 General

The following results were obtained in an interlaboratory trial conducted by the Scandinavian Pulp, Paper and Board Testing Committee.

The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 of 20 instances, when comparing two test results for material similar to those described under similar test conditions. These estimates may not be valid for different materials or different test conditions.

NOTE Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where $2{,}77 = 1{,}96\sqrt{2}$.

A.2 Repeatability

Data not available.

A.3 Reproducibility

Nine laboratories analysed four samples as specified in this International Standard. Each sample was analysed in duplicate. The mean mass fraction of chloride ion and the standard deviation (between laboratories) were calculated according to ISO/TR 24498. The results are given in Table A.1.

Table A.1

Sample	Mean mass fraction of chloride ion mg/kg	Reproducibility standard deviation mg/kg s_R	Coefficient of variation CoV,*R* %	Reproducibility limit *R* mg/kg
Machine-glazed (MG) paper from bleached kraft pulp	(14,6)[a]	(3,6)	24,7	10,0
Birch bleached kraft pulp	27,1	6,6	24,4	18,3
Copy paper 1	297	25	8,4	69,2
Copy paper 2	1 240	76	6,1	211

[a] The value is under the lower limit of determination.