INTERNATIONAL STANDARD

ISO 9197

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

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

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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9197 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps,* Working Group WG 4, *Chemical analysis*.

It cancels and replaces ISO 9197-1:1989 and ISO 9197-2:1990.

There are two major differences between the former version of ISO 9197 and this International Standard.

The extraction is now made with cold water in a disintegrator for a short time whereas the former version specified hot water for 60 min, but no disintegrator. Therefore, the results obtained by this method may differ from those obtained with the former version.

The potentiometric titrations using silver nitrate solutions in ISO 9197-1:1989 and ISO 9197-2:1990 are replaced by ion chromatography. This technique is now widely used for the determination of anions because of its high sensitivity and selectivity. It can be used for all types of paper, board and pulp including high purity products, so there is no longer any need to divide the ISO 9197 into two parts.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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 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 indicated 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 purpose of this International Standard, the following definition applies.

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(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 1 week old.

5.4 Nitric acid, $c(HNO_3)$ = 1,3 mol/l.

Add **with caution** 90 ml of concentrated nitric acid, $c(HNO_3)$ = 14 mol/l (about 65 % 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 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

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 [2] or ISO 186 [1], 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 samples protected, wrapped in aluminium foil or in plastic bags, until required for analysis.

Analyse samples as soon as possible after sampling.

Determine the dry matter content on a separate sample 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 portion, 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 portion so that the mass fraction of chloride ion of the extract is within the optimum range of the ion chromatograph.

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 until it is completely disintegrated, but no longer.

Immediately after stopping the disintegrator, 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 sample 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/I.

Run the calibration solutions and the sample 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

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

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 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 mg/kg	Standard deviation mg/kg
Machine-glazed (MG) paper from bleached kraft pulp	14,6	3,6
Bleached kraft pulp from birch	27,1	6,6
Copy paper 1	297	25
Copy paper 2	1 240	76

11 Test report

The 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 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.

Annex B
(informative)

Bibliography

[1] ISO 186:1994, *Paper and board — Sampling to determine average quality.*

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