



Wood chips for pulp production and pulp

Content of acetone-soluble matter

0 Introduction

This SCAN-test Method replaces SCAN-CM 49:93 from which it differs in the following ways:

- the method is also applicable to wood chips
- 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.

When publishing this Method, SCAN-CM 50:94 was also withdrawn.

1 Scope

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

Note 1 – For the determination of the content of lipophilic extractives SCAN-CM 67 is recommended, especially for wood chips and mechanical pulps.

Note 2 – 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 3 – The limit of quantification can be lowered by increasing the amount of sample analysed.

2 References

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Note – SCAN-test has withdrawn a number of test methods and refers instead to the corresponding ISO and/or EN Standards.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

3.1 *Acetone-soluble matter* – The material, which can be extracted with acetone from a wood chip sample or a pulp sample under the conditions specified in this Method.

Note 1 – The amount of acetone-soluble matter in wood chips or pulp provides a measure of the content of wood extractives, often called resin. The acetone-soluble matter includes e.g. fatty acids, resin acids, fatty alcohols, sterols, di- and triglycerides, steryl esters and waxes.

In addition, acetone-extracts of wood chips and mechanical pulps may also contain phenolic compounds such as lignans. For this reason SCAN-CM 67 is recommended for the determination of the content of lipophilic extractives in these kinds of samples. For well-washed chemical pulps, this is

normally not a problem, since such water-soluble compounds are removed during washing. In the case of incompletely washed chemical pulps, the acetone extract will also include dissolved kraft lignin.

Note 2 – If the sample is acidified to a pH-value below 3 before the extraction, a somewhat larger amount of acetone-soluble matter is often obtained. This procedure is described in the Annex and is recommended if the content of acetone-soluble matter in pulp samples having different pH-values are to be compared. The primary reason for the larger amount is that upon acidification 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 is extracted with acetone 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 a temperature of 105 °C.

The content of acetone-soluble matter is reported as a percentage of dry wood chips or pulp.

5 Reagents

5.1 *Acetone*, CH₃COCH₃, pro analysi.

Warning – Acetone is a very hazardous solvent with respect to fire. Avoid sources of ignition. Do not leave the extraction unattended for any length of time. Work in a fume-cupboard.

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 acetone-soluble matter, an extractor with a larger volume may be used.

6.1.2 *Electric heater*, with a suitable capacity (approximately 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*, of aluminium or glass.

6.3 Alternatives A and B

6.3.1 *Extraction thimbles* of bleached pulp. New extraction thimbles shall be pre-extracted in acetone (5.1) 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 acetone.

6.3.3 *Glass fibre wool*, cleaned before use by extraction with acetone.

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 to 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 SCAN-CM 39 or in ISO 638, 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 sample to determine the dry matter content should take place at the same time as weighing the test portions for extraction.

8 Extraction with 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 acetone-soluble matter, the limit of quantification can be lowered by 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 portions before evaporation.

To the flask (6.1.1), add the boiling beads (6.3.2) and a volume of acetone (5.1) 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 a test portion to the nearest 0,01 g and transfer it to an extraction thimble (6.3.1). A suitable test portion size for the Soxtec® HT2 and other extraction equipment of similar volume is 2 g for disintegrated wood chips and 5 g for pulp. 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 acetone (5.1) to the extraction beaker (6.2.2) and start the boiling. The test portion shall be covered with acetone. Choose the temperature for the extraction as recommended by the producer of the extraction apparatus.

Allow the test portion to boil for 15 min for pulps or 30 min for wood chips, and then move the extraction thimble to rinsing position and rinse for 1 h for pulp or 2 h 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 or new batch of recycled 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.

Note – If a batch of recycled solvent is used, the user should define the upper limit for the mass of the blank residue.

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 this 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 (5.1) 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 acetone 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 carried out 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 acetone-soluble matter according to:

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

where

X is the content of acetone-soluble matter of the sample, as a percentage;

a is the mass of the extraction residue, in grams;

b is the mass of the blank residue, in grams;

m is the mass of the oven-dry sample, in grams.

Calculate and report the mean content of acetone-soluble matter with two decimals. Report results corresponding to a weighed residue of less than 5 mg as being "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) information as to whether or not the pulp has been acidified;
- (d) the result;
- (e) 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

Four pulp samples and two wood-chip samples were analysed. Five parallel determinations were carried out for each sample. The following results were obtained:

Sample type (number of laboratories taking part)	Content of acetone- soluble matter, %	Coefficient of variation,* %
Bleached softwood kraft (13)	0,03	16
Unbleached softwood kraft (13)	0,09	8,2
Bleached hardwood kraft (13)	0,24	3,6
Peroxide-bleached groundwood (13)	0,74	3,5
Wood chips from pine (7)	4,33	4,0
Wood chips from birch (7)	2,20	3,2

* Mean CV of the single laboratories.

