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

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

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Published in Switzerland

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9197 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 9197:1998), of which it constitutes a minor revision. A new sentence was added at the beginning of the fifth paragraph of Clause 8, which states: "After disintegration, soak the sample for about 1 h while stirring gently to ensure complete extraction of chloride." There were some minor editorial changes.

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# 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 referenced documents are indispensable for the application of this document. 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 — Oven-drying method*

ISO 638, *Pulps — Determination of dry matter content*

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

ISO 9197:2006

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## 3 Terms and definitions

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

### 3.1

#### **water-soluble chlorides**

(paper, board and pulp) 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**,  $c(\text{Cl}) = 1\,000$  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,  $c(\text{Cl}) = 10 \text{ mg/l}$ . Do not use chloride matching solutions that are more than 1 week old.

### 5.4 Nitric acid, $c(\text{HNO}_3) = 1,3 \text{ mol/l}$ .

Add **with caution** 90 ml of concentrated nitric acid,  $c(\text{HNO}_3) = 14 \text{ mol/l}$  (about 65 %  $\text{HNO}_3$ ), to 500 ml of water (5.1) and dilute to 1 litre.

### 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  $0,2 \mu\text{m}$  pore width.

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

## 7 Sampling and preparation of sample

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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  $\pm$  2 ml of water (5.1) at 23 °C  $\pm$  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 are 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 the expression

$$w_{\text{Cl}} = \frac{100V(\rho_{\text{Cl}} - \rho_{\text{Cl},0})}{mX}$$

where

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

$\rho_{\text{Cl}}$  is the chloride ion concentration, in milligrams per litre, of the filtered sample solution;

$\rho_{\text{Cl},0}$  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 Precision

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

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. The results are given in Table 1.

**Table 1**

Sample	Mean mass fraction of chloride ion	Standard deviation
	mg/kg	mg/kg
Machine-glazed (MG) paper from bleached kraft pulp	(14,6 <sup>a</sup> )	(3,6)
Bleached kraft pulp from birch	27,1	6,6
Copy paper 1	297	25
Copy paper 2	1 240	76

<sup>a</sup> The value is under the lower limit of determination.

## 11 Test report

The report shall include the following information:

- reference to this International Standard;
- date and place of testing;
- complete identification of the sample tested;
- the result, expressed as indicated in Clause 9;
- any departure from the procedure described in this International Standard or any other circumstances which can have affected the result.



## **Annex A** (informative)

### **Laboratory manuals**

The procedure specified in this International Standard relies upon instruments of considerable complexity. Several manufacturers have introduced such instruments to the world market. They are all based on the same principle, but differ in details.

It is a principle of standardization not to specify the use of equipment produced by a particular manufacturer. The reason for this is not only that a standardization body should be neutral with respect to the competition between companies, but also to avoid specifications that will unnecessarily prevent further development of equipment.

In practice, this means that the course of the analysis cannot be described in this International Standard in such detail that it can be used as a laboratory bench manual. For the performance of the analysis, a number of informational details have to be taken from the manufacturer's manual or to be established locally in preliminary tests. Examples are settings of liquid flow, temperatures, power, waiting times.

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