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Pulp, paper and board — Determination of total chlorine and organically bound chlorine

Pâtes, papier et carton — Dosage du chlore total et du chlore lié aux matières organiques

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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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11480:1997), which has been technically revised to include the option of using the Schöniger combustion method combined with ion chromatography.

Introduction

This document specifies two procedures for the determination of total and organically bound chlorine in pulp, paper and board. The microcoulometric method (Clause 4) is based on combustion in a heated tube followed by microcoulometric titration. The Schöniger method (Clause 5) is based on the so-called Schöniger combustion followed by ion chromatography.

The choice of procedure depends on the availability of the apparatus and on the risk of interference by bromine compounds in the sample. In the microcoulometric method, the result does not strictly represent the chlorine content since it includes part of any bromine present in the sample. This procedure should thus be avoided if there is a risk that the sample has a high bromine content.

More work is involved in the Schöniger method than in the microcoulometric method. However, since the end determination is performed by ion chromatography, the result is unambiguously chlorine. The Schöniger method also requires a larger sample. Consequently, the result is more representative. On the other hand, the microcoulometric method is faster and several replicate determinations can be performed in a short time.

Pulp, paper and board — Determination of total chlorine and organically bound chlorine

1 Scope

This document specifies two alternative procedures for the determination of total and organically bound chlorine in pulp, paper and board. It is applicable to all types of pulp, paper and board. The lower limit of the determination is about 20 mg/kg.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
total chlorine
total amount of the element chlorine present

3.2
organically bound chlorine
amount of organically bound chlorine present

4 Microcoulometric method

4.1 Principle

Determination of the chlorine content of the sample by combustion under controlled conditions in a quartz tube at high temperature. The combustion gases are fed through an electrolyte solution where all chlorine, now transformed to hydrogen chloride, is absorbed and determined by microcoulometry. For determination of the organically bound chlorine content, the inorganic chlorine compounds are extracted with dilute nitric acid before combustion.

NOTE 1 If determination of organically bound chlorine is not required, the extraction with acid nitrate is omitted.

NOTE 2 Any bromine present will interfere and cause a positive error. For details, see 4.11.

4.2 Chemicals and reagents

All chemicals used shall be of analytical reagent grade. Water for the preparation of solutions, or used in the procedure, shall be distilled water of high purity or equivalent. High blanks (see 4.8) can be due to impure water. The water can then be purified by treating it with activated carbon (4.2.4).

4.2.1 Acid nitrate solution, stock solution. Dissolve 17 g of sodium nitrate ($NaNO_3$) in distilled water. Add 1,4 ml of nitric acid (HNO_3) density 1,40 g/ml, and dilute to 1 l with distilled water. (This solution is only required for the determination of organically bound chlorine.)

4.2.2 Acid nitrate solution, working solution. Dilute 50 ml of the stock solution (4.2.1) to 1 l with distilled water. (This solution is only required for the determination of organically bound chlorine.)

4.2.3 Sulfuric acid (H_2SO_4), density 1,84 g/ml.

4.2.4 Activated carbon, for adsorption of water-soluble organic material in the determination of organically bound chlorine. (Suitable carbon is provided by manufacturers of combustion apparatus.)

4.2.5 Electrolyte solution, for use in the microcoulometer. Dilute 75 ml of glacial (98 %) acetic acid (CH_3COOH) to 100 ml with water.

Addition of sodium perchlorate ($NaClO_4$) and sulfamic acid (NH_2SO_3H) to the absorption solution is recommended by some manufacturers of microcoulometers. Such additions are optional.

One manufacturer recommends a solution prepared by dissolving 1,35 g of sodium acetate ($NaCH_3COO$) in 850 ml of glacial acetic acid and diluting to 1 000 ml with water.

