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Wood chips for pulp production and pulp

Content of extractable lipophilic matter

0 Introduction

This SCAN-test Method replaces SCAN-CM 50:94 from which it differs in the following way:

- the extraction solvent has been changed from acetone to a cyclohexane:acetone (9:1) mixture
- the method is also applicable to pulp
- the limit of quantification has been adjusted
- the drying procedure has been changed from 2 h at 40 °C to 30 min at 105 °C.

1 Scope

This SCAN-test Method describes two alternative procedures for the determination of the content of extractable, lipophilic, non-volatile matter in wood chips for pulp production and in pulp.

Note 1 – The method has not been evaluated for papers or for pulp containing recycled fibre.

The lower limit of quantification is 0,05 % for alternative A and 0,1 % for alternative B. The limit of quantification is based on a minimum extract mass of 5 mg.

Note 2 – The limit of quantification can be lowered by increasing the amount of sample analysed.

2 References

ISO 638	Pulps – Determination of dry matter content (EN 20638)
SCAN-CM 39	Wood chips for pulp production – Dry matter content

Note – SCAN-test has withdrawn a number of test methods and refers instead to the corresponding ISO and/or EN Standards.

3 Definition

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

3.1 *Extractable lipophilic matter* – The lipophilic material that can be extracted with a cyclohexane:acetone (9:1) mixture from a wood chip sample or a pulp sample under the conditions specified in this Method.

Note 1 – The extractable lipophilic matter consists mainly of fatty acids, resin acids, fatty alcohols, sterols, di- and triglycerides**,** steryl esters and waxes. In contrast to acetone, a cyclohexane: acetone (9:1) mixture does not dissolve low-molecular phenolic compounds like lignans to any significant extent.

The content of extractable matter according to this method is less than the content extracted with acetone but similar to the content extracted with dichloromethane.

Note 2 – If the sample is acidified to a pH-value below 3 before the extraction, a somewhat larger amount of lipophilic matter is often extracted. This procedure is described in the Annex to SCAN-CM 49 and is recommended for the comparison of the content of solvent-soluble matter in pulp samples with different pH-values. Primarily, the effect of acidification is that water-soluble sodium soaps of fatty acids and resin acids are converted into a water-insoluble acid form and thus become extractable.

Note 3 – In the case of calcium soaps, the acidification described in the Annex is not sufficiently strong and the fatty acids in these soaps will not be fully included in the extract. For this reason, this method cannot be recommended for deinked pulps.

4 Principle

The pulp or the disintegrated wood chips are extracted with a cyclohexane:acetone (9:1) mixture in a Soxhlet apparatus (Alternative A), or in a Soxtec® apparatus or similar extraction equipment (Alternative B). After extraction, the solvent is evaporated and the residue is dried at 105 °C.

The content of extractable lipophilic matter is reported as a percentage of dry wood chips or pulp.

5 Reagents

Warning – Both cyclohexane and acetone are very hazardous solvents with respect to fire. Avoid sources of ignition. Do not leave the extractions unattended for any length of time. Work in a fume-cupboard.

5.1 *Cyclohexane*, C_6H_{12}, pro analysi.

5.2 *Acetone,* CH_3COCH_3, pro analysi.

5.3 *Cyclohexane:acetone (9:1) mixture*, C_6H_{12}: CH_3COCH_3, pro analysi. Mix the solvents in the proportion 9:1 by volume.

6 Apparatus

6.1 Alternative A

6.1.1 *Extraction apparatus of the Soxhlet type* made entirely of glass with ground-in condenser, extractor and flask. Recommended volume of the extractor is (120 – 250) ml.

Note 1 – For pulp samples with a low content of extractable lipophilic matter, an extractor with a larger volume may be used.

6.1.2 *Electric heater*, with a suitable capacity (approx. 200 W per extraction unit), giving an extraction rate of at least 4 cycles per hour.

6.2 Alternative B

6.2.1 *Extraction apparatus of the Soxtec® type*, or similar.

Note 2 – The Soxtec® HT6 apparatus is not recommended for chemical pulps since the smaller amount of pulp used in the determination will increase the limit of quantification.

6.2.2 *Beakers for the extraction* made of aluminium or glass.

6.3 Alternatives A and B

6.3.1 *Extraction thimbles* made of bleached pulp. New extraction thimbles shall be pre-extracted in cyclohexane:acetone (5.3) before use.

Note 3 – Extraction thimbles are not needed for the analysis of pulp samples according to procedure A.

6.3.2 *Boiling beads***,** made of porcelain or similar material, cleaned before use by extraction with cyclohexane:acetone (5.3).

6.3.3 *Glass fibre wool*, cleaned before use by extraction with cyclohexane (5.1).

6.3.4 *Glass filter*, porosity 3.

6.3.5 *Aluminium dishes*, of the patty-pan type, disposable.

6.3.6 *Drying oven*, ventilated, capable of maintaining an air temperature of (105 ± 2) °C.

6.3.7 *Balance,* readable with an accuracy of 0,1 mg.

6.3.8 *Desiccator*.

6.3.9 *Mill of the Wiley type*, or similar (only for wood chips).

7 Sampling and preparation of sample

The sampling procedure is not covered by this method. Make sure that the test portions taken are representative of the sample received.

Keep the samples in a refrigerator in polyethylene bags or in packages of aluminium foil. For long-term storage, samples should be placed in a freezer.

Pulp
Use protective gloves whenever the sample is handled. If necessary, cut or tear the test portion into small pieces, 1 cm by 1 cm.

Wood chips
Disintegrate approximately 50 g of wood chips, to pieces with an approximate length of 2 mm, in a Wiley mill (6.3.9). Make sure that the sample temperature does not exceed 40 °C.

Pulp and wood chips
Using a separate test portion, determine the dry matter content as described in ISO 638 or SCAN-CM 39, as relevant.

If the dry matter content is below 90 %, allow the whole sample to dry overnight in the air at room temperature or in a drying oven at a temperature not exceeding 40 °C. Homogenize the sample and repeat the determination of the dry matter content.

> *Note* – Weighing of a test portion to determine the dry matter content should take place at the same time as weighing of the test portions for extraction.

8 Extraction with cyclohexane:acetone

8.1 Alternative A

Carry out the extraction procedure in duplicate.

If an extraction thimble is not to be used, place a small pad of glass fibre wool (6.3.3) into the draining tube of the extractor (6.1.1).

Weigh the test portion to the nearest 0,01 g and transfer it to the extractor. A suitable test portion size is 2 g – 5 g for disintegrated wood chips and 10 g for pulp samples. For disintegrated wood chips use an extraction thimble. For pulp no extraction thimble is normally needed. Cover the sample with a pad of glass fibre wool (6.3.3).

> *Note 1* – Adjust the test portion weight so that the extract weight will exceed 5 mg.

> *Note 2* – For samples with a low content of extractable lipophilic matter, the limit of quantification can be lowered using an extractor with a larger volume to make it possible to increase the amount of sample extracted. It is also possible to extract two or more sample portions and combine the solvent portion before evaporation.

To the flask (6.1.1), add the boiling beads (6.3.2) and a volume of cyclohexane:acetone (5.3), corresponding to 1,5 times the volume of the extractor. Connect the condenser and start the extraction.

Bring the solvent to the boil and continue the extraction for at least 4 hours for pulp and 6 hours for wood chips. Adjust the rate of emptying of the extractor to at least 4 times per hour. The total number of extraction cycles shall be at least 16 for pulp and at least 24 for wood chips.

8.2 Alternative B

Carry out the extraction procedure in duplicate.

Weigh the test portion to the nearest 0,01 g and transfer it to an extraction thimble (6.3.1). A suitable test portion size for Soxtec® HT2 and other extraction equipment of similar volume is 2 g for disintegrated wood chip samples and 5 g for pulp samples. Cover the test portion with a pad of glass fibre wool (6.3.3).

> *Note 1* – Adjust the test portion weight so that the extract weight will exceed 5 mg.

> *Note 2* – If the extract content is below 5 mg, alternative A is recommended. It is also possible to extract two or more sample portions and combine the solvent portions before evaporation.

Add between 50 ml and 100 ml of cyclohexane:acetone (5.3) to the extraction beaker (6.2.2) and start the boiling. The test portion shall be covered with the solvent. Choose the temperature for the extraction as recommended for cyclohexane by the producer of the extraction apparatus.

Allow the test portion to boil for 15 minutes for pulp or for 30 minutes for wood chips and then move the extraction thimble to rinsing position and rinse for 1 hour for pulp or for 2 hours for wood chips.

8.3 Blank

Carry out a blank extraction using the same solvent and extraction procedure as for the sample, including, if relevant, an empty extraction thimble. Run a blank for each new supply of solvent.

If the mass of the blank residue, *b*, exceeds 0,1 mg, it shall be recorded and taken into consideration in the calculation of the result.

9 Evaporation and drying of the residue

9.1 Evaporation and drying

The solvent can be partially evaporated directly in the Soxhlet (Alternative A) or in the Soxtec® (Alternative B) apparatus.

> *Note 1* – Alternatively, the extract can be transferred to a Zymark tube and evaporated in a Zymark apparatus at 40 °C with nitrogen.

Evaporate the solvent to a residual volume between 25 ml and 30 ml and transfer the extract to a weighed aluminium dish (6.3.5).

Note 2 – If the extraction residue is to be further analysed, glass flasks should be used instead of aluminium dishes, since it is difficult to dissolve the extraction residue and to transfer it quantitatively from the aluminium dishes.

Rinse the flask or the beaker with 3 x 5 ml acetone (5.2) and transfer the rinsings to the aluminium dish.

Note 3 – If the solution with the extraction residue contains visible fibres, it should be filtered through a glass filter (6.3.4) before final evaporation.

Allow the solvent in the aluminium dish to evaporate in a fume-cupboard and finally dry the extraction residue in a drying oven (6.3.6) at 105 °C to constant mass, about 30 minutes.

Note 4 – If the extract is to be analysed further with respect to its composition, drying should be performed at a lower temperature to prevent oxidation, e.g. at 40 °C for 2 hours.

Allow the extraction residue in the aluminium dish to cool to room temperature in a desiccator (6.3.8) and weigh it on a balance (6.3.7) to the nearest 0,1 mg, *a*. A residue less than 5 mg should be reported as being below the quantification limit.

10 Calculation

Calculate the content of extractable lipophilic matter from the expression:

$$X = \frac{(a - b)\,100}{m} \qquad [1]$$

where

X is the content of extractable lipophilic matter in the sample, as a percentage;
a is the mass of the extraction residue, in gram;
b is the mass of the blank residue, in gram;
m is the mass of the oven-dry sample, in gram.

Calculate and report the mean content of the extractable lipophilic matter with two decimals. Report results corresponding to a weighed residue less then 5 mg as “below the quantification limit”.

11 Report

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

(a) date and place of testing;
(b) precise identification of the sample;
(c) the result;
(d) any departure from the procedure described in this method or any other circumstances which may have affected the test results.

12 Precision

12.1 Repeatability

Ten laboratories analysed two wood-chip samples, three kraft pulp samples and three mechanical pulp samples. Six of the laboratories were using Alternative A and four laboratories Alternative B. Double determinations were performed at each laboratory. The repeatablity has been calculated as the coefficient of variation from the two replicates from each laboratory. The following coefficients of variation were obtained:

Sample type (number of laboratories taken part)	Content of extractable lipophilic matter, %	Coefficient of variation, %
Wood chips from birch (10)	1,37	2,4
Wood chips from spruce (10)	0,99	5,7
Spruce groundwood (10)	0,28	4,7
Aspen CTMP (10)	0,20	14,5
Spruce CTMP (10)	0,10	5,4
Bleached softwood kraft (9)	0,06	11,2
Bleached hardwood kraft (9)	0,12	5,5
Unbleached softwood kraft (9)	0,05	6,3

12.2 Reproducibility

Ten laboratories analysed two wood-chip samples, three kraft pulp samples and three mechanical pulp samples. Six of the laboratories used Alternative A and four laboratories Alternative B. The following results were obtained:

Sample type	Content of extractable lipophilic matter, %	Coefficient of variation, %
Wood chips from birch	1,37	10,8
Wood chips from spruce	0,99	10,7
Spruce groundwood	0,28	16,9
Aspen CTMP	0,20	16,8
Spruce CTMP	0,10	23,8
Bleached softwood kraft	0,06	15,1
Bleached hardwood kraft	0,12	10,1
Unbleached softwood kraft	0,05	33,3

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.
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