12.2 Reproducibility

For the same samples, the following reproducibility data were obtained:

Sample type (number of laboratories taking part)	Content of acetone- soluble matter, %	Coefficient of variation, %
Bleached softwood kraft (13)	0,03	23
Unbleached softwood kraft (13)	0,09	19
Bleached hardwood kraft (13)	0,24	8,2
Peroxide-bleached groundwood (13)	0,74	15
Wood chips from pine (7)	4,33	17
Wood chips from birch (7)	2,20	9,2

Note 3 – The repeatability and reproducibility values have been taken from SCAN-CM 49:93 and SCAN-CM 50:93.

Annex – Acidification of pulps before extraction

A.1 Scope and field of application

This Annex describes the acidification procedure for pH correction before the extraction. Normally, a somewhat larger amount of acetone-soluble matter is obtained if the pH of the pulp sample is adjusted with acid to a pH-value below 3 before the extraction.

The procedure described is recommended if the content of acetone-soluble matter in pulps having different pH-values are to be compared, and it is also recommended if the content of acetone-soluble matter is to be determined for incompletely washed pulps, e.g. pulp samples taken from the washing department in a kraft pulp mill.

The difference in the content of acetone-soluble matter between an acidified and a non-acidified pulp depends mainly on the fact that, as a result of the acidification, water-soluble sodium soaps of fatty and resin acids are converted to water-insoluble acid forms and are thus extractable.

A.2 Reagents

A.2.1 *Acetic acid*, CH₃ COOH, 100 % glacial acetic acid.

A.2.2 *Water*, distilled or deionized.

A.2.3 *Acetic acid solution*, pH 2,5. Add approximately 10 ml of acetic acid (2.1) to 340 ml of water (2.2).

A.3 Apparatus

A.3.1 *pH-meter*.

A.3.2 *Food processor*, max. speed 125 rpm.

A.3.3 *Mixer*, type Turmix, max. speed 10000 rpm.

A.3.4 *Büchner funnel*, with a diameter of approximately 130 mm and equipped with a plastic wire with 50 µm sieve opening.

A.3.5 *Filtering flask*, volume 500 ml.

A.3.6 *Drying oven*, adjustable to 40 °C.

A.4 Preparation of sample

If necessary, cut or tear the sample into pieces, approximately 1 cm by 1 cm.

A.5 Acidification

A.5.1 Determine the dry matter content of the sample as described in ISO 638.

A.5.2 Weigh, with an accuracy of 0,01 g, a pulp sample corresponding to 20 g of oven-dried sample and add 350 ml acetic acid solution (2.3).

Note 1 – If necessary, a larger test portion can be acidified at the same time.

Let the test portion stand for 10 minutes under constant stirring in a food processor (3.2.1). Alternatively, the test portion can be disintegrated in a mixer (3.2.2): Disintegrate the test portion for 1 minute, let it stand for 8 minutes and disintegrate once more for 1 minute.

Note 2 – If the test portion is disintegrated in a mixer for 10 minutes, the test material will become too hot.

A.5.3 Separate the acetic acid solution from the test portion using the Büchner funnel (3.3) and collect the filtrate in a clean suction flask (3.4). Rinse the food processor (3.2.1) or the mixer (3.2.2) with the filtrate. Let the filtrate pass through the fibre mat four times with intermediate suction. The pH of the filtrate shall be below 3, otherwise add more acetic acid solution (2.3) to the test portion and repeat the procedure.

A.5.4 Tear the acidified test portion into small pieces and let it dry in a drying oven (3.5) at 40 °C overnight.

A.6 The influence of acidification

The following results have been obtained in an interlaboratory study, in connection with the preparation of the procedure for the determination of the content of acetone-soluble matter from pulps:

Sample type	Content of acetone-soluble matter, %	
	Without acidification	With acidification
Peroxide-bl. groundwood pulp	0,66 ± 0,09 (6)	0,71 ± 0,06 (10)
Unbl. kraft pulp after washing	0,09 ± 0,01 (11)	0,15 ± 0,01 (13)

The table gives the mean value, the 95 % confidence interval and, within brackets, the number of measurements.