4.2.6 Hydrochloric acid, $c(HCl) = 0{,}010\,0$ mol/l.

4.2.7 2-Chlorobenzoic acid reference solution. Dissolve 110,3 mg of ClC_6H_4COOH in distilled water and dilute to 100 ml in a volumetric flask. This solution contains 250 mg of organically bound chlorine per litre. Dilute the solution as required before use.

4.2.8 Compressed gases. Oxygen is required for the combustion. Other gases may also be required in order to control the combustion. It is essential that all gases used are free from chlorine and bromine in any form.

NOTE It has been reported that chlorinated solvents have been used to clean the gas containers.

4.3 Precautions

Chlorine compounds in small quantities are present almost everywhere, in chemicals, on the surfaces of the equipment, on the skin and in the laboratory air. It is therefore of utmost importance to take every measure to avoid contamination of samples and solutions. In particular, the risk of contamination from the laboratory air should be observed. Such contamination can come from reagents (solvents) stored in the laboratory, as well as from outdoor sources, for example, a bleaching plant.

Clean all equipment before use with dilute nitric acid and flush with pure water.

4.4 Apparatus

Of the items listed below, items 4.4.1 through 4.4.4 are required only if organic chlorine has to be determined. Item 4.4.7 is required only if total chlorine has to be determined.

4.4.1 Conical flasks, 250 ml, of chemically resistant glass, with standard tapered glass stoppers or polytetrafluoroethylene (PTFE)-lined screw caps.

4.4.2 Shaker for the flasks (4.4.1), giving their contents a circular motion. Its power shall be adjustable so that the contents are kept in motion without reaching the stopper.

4.4.3 Filtering device, for vacuum filtration on filters of diameter about 25 mm.

4.4.4 Filters, polycarbonate, nominal pore width 0,4 µm, diameter to fit the filtering device (4.4.3) and having a maximum chlorine content of 0,5 µg.

Alternatively, specially designed filtering cups of heat-resistant glass or ceramic material may be used.

If high blank values (4.8) are obtained, the reason could be contaminated filters. They should be washed before use with stock solution (4.2.1) and then with water.

4.4.5 Combustion apparatus, consisting of a quartz tube connected to a cell for microcoulometric titration (4.4.6). A multizone furnace can heat the middle section of the tube to at least 950 °C and preferably 1 000 °C. A boat of quartz or other heat-resistant material fits into the tube. The boat can be moved from the cold end of the tube to its hot section. The tube shall be wide enough to accommodate a boat loaded with a filter (4.4.4). The apparatus has an oxygen supply and some means for maintaining a constant flow of oxygen through the tube. The oxygen stream may be diluted with an inert gas, such as argon or nitrogen. The combustion gases are fed through the microcoulometric cell for continuous titration of chloride ions.

If required, a heated washing device containing sulfuric acid (4.2.3) for cleaning and drying the gas stream may be inserted between the outlet of the combustion tube and the cell.

4.4.6 Microcoulometer, enabling the determination of 2 µg of chloride ion with a coefficient of variation of less than 10 %, calculated from repeated determinations of chloride ion.

4.4.7 Sample cups, capacity about 1 ml, of quartz or other heat-resistant material, designed to fit into the sample boat. The use of these cups is optional.

4.5 Pretreatment of the sample

Keep samples protected from ambient air in polyethylene bags or in packages of aluminium foil. Use protective gloves whenever handling the sample. Check that no chlorine-containing material is transferred from the gloves to the sample.

Air-dry wet pulp at a temperature not exceeding 40 °C. Using a separate sample, determine the dry matter content as described in ISO 638 or ISO 287, as relevant.

Tear or cut the sample into small pieces using tweezers or scissors. The size of the pieces shall be adapted to the nature of the sample, as well as to the dimensions of the combustion equipment.

Split samples of pulp or paperboard to ensure complete soaking of the sample during the extraction step. If the soaking is incomplete, the results for organically bound chlorine may be too high.

Grind samples of coated or multi-ply paper and paperboard in a small mill, Wiley type or similar. The mill shall not be used for other purposes in order to avoid contamination of samples. Clean the mill carefully after each use.

The total amount of sample required depends on its chlorine content and is limited by the dimensions of the combustion apparatus. Normally, the amount of sample required for each single determination is between 10 mg to 60 mg.

For the determination of both parameters, a total of four sample portions is required. Weigh each sample portion to the nearest 0,1 mg. All samples should have approximately the same mass.

Of the four samples, two are taken for determination of total chlorine, and two are extracted with acid nitrate solution.

NOTE If determination of organically bound chlorine is not required, proceed directly to 4.7.

4.6 Extraction with acid nitrate solution

Transfer the two duplicate samples to 250 ml conical flasks with glass stoppers. Add 100 ml of the acid nitrate solution (4.2.2) and 15 mg of activated carbon (4.2.4) to each flask. At the same time, start two blanks without any sample. Close the flasks and shake them vigorously to wet the sample entirely. Place the flasks in a mechanical shaking machine and shake them for at least 1 h.

When analysing coated or filled papers containing large amounts of carbonate pigments, check that the mixture in the flask is still acid. If not, acidify the mixture by adding more of the acid nitrate solution (4.2.2).

Using the filtering device (4.4.3), filter the contents of the flasks on a polycarbonate filter or in a filtering cup (4.4.4).

Rinse the flask and the sides of the filtering funnel with small portions of the acid nitrate solution (4.2.2), about 25 ml in all. Finally, wash with a small portion of water. Apply suction until excess liquid has been removed.

Drawing large volumes of air through the filter should be avoided, as this can lead to contamination from halogen compounds present in the ambient atmosphere. On the other hand, any excess washing solution present in the filter can result in condensation of water in the combustion tube. Condensed water in the tube can interfere with the combustion (4.7). If the filter is too dry, it can ignite in the drying zone of the oven, which can lead to a low result.

4.7 Combustion

In principle, the procedure is the same for unextracted and extracted samples. In practice, the procedure has to be adapted to the condition of the sample. Extracted samples (for the determination of organically bound chlorine) are wet, and samples for the determination of total chlorine are normally dry.

Operate the combustion apparatus (4.4.5) as instructed by the manufacturer. Several makes of apparatus are on the market. They differ in details and the procedure to be followed has to be adapted to the type of apparatus used. (See Annex C.)

Check the performance of the microcoulometer (4.4.6) by adding known amounts of hydrochloric acid (4.2.6) to the cell. The results should be within 5 % of the theoretical value.

Check the instrument regularly by operating it as instructed for samples, but use no sample.

NOTE Memory effects (false readings are obtained with no sample, in particular after running a sample with a high chlorine content) can be due to corroded combustion tubes.

If the sample is a wet fibre pad on a polycarbonate filter, fold the filter with a pair of forceps and place it in the boat.

If the sample is dry, place it in a sample cup (4.4.7) and load the boat with this cup. The use of sample cups may not be possible with all types of apparatus. The procedure may be modified as required.

Move the boat with the sample to the drying zone of the furnace and allow the water to evaporate. The time required for this depends not only on the amount of water to be removed but also on the design of the apparatus. No water should condense in the cooler parts of the combustion tube.

It is essential that the combustion be controlled at a slow rate so that no soot or condensed water remains upstream of the furnace. If this is the case, take the necessary steps to move all soot and water downstream before taking any reading.

Move the boat to the hot section of the tube. Follow the course of the combustion on the recording device of the instrument. Ensure, by the procedures relevant to the instrument, that the combustion is complete.

If soot is detected downstream from the furnace, the combustion has been incomplete and the result shall be rejected.

There should be no soot in the sulfuric acid in the heated washing device (4.4.5). If soot is observed, clean the device, add fresh sulfuric acid and repeat the determination.

4.8 Blanks

On each day that samples are analysed, check the combustion apparatus (4.4.5) and the microcoulometer (4.4.6), by running at least two blanks by the same procedure as for the samples. The blank value should not exceed 2 µg.

4.9 Checks

Check the entire procedure regularly by running a reference sample of known chlorine content. The reference sample should preferably be of the same type (pulp, paper, board, etc.) as the samples to be analysed.

If no reference sample is available, the chlorine content of a selected sample should be determined by the method of standard additions, using known amounts of the chlorobenzoic acid reference solution (4.2.7). This sample should be used as the reference sample.

The result of the check run should not be less than 91 % or more than 110 % of the assigned value. If the value falls outside the limits given, run a second check. If the deviation persists, check the apparatus for leaks and other defects. Instructions for performing this check should be given in the operating manual.

4.10 Calculation

The procedure for determining the chlorine content of the samples depends on the design of the microcoulometer. See the manufacturer’s manual for instructions.

In calculation, ensure that the blank value is allowed for and that a correction for the moisture content of the sample is made.

Calculate the mean result of parallel determinations. The individual results should not deviate from the mean by more than 10 % (see Annex A for additional information). For mean values less than 50 mg/kg, deviations of up to 5 mg/kg are acceptable. If this criterion is not met, the sample may be heterogeneous. In this case, repeat the preparation of sample and the determination.

Express the results as total chlorine or organically bound chlorine, as relevant, in milligrams per kilogram, with two significant figures.

4.11 Interference from bromine compounds

Chemically bromine will react in the same way as chlorine under the conditions prevailing during the analysis described in this document. This means that any bromine present in the sample will cause a positive error in the result. The magnitude of this error is proportional, but not equal, to the amount of bromine present. The coulometers used are programmed to measure chlorine. If bromine is to be determined, the coulometer has to be recalibrated since bromine has a much higher atomic mass than chlorine.

Normally, the amounts of bromine present in pulp and paper are negligible. Occasionally, significant amounts of bromine have been found in products of recycled fibre from waste paper containing brominated slimicides.

5 Schöniger method

5.1 Principle

Determination of the chlorine content by combustion with oxygen in a Schöniger flask. The chloride formed in the combustion from chlorine compounds present in the sample is determined by ion chromatography.

NOTE If determination of organically bound chlorine is not required, the extraction with acid nitrate is omitted.

5.2 Chemicals and reagents

All chemicals used shall be of analytical reagent grade. Water for the preparation of solutions, or used in the procedure, shall be distilled water of high purity or equivalent. High blanks (see 5.8) can be due to impure water. The water can then be purified by treating it with activated carbon (5.2.4).

5.2.1 Nitric acid (HNO_3), density 1,40 g/ml (approximately 65 % HNO_3) for washing the platinum basket.

5.2.2 Acid nitrate solution, stock solution. Dissolve 17 g of sodium nitrate ($NaNO_3$) in distilled water. Add 1,4 ml of nitric acid (5.2.1), and dilute to 1 l with distilled water. (This solution is only required for the determination of organically bound chlorine.)

5.2.3 Acid nitrate solution, working solution. Dilute 50 ml of the stock solution (5.2.2) to 1 l with distilled water. (This solution is only required for the determination of organically bound chlorine.)

5.2.4 Activated carbon, for purification of water, if necessary.

5.2.5 Eluent solution for ion chromatography, stock solution. The composition of this solution is highly variable depending on the type of ion chromatography column to be used. Therefore, follow the recommendations given by the ion chromatography column supplier.

5.2.6 Eluent solution for ion chromatography, working solution. Prepare, from the concentrated eluent solution (5.2.5), the eluent working solution as recommended by the supplier of the ion chromatography column. Filter the solution through a membrane filter of cellulose acetate or cellulose nitrate before use.

5.2.7 Chloride standard solution, stock solution (1000 mg/l). Dry a portion of potassium chloride, (KCl), at 140°C and dissolve 0,210 2 g thereof in distilled water in a 100 ml volumetric flask and make up to the mark with distilled water.

Commercially available chloride standard solutions may be used.

5.2.8 Nitric acid, for washing the equipment. Add 90 ml of the nitric acid (5.2.1) with caution to 500 ml of distilled water. Dilute to 1 l with distilled water.

5.2.9 Paper with low chlorine content, made, for example, of unbleached kraft pulp.

5.2.10 Compressed gases. Oxygen is used for the combustion. It is essential that compressed gases used in the determination be free from chlorine in any form.

NOTE It has been reported that chlorinated solvents have been used to clean the gas containers.

5.3 Precautions

Chlorine compounds in small quantities are present almost everywhere, in chemicals, on the surfaces of the equipment, on the skin and in the laboratory air. It is therefore of utmost importance to take every measure to avoid contamination of samples and solutions. Especially, the risk of contamination from the laboratory air should be observed. Such contamination can come from reagents (solvents) stored in the laboratory as well as from outdoor sources, for example, a bleaching plant.

Clean all equipment before use with dilute nitric acid and flush with pure water.

5.4 Apparatus

Of the items listed below, items 5.4.1 through 5.4.4 are required only if organic chlorine has to be determined.

5.4.1 Erlenmeyer flasks, 500 ml of chemically resistant glass with standard glass stoppers or polytetrafluoroethylene (PTFE)-lined screw caps.

5.4.2 Shaker for the flasks (5.4.1), giving their contents a circular motion. Its power shall be adjustable so that the contents are kept in motion without reaching the stopper.

5.4.3 Filtration device, for paper filters.

5.4.4 Paper filter, having a low and known chlorine content.

5.4.5 Schöniger flask, volume between 750 ml to 1000 ml. The flask is made of thick, heat-resistant glass and is provided with a ground-in stopper. A basket or net made of platinum is attached to the stopper. An automatic ignition device may also be fitted.

5.4.5.1 Check the flask carefully before using it. A flask with a suspected crack shall not be used.

5.4.5.2 Clean the platinum basket or net with nitric acid (5.2.1). Rinse with distilled water. Clean the basket or net in a flame if any contamination is suspected.

5.4.6 Ion chromatograph, with a fractionating column suitable for the determination of chloride and a conductivity detector.

5.5 Sampling and sample preparation

Keep samples protected from ambient air in polyethylene bags or in packages of aluminium foil. Use protective gloves whenever handling the sample. Check that no chlorine-containing material is transferred from the gloves to the sample.

Air-dry wet pulp at a temperature not exceeding 40 °C. Using a separate sample, determine the dry matter content as described in ISO 638 or ISO 287, as relevant.

If the sample is in sheet form, cut a piece suitable to be attached to the platinum basket. Leave a tail for automatic ignition. Use scissors or forceps, cleaned before use by rinsing them thoroughly with distilled water.

Homogenize samples of coated or of multiply paper or board by grinding them in a small mill, for example of the Wiley type. The mill shall not be used for other purposes in order to avoid contamination of samples. Clean the mill carefully after each use.

Finely divided pulp, for example, fluff pulp, can be compressed into a tablet or wrapped in a chlorine-free paper (5.4.4) before ignition. For each parameter, run the determination in duplicate. Weigh out two different amounts (200 mg to 500 mg) of the original sample to the nearest 0,1 mg.

NOTE If determination of organically bound chlorine is not required, proceed directly to 5.6.

5.6 Extraction with acid nitrate solution

Transfer each sample to an Erlenmeyer flask (5.4.1). Add 200 ml of the acid nitrate solution (5.2.3) to the Erlenmeyer flask. Stopper the flask and agitate it by hand in order to ensure that all sample pieces are soaked. Place the flask in the shaker (5.4.2) and continue the agitation of the sample for 1 h.

NOTE If the sample contains low levels of organic chlorine, the amount of sample used in the determination can be increased up to 500 mg (paper filter included).

Filter the contents of the Erlenmeyer flask using the filtration device (5.4.3) and a paper filter (5.4.4). Wash the sample with 100 ml acid nitrate solution (5.2.3) and rinse the flask and the filtration device with acid nitrate solution. Rinse the sample with 100 ml distilled water. Use vacuum suction until all the water has been removed from the sample.

Dry the sample together with the paper filter at a temperature not exceeding 40 °C.

5.7 Combustion

WARNING — It has been reported that Schöniger flasks can explode during the combustion step. Use protective glasses. If the combustion is carried out in the open, use a face shield.

Using a pair of forceps, place the sample in the platinum basket or net (5.4.5). Add 25,0 ml of distilled water to the Schöniger flask. Flush the flask with oxygen for about 1 min. Ignite the paper strip and stopper the flask immediately as follows:

If the flask has no device for ignition, it is recommended to use a paper strip (5.4.4) of about 3 mm to 5 mm × 40 mm to 50 mm as a wick. Place the strip in the basket or on the net together with the test piece. Ignite the strip, for example, with the spirit lamp. Quickly insert the basket or the net with the burning strip into the flask. Close the flask immediately, turn it upside down and keep it in this position during the combustion.

If an automatic ignition device is fitted on the Schöniger flask, the ignition procedure can be modified and no paper strip is needed.

Watch the combustion carefully. If the combustion is not complete (some carbon remains), the analysis shall be repeated with a new sample.

Do not open the flask when the combustion is complete. Shake the flask and allow it to cool. Shake again vigorously for 15 s and leave the flask for 45 min. It is important that all mist (smoke) has disappeared before the flask is opened.

NOTE More samples are needed if the organic chlorine content is low. In this case, make several combustions consecutively in the same flask. Shake the flask and let it stand without opening it for at least 45 min between the combustions.

5.8 Ion chromatographic determination

The optimum conditions depend on the apparatus and the column. Use the conditions recommended by the manufacturer or determine the conditions empirically. See Annex C.

Transfer the content of the Schöniger flask to a 50 ml volumetric flask. Rinse the flask carefully (shake it) twice with 10 ml portions of distilled water. Transfer the rinsings to the volumetric flask and make up to the mark with distilled water.

NOTE Some chromatographs require the addition of concentrated eluent (5.2.5). Make this addition before filling the volumetric flask to the mark.

Run the standard solution (5.2.7). Check from the chromatogram that the separation is adequate. Measure either the area or height of the chloride peak.

NOTE It has been reported that more reproducible results are obtained when using peak heights instead of peak area.[3]

Run the sample solution in the same manner.

5.9 Blanks

Carry out the whole determination without any sample. If a paper strip (5.4.4) is used as a wick in the combustion, a strip of the same size shall be used in the blank.

5.10 Calculation

5.10.1 Calculate the result from Formula (1):

$$X = \frac{V\left(A_x - A_0\right)CD}{mA_{st}} \tag{1}$$

where

X is the content of total chlorine or organically bound chlorine in the dry sample, in mg/kg;

V is the volume of the sample solution (here 50 ml);

A_x is the area or height of the chloride peak for the sample solution;

A_0 is the area or height of the chloride peak for the blank solution;

A_{st} is the area or height of the chloride peak for the standard solution;

C is the chloride content in the standard solution (5.2.7);

m is the dry mass of the test piece, in grams;

D is the dilution factor (=1, if no dilution has been made).

5.10.2 Mean

Calculate the mean of the results of the duplicate determinations. The results of duplicate determinations should not deviate by more than 10 % from their mean (see Annex B for additional information). For means below 50 mg/kg, deviations up to 5 mg/kg are acceptable.

Express the results as total chlorine or organically bound chlorine, as relevant, in milligrams per kilogram, with two significant figures.

