INTERNATIONAL STANDARD

ISO 14453

Second edition
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Pulps — Determination of acetone-soluble matter

Pâtes — Détermination des matières solubles dans l'acétone

Reference number
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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. www.iso.org/directives

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 14453:1997), which has been technically revised to include the use of automated extraction, and to add a new precision statement that complies with the requirements of ISO/TR 24498.

Introduction

The amount of acetone-soluble matter in pulp provides a measure of the content of wood extractives, often called resin. The acetone-soluble matter includes fatty acids, resin acids, fatty alcohols, sterols, diglycerides and triglycerