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Mechanical and chemical pulps

# **Extractable organic chlorine**

#### 0 Introduction

This SCAN-test Method specifies two alternative procedures for determination of organic chlorine in the substance that can be extracted from pulp with acetone.

In Procedure A, the extractable organic chlorine compounds are combusted in oxygen in a Schöniger flask. The chloride ions formed are determined by ion chromatography.

In Procedure B, the extractable organic chlorine compounds are combusted in a stream of oxygen in a tube and the chloride formed is determined by microcoulometric titration, for example in an AOX apparatus.

Laboratory equipment and facilities. The determination is very sensitive to contamination. All equipment used in taking and treating the sample must be carefully cleaned.

Warning - The determination can be carried out only in facilities where there is no chlorine in the air. Laboratories situated in or next to bleach plants using chlorine do not fulfil this requirement. No hydrochloric acid or chlorinated solvents must be handled in the laboratory.

#### 1 Scope

This method is applicable to all kinds of pulp, including pulps made from recycled fibres, having a content of extractable organic chlorine exceeding 10 mg/kg.

Note - The method can, although it has been developed for pulp, also be used for paper and board.

#### 2 Reference

**ISO 638** Pulps - Determination of dry matter content (EN ISO 638)

Note - SCAN-test has withdrawn a number of test methods and refers instead to the corresponding ISO and/or EN Standards.

Accepted 1999

#### 3 Definitions

For the purpose of this Method, the following definitions apply:

3.1 Extractable organic chlorine, EOCl - The amount of organically bound chlorine in the substance that can be extracted with acetone as specified in this Method.

Total chlorine, TotCl - The total amount of the 3.2 element chlorine present in the material.

3.3 Organic chlorine, OCl - The amount of organically bound chlorine present in the material.

3.4 Inorganic chlorine, InorgCl - The amount of chlorine present in an inorganic form in the material.

Note-The content of chlorine compounds is expressed in milligrams of chlorine per kilogram of oven-dry sample.

#### 4 Literature

SCAN-CM 51 Pulps, papers and boards - Total chlorine

- SCAN-CM 52 Pulps, papers and boards - Organic chlorine
- EN 1485 Water quality - Determination of absorbable organically bound halogens (AOX)

## **Procedure A – The Schöniger combustion method**

#### A.5 Principle

The inorganic chlorine compounds in the sample are displaced by an acid nitrate solution. The sample is dried and extracted by shaking or leaching with acetone.

The extract is concentrated and transferred to a piece of filter paper, which is then combusted with oxygen, by the Schöniger procedure. The hydrogen chloride formed is absorbed in water and the chloride ions are determined with ion chromatography.

#### A.6 Reagents

All chemicals must be of analytical grade. Distilled (or deionized) water used in the preparation of reagents shall contain not more than 0,01 mg/l of chlorine.

A.6.1 *Acetone*, CH<sub>3</sub>COCH<sub>3</sub>, maximum chlorine content 0,025 mg/l. Check the chlorine content of each new batch taken into use.

*Warning* – Acetone is very inflammable. Avoid sources of ignition. Do not leave extractions unattended for any length of time. Work in a fume-cupboard. Avoid skin contact and inhalation of acetone fumes.

A.6.2 *Nitric acid*,  $c(HNO_3) = 14 \text{ mol/l}$  (approx. 65 % HNO<sub>3</sub>, density 1,40 g/ml).

A.6.3 *Nitrate solution*. Dissolve 17 g of sodium nitrate, NaNO<sub>3</sub>, in water. Add 1,4 ml of the nitric acid (A.6.2) and make up to 1 litre with water.

A.6.4 *Washing solution*. Dilute 50 ml of the nitrate solution (A.6.3) with water to 1 litre.

A.6.5 *Compressed gases*. Oxygen is used for the combustion. It is essential that compressed gases used in the determination are free from any chlorinated compounds such as chlorinated solvents.

A.6.6 *Concentrated eluent solution*, for ion chromatography. The composition of this solution is highly variable depending on the type of ion chromatography column used. Therefore follow the recommendations given by the ion-chromatography column supplier.

A.6.7 Concentrated chloride standard solution,  $c(Cl^{-}) = 1000 \text{ mg/l}$ . Dry a portion of potassium chloride, KCl, at 140 °C. Dissolve 0,2102 g thereof in water in a 100 ml volumetric flask and make up to the mark with water.

Commercially available chloride standard solutions may be used.

A.6.8 *Standard solution*. Prepare, from the concentrated standard solution (A.6.7), a working standard solution as recommended by the supplier of the ion-chromatography column.

A.6.9 *Eluent solution* for ion chromatography. Prepare, from the concentrated eluent solution (A.6.6), the eluent solution as recommended by the supplier of the ion-chromatography column. Before use, filter the solution through a membrane of cellulose acetate or of cellulose nitrate.

A.6.10 *Nitric acid*,  $c(\text{HNO}_3)$  approximately 1,3 mol/l, for washing the equipment. Add, with caution, 90 ml of the nitric acid (A.6.2) to 500 ml of water. Dilute to 1 litre with water.

#### A.7 Apparatus

Ordinary laboratory equipment and the items listed below. Wash all equipment with nitric acid (A.6.10) and rinse with distilled water.

A.7.1 Kitchen mixer or similar device.

A.7.2 *Büchner funnel*, diameter for example 110 mm or 130 mm, provided with a wire cloth, mesh width between 45  $\mu$ m and 70  $\mu$ m.

A.7.3 Filtering flask, volume 500 ml.

A.7.4 *Erlenmeyer flask*, capacity 500 ml, of chemically resistant glass, with standard tapered glass stopper or PTFE-lined screw cap (PTFE = polytetrafluorethylene).

A.7.5 *Shaker* for the flasks (A.7.4) giving their contents a circular motion. Its power shall be adjustable so that the contents are kept in motion without reaching the stopper;

or

*an ultrasonic bath*, capacity at least 4 litres (see A.10.2.2).

A.7.6 *Rotary evaporator* or similar device for evaporation at low temperature.

A.7.7 *Filter paper* with a low chlorine content, below 20 mg/kg of total chlorine (TotCl).

A.7.8 *Schöniger flask*, volume between 750 ml and 1000 ml. The flask is made of thick, heat-resistant glass and is provided with a ground-in stopper. A basket or net of platinum is attached to the stopper. An electric ignition device may also be fitted.

Check the flask carefully before using it. Any flask with a suspected crack must not be used.

Clean the platinum basket or net with nitric acid (A.6.2). Rinse with water. Clean the basket or net in a flame if any contamination is suspected.

A.7.9 *Paper of low chlorine content*, made for example from unbleached kraft pulp, to be used as a wick in the combustion flask if it has no electric ignition device.

A.7.10 *Ion chromatograph*, with a fractionating column suitable for the determination of chloride and a conductivity detector.

A.7.11 *Pear shape flask*, volume between 300 ml and 500 ml, having a ground joint that fits into the rotary evaporator (A.7.6).

#### A.8 Sampling and sample pretreatment

Do not touch the sample with unprotected hands. Use protective gloves or relevant tools. Knives, scissors, forceps and other tools must be rinsed with distilled water before use.

Carry out the sampling in such a way that a representative sample is obtained. Keep the sample wrapped in aluminium foil or in a black polyethylene bag. Protect it from chlorine-contaminated air. Dry wet samples at a temperature not exceeding 40  $^{\circ}$ C.

If the sample has to be stored for more than two months before extraction, keep it in a deep-freeze (frozen below -18 °C).

#### A.9 Preparation of sample

*Warning* – See Introduction.

A.9.1 *Weighing*. Carry out the determination in triplicate. Weigh a test portion of between 5 g and 7 g oven-dry basis. Determine the dry matter content as described in ISO 638.

*Note* – If the procedure is used for paper or board, note that samples that are difficult to disintegrate in water can be milled in the dry state. Use a type of mill that does not warm the sample or accumulate resins deposits from the sample.

A.9.2 *Blanks*. In parallel with the real samples, carry out the whole procedure without any sample, including extraction and evaporation of acetone. If a paper strip is used as a wick in the ignition, use a wick of the same size in the blank.

#### A.10 Procedure

A.10.1 *Displacement of inorganic chlorine compounds.* Using the kitchen mixer (A.7.1) disintegrate the sample for 30 s in about 250 ml of the washing solution (A.6.4).

Filter the suspension using the vacuum filtration assembly (A.7.2 and A.7.3). Return the filtrate to the funnel and filter again. Repeat this procedure three more times (5 filterings in total).

Disconnect the vacuum line and moisten carefully the pulp pad in the funnel with a new portion (100 ml) of the washing solution (A.6.4).

After 5 min, connect the filter pump again and drain off the aqueous phase. Finally allow another portion of 100 ml to pass the pulp pad.

Break up the pad into small pieces and let them dry overnight in an oven at a temperature not exceeding 40  $^{\circ}$ C.

A.10.2 *Extraction*. The acetone-soluble material can be extracted either by a shaking procedure or by leaching in an ultrasonic bath: Transfer the dry sample to an Erlenmeyer flask (A.7.4) and add 200 ml of acetone (A.6.1).

A.10.2.1 *Shaking procedure*. Close the flask and shake it for 1 h in the shaker (A.7.5). Filter the suspension through the Büchner funnel (A.7.2) into a filter flask (A.7.3). Transfer the filtrate to a spear shape flask (A.7.1) which can be connected to the evaporator (A.7.6). Wash the pulp pad with 30 ml of acetone and combine the washings with the filtrate. Rinse the filter flask with one more portion (10 ml) of acetone and add this to the combined filtrates. Evaporate the acetone until about 1 ml remains;

A.10.2.2 *Leaching procedure.* Close the flask and shake it manually for 30 s. Place it in an ultrasonic bath (A.7.5) for 30 min. Filter the suspension through the Büchner funnel (A.7.2) into a filter flask (A.7.3). Transfer the filtrate to a pear shape flask (A.7.1) which can be connected to the evaporator (A.7.6). Wash the pulp pad with 30 ml of acetone and combine the washings with the filtrate. Rinse the filter flask with one more portion (10 ml) of acetone and add this to the combined filtrates. Evaporate the acetone until about 1 ml remains.

#### A.10.3 Combustion

*Warning* – It has been reported that Schöniger flasks can explode during the combustion step. Use protective spectacles. If the combustion is carried out in the open, use a face shield.

Using protective gloves, prepare a pad from a piece of filter paper (A.7.7) and place it in the platinum basket or net of the Schöniger flask (A.7.8). Add the extract

or

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dropwise to the pad and let the solvent evaporate. Rinse the flask from the evaporator with 2 ml of acetone (A.6.1) and transfer the rinsing dropwise to the pad. Repeat this step once. Allow the pad to dry exposed to the air for about 1 h.

Add 25 ml of distilled water to the Schöniger flask (A.7.8). Flush the flask with oxygen (A.6.5) for about 1 min.

If the flask has no device for ignition use a paper strip (A.7.9) of about 4 mm by 50 mm as a wick. Place the strip in the basket or on the net together with the pad. Ignite the strip, for example with a spirit-lamp. Quickly insert the basket or the net with the burning strip into the flask. Close it immediately, turn it upside down and keep it in this position during the combustion.

If an electric ignition device is fitted to the Schöniger flask, the ignition procedure can be modified accordingly and no wick is needed.

Watch the combustion carefully. If the combustion is incomplete (some carbon remains) the analysis must be repeated with a new sample.

Do not open the flask directly after combustion, but 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.

A.10.4 *Ion chromatographic determination*. The optimum conditions depend on the apparatus and the column (A.7.10). Use the conditions recommended by the manufacturer or determine the conditions empirically.

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

*Note* – Some ion chromatographs require the addition of a concentrated eluent (A.6.6). Make this addition before making up to the mark.

Run the standard solution (A.6.8). Check from the chromatogram that the separation is adequate. Measure either the area or the height of the chloride peak.

Run the sample and blank solutions in the same manner.

#### A.11 Calculation

A.11.1 Calculate the result from the expression:

$$X = \frac{V(A_x - A_o)C}{A_{st} m}$$
[A.1]

where

- *X* is the EOCl content of the dry sample, in milligrams per kilogram;
- *V* is the volume of the sample solution, in millilitres (here 50 ml);

- $A_x$  is the area or the height of the chloride peak for the sample solution;
- $A_o$  is the area or the height of the chloride peak for the blank solution;
- $A_{st}$  is the area or the height of the chloride peak for the standard solution;
- *C* is the chloride content of the standard solution, in milligrams per litre;
- *m* is the mass of the oven-dry test portion, in grams.

A.11.2 *Mean.* Calculate the mean of replicate determinations and report the mean with two significant figures.

The results of the replicate determinations should not deviate by more than 10% from the mean. For means below 50 mg/kg, deviations of up to 5 mg/kg are acceptable.

### A.12 Report

The test report shall include reference to this SCAN-test Method, Procedure A, and the following particulars:

- (a) date and place of testing
- (b) identification mark of the sample tested;
- (c) the result given to two significant figures;
- (d) any departure from the standard procedure or any other circumstances that may have affected the result.

### A.13 Precision

#### A.13.1 Repeatability

Three samples were analysed in one laboratory using the leaching procedure in the extraction. Three replicate determinations were made for each sample. The results were as follows:

Sample type	Mean,	Coeff. of
	mg/kg	variation, %
Bl. hardwood sulphate pulp	60	2,5
Bl. recovered-paper pulp	12,7	1,5
Unbl. deinked pulp	13	2,5

#### A.13.2 Reproducibility

Two samples were analysed in three laboratories, one of the samples having a content of extractable organic chlorine less than the limited of determination. The following results were obtained:

Sample type	Mean, mg/kg	Coeff. of variation, %
Bl. (Cl-free) hardwood sulphate pulp	8,4	35
Bl. hardwood sulphate pulp	407	12,5

## **Procedure B – The microcoulometric method**

#### **B.5** Principle

The inorganic chlorine compounds in the sample are displaced by an acid nitrate solution. The sample is dried and extracted by shaking or leaching with acetone.

The extract is concentrated and transferred to a sample cup and combusted with oxygen in a quartz tube at a temperature of about 1000 °C. Alternatively, the extract is transferred to a quartz tube by using a needle-injection device and combusted with a mixture of oxygen and inert gas at a temperature of 850 °C.

The chloride formed is absorbed in an electrolyte solution and determined by microcoulometric titration. (This procedure is similar to that described in EN 1485.)

#### **B.6** Reagents

All chemicals must be of analytical grade. Distilled (or deionized) water used in the preparation of reagents shall not contain more than 0,01 mg/l of chlorine.

B.6.1 *Acetone*, CH<sub>3</sub>COCH<sub>3</sub>, maximum chlorine content 0,025 mg/l. Check the chlorine content of each new batch taken into use.

*Warning* – Acetone is very inflammable. Avoid sources of ignition. Do not leave extractions unattended for any length of time. Work in a fume-cupboard. Avoid skin contact and inhalation of acetone fumes.

B.6.2 *Nitric acid*,  $c(\text{HNO}_3) = 14 \text{ mol/l}$  (approx. 65 % HNO<sub>3</sub>, density 1,40 g/ml).

B.6.3 *Nitrate solution*. Dissolve 17 g of sodium nitrate, NaNO<sub>3</sub>, in water, add 1,4 ml of the nitric acid (B.6.2) and make up to 1 litre with water.

B.6.4 *Washing solution*. Dilute 50 ml of the nitrate solution (B.6.3) with water to 1 litre.

B.6.5 *Compressed gases*. Oxygen is used for the combustion. It is essential that compressed gases used in the determination are free from any chlorinated compounds such as chlorinated solvents.

Note 1 – Some instruments require a mixture of oxygen and an inert gas (e.g. argon). Follow the instructions given by the manufacturer.

B.6.6 *Sulphuric acid*,  $H_2SO_4$ , density 1,84 mg/l. (To be used in the washing device in B.7.7.)

B.6.7 Acetic acid, CH<sub>3</sub>COOH, 100 %.

B.6.8 *Electrolyte solution*. Dilute 75 ml of the acetic acid (B.6.7) with water to 100 ml. To be used in the microcoulometer (B.7.8).

*Note* 2 - The addition of sodium perchlorate, NaClO<sub>4</sub>, and sulfamic acid, NH<sub>2</sub>SO<sub>3</sub>H, which is recommended by some manufacturers of micro-coulometers, is optional.

B.6.9 *Nitric acid, c*(HNO<sub>3</sub>) approximately 1,3 mol/l, for washing the equipment. Add 90 ml of the nitric acid (B.6.2) with caution to 500 ml of water. Dilute to 1 litre with water.

B.6.10 2-Chlorobenzoic acid stock solution. Dissolve 110,3 mg of  $ClC_3H_4COOH$  in water and dilute to the mark in a 100 ml volumetric flask. The solution contains 250 mg of organic chlorine per litre.

B.6.11 2-Chlorobenzoic acid standard solution. Dilute 1,00 ml of the stock solution (B.6.10) to 100 ml with acetone. Prepare this solution daily.

#### **B.7** Apparatus

Ordinary laboratory equipment and the items listed below. Wash all equipment with nitric acid (B.6.9) and rinse with distilled water.

B.7.1 Kitchen mixer or similar device.

B.7.2 *Büchner funnel*, diameter for example 110 mm or 130 mm, provided with a wire cloth, mesh width between  $45 \mu m$  and  $70 \mu m$ .

B.7.3 Filtering flask, volume 500 ml.

B.7.4 *Erlenmeyer flask*, capacity 500 ml, of chemically resistant glass, with standard tapered glass stopper or PTFE-lined screw cap (PTFE = polytetrafluorethylene).

B.7.5 *Shaker* for the Erlenmeyer flasks (B.7.4) giving their contents a circular motion. Its power shall be adjustable so that the contents are kept in motion without reaching the stopper;

or

*an ultrasonic bath*, capacity at least 4 litres (see B.10.2.2).

B.7.6 *Rotary evaporator* or similar device for evaporation at low temperature.

B.7.7 *Cups*, capacity 1 ml, of quartz or platinum, fitting in the boat used in the combustion tube (B.7.8). Clean the cups by washing them in distilled water and igniting them in the furnace. When not in use, keep the cups in a clean, closed glass jar;

#### or

*needle-injection device*, arrangement of manual or automatic injector system, using a syringe having a volume between  $10 \ \mu$ l and  $100 \ \mu$ l.

B.7.8 *Combustion apparatus*, a so-called AOX apparatus, consisting of a quartz tube connected to a cell for microcoulometric titration (B.7.9). A multizone furnace can heat the middle section of the tube to  $1000 \text{ }^{\circ}\text{C}$  (850  $^{\circ}\text{C}$  for needle injection). A boat of quartz or other heat-resistant material or the needle of a syringe fits into the tube. The boat should be moved from the cold end of the tube to its hot section. The apparatus has an oxygen supply and some means for maintaining a constant flow of oxygen through the tube.

A heating device and a washing device containing sulphuric acid (B.6.6) for drying and cleaning the gas stream are inserted between the outlet of the combustion tube and the microcoulometer (B.7.9).

B.7.9 *Microcoulometer*, connected to a strip chart recorder or an integrator, enabling to determine  $2 \mu g$  of chloride ion with a standard deviation of less than 10 %.

B.7.10 *Pear shape flask*, volume between 300 ml and 500 ml, having a ground joint that fits into the rotary evaporator (B.7.6).

#### **B.8** Sampling and sample pretreatment

Do not touch the sample with unprotected hands. Use protective gloves or relevant tools. Knives, scissors, forceps and other tools must be rinsed with distilled water before use.

Carry out the sampling in such a way that a representative sample is obtained. Keep the sample wrapped in aluminium foil or in a black polyethylene bag. Protect it from chlorine-contaminated air. Dry wet samples at a temperature not exceeding 40 °C.

If the sample has to be stored for more than two months before extraction, keep it in a deep-freeze (frozen below -18 °C).

#### **B.9** Preparation of sample

*Warning* – See Introduction.

B.9.1 *Weighing*. Carry out the determination in triplicate. Weigh a test portion of between 5 g and 7 g oven-dry basis. Determine the dry matter content as described in ISO 638.

Note – If the procedure is used for paper or board, note that samples that are difficult to disintegrate in water can be milled in the dry state. Use a type of mill that does not warm the sample or accumulate deposits of resins from the sample.

B.9.2 *Blanks*. In parallel with the real samples, carry out a blank through the whole procedure, including extraction and evaporation of acetone.

#### **B.10** Procedure

B.10.1 *Displacement of inorganic chlorine compounds*. Using the kitchen mixer (B.7.1) disintegrate the sample for 30 s in about 250 ml of the washing solution (B.6.4).

Filter the suspension using the vacuum filtration assembly (B.7.2 and B.7.3). Return the filtrate to the funnel and filter again. Repeat this procedure three more times (5 filterings in total).

Disconnect the vacuum line and moisten carefully the pulp pad in the funnel with a new portion (100 ml) of the washing solution (B.6.4).

After 5 min, connect the filter pump again and drain off the aqueous phase. Finally allow another portion of 100 ml to pass the pulp pad.

Break up the pad into small pieces and let them dry overnight in an oven at a temperature not exceeding  $40 \ ^{\circ}C$ .

B.10.2 *Extraction.* The acetone-soluble material can be extracted either by a shaking procedure or by leaching in an ultrasonic bath: Transfer the dry sample to an Erlenmeyer flask (B.7.4) and add 200 ml of acetone (B.6.1).

B.10.2.1 *Shaking procedure.* Close the flask and shake it for 1 h in the shaker (B.7.5). Filter the suspension through the Büchner funnel (B.7.2) into a filtering flask (B.7.3). Transfer the filtrate to a pear shape flask (B.7.10) which can be connected to the evaporator (B.7.6). Wash the pulp pad with 30 ml of acetone and combine the washings with the filtrate. Rinse the filter flask with one more portion (10 ml) of acetone and add this to the combined filtrates. Evaporate the acetone until about 1 ml remains;

or

B.10.2.2 *Leaching procedure*. Close the flask and shake it manually for 30 s. Place it in an ultrasonic bath (B.7.5) for 30 min. Filter the suspension through the Büchner funnel (B.7.2) into a filtering flask (B.7.3). Transfer the filtrate to a pear shape flask (B.7.10) which can be connected to the evaporator (B.7.6). Wash the pulp pad with 30 ml of acetone and combine the washings with the filtrate. Rinse the filter flask with one more portion (10 ml) of acetone and add this to the combined filtrates. Evaporate the acetone until about 1 ml remains.

B.10.3 *Transfer of the extract to a sample cup or to a syringe.* Transfer the acetone extract to a 5 ml volumetric flask and make up to the mark with acetone. By using a pipette, take an aliquot of suitable volume into a sample cup (B.7.7). Evaporate the acetone.

Alternatively, inject by using a syringe an aliquot of suitable volume between  $10 \ \mu$ l and  $100 \ \mu$ l to the combustion apparatus (B.7.8).

B.10.4 *Combustion*. Operate the apparatus as instructed by the manufacturer. Check each day the combustion and the microcoulometer using the 2-chlorobenzoic acid standard solution (B.6.11).

Place the sample cup in the boat by using a pair of forceps.

*Note* – The precise procedure to follow in the combustion step depends on the design of the apparatus.

If any soot flakes are observed in the sulphuric acid, clean and refill the washing device (B.7.8). Wait until the coulometer reading has levelled off and burn a new aliquot of the sample.

#### **B.11** Calculation

B.11.1 The procedure for calculation of the result depends on the design of the microcoulometer (B.7.9). If the coulometer reading is expressed in millicoulombs, the following expression can be used:

$$X = \frac{Mf(A_x - A_o)}{Fm}$$
[B.1]

where

- *X* is the EOCl content of the dry sample, in milligrams per kilogram
- *M* is the relative molecular mass of chlorine,  $35,45 \times 10^{6} \,\mu\text{g}$  per mol;
- f is the correction factor (here 5 ml divided by the volume used in the analysis, see B.10.3);
- $A_x$  is the reading for the sample, in millicoulombs;
- $A_o$  is the reading for the blank, adjusted so that the dilution is the same as for the sample, in milli-coulombs;
- *F* is the Faraday constant,  $96,485 \times 10^6$  millicoulomb per mol;
- *m* the oven dry mass of the test portion, in grams.

If the coulometer reading is in other units than millicoulombs, the calculation has to be modified accordingly. B.11.2 *Mean*. Calculate the mean of replicate determinations and report the result with two significant figures.

The results of the replicate determinations should not deviate by more than 10 % from the mean. For means below 50 mg/kg, deviations of up to 5 mg/kg are acceptable.

#### **B.12** Report

The test report shall include reference to this SCAN-test Method, Procedure B, and the following particulars:

- (a) date and place of testing
- (b) identification mark of the sample tested;
- (c) the result given to two significant figures;
- (d) any departure from the standard procedure or any other circumstances that may have affected the result.

### **B.13** Precision

### B.13.1 Repeatability

Two samples were analysed in one laboratory using extraction by the shaking technique, one of the samples having a content of extractable organic chlorine less than the limited of determination. Four replicate determinations were made for each sample. The results were as follows:

Sample type	Mean,	Coeff. of
	mg/kg	variation, %
Bl. (Cl-free) hardwood	9,2	5,4
sulphate pulp Bl. hardwood sulphate pulp	378	2,2

#### B 13.2 Reproducibility

Two samples were analysed in six laboratories, one of the samples having a content of extractable organic chlorine less than the limited of determination. The following results were obtained:

Sample type	Mean, mg/kg	Coeff. of variation, %
Bl. (Cl-free) hardwood sulphate pulp	10,3	26
Bl. hardwood sulphate pulp	408	12,2

SCAN-test Methods are issued and recommended by KCL, PFI and STFI-Packforsk for the pulp, paper and board industries in Finland, Norway and Sweden. Distribution: Secretariat, Scandinavian Pulp, Paper and Board Testing Committee, Box 5604, SE-114 86 Stockholm, Sweden.