6 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 11480;

b) the method used (Microcoulometric or Schöniger method);

c) date and place of testing;

d) complete identification of the sample tested;

e) mean of the results, expressed in milligrams per kilogram;

f) any departure from the standard procedure and any other circumstances that may have affected the result.

Annex A
(informative)

Precision (Microcoulometric method)

A.1 General

An interlaboratory study comprising 14 laboratories in eight different countries gave the results shown in Tables A.1 to A.2. The laboratories were asked to analyse the samples in triplicate. The means and standard deviations given are based on the average values obtained in each laboratory.

The calculations have been made according to ISO/TR 24498.

The repeatability and reproducibility limits (Tables A.1 and A.2, respectively) 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

Table A.1 — Estimation of repeatability

Sample	**Total chlorine** **Mean value** mg/kg	**Repeatability standard deviation** s_r mg/kg	**Coefficient of variation** CoV, %	**Repeatability limit** R mg/kg
1	66	3,3	5,0	9,1
2	310	3,1	1,0	8,6
3	650	13	2,0	36
4	970	19	2,0	53
Sample	**Organically bound chlorine** **Mean value** mg/kg	**Repeatability standard deviation** s_r mg/kg	**Coefficient of variation** CoV, %	**Repeatability limit** R mg/kg
1	17	0,6	3,5	1,7
2	31	1,5	4,8	4,2
3	120	3,7	3,1	10
4	290	7,3	2,5	20
5	1 600	89	5,6	247

A.3 Reproducibility

Table A.2 — Estimation of reproducibility

Sample	**Number of laboratories**	**Total chlorine** **Mean value** mg/kg	**Reproducibility standard deviation** S_R mg/kg	**Coefficient of variation** CoV, %	**Reproducibility limit** R mg/kg
Copy paper	14	560	28	5,1	78
ECF[a] birch pulp	14	367	25	6,9	70
ECF[a] pine pulp	14	207	10	4,9	28
Copy paper made from TCF[b] pulp	13	347	20	6,0	57
Sample	**Number of laboratories**	**Organically bound chlorine** **Mean value** mg/kg	**Reproducibility standard deviation** S_R mg/kg	**Coefficient of variation** CoV, %	**Reproducibility limit** R mg/kg
Copy paper	14	224	25	11	70
ECF[a] birch pulp	14	339	27	8,0	75
ECF[a] pine pulp	14	193	18	9,6	51
Copy paper made from TCF[b] pulp	11	26	10	39	28

[a] ECF means “elemental chlorine-free pulp”.

[b] TCF means “totally chlorine-free pulp”.

Annex B
(informative)

Precision (Schöniger method)

B.1 General

Total chlorine: For the repeatability data, six samples were analysed in one laboratory. Five replicate analyses were made in each case. For the reproducibility data, three samples were analysed in four laboratories.

Organically bound chlorine: For the repeatability data, two samples of bleached kraft pulp were analysed in one laboratory. Five replicate analyses were made in each case. For the reproducibility data, two samples of bleached kraft pulp were analysed in three laboratories.

The calculations have been made according to ISO/TR 24498.

The repeatability and reproducibility limits (Tables B.1 and B.2, respectively) 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 √2.

B.2 Repeatability

Table B.1 — Estimation of repeatability

Sample	**Total chlorine** **Mean value** mg/kg	**Repeatability standard deviation** s_r mg/kg	**Coefficient of variation** CoV, %	**Repeatability limit** r mg/kg
1	74	3,0	4,0	8,3
2	120	8,4	7,0	23
3	330	16	4,8	44
4	650	20	3,1	55
5	1 090	22	2,0	61
6	1 810	54	3,0	150
Sample	**Organically bound chlorine** **Mean value** mg/kg	**Repeatability standard deviation** s_r mg/kg	**Coefficient of variation** CoV, %	**Repeatability limit** r mg/kg
1	130	6,5	5,0	18
2	300	9,0	3,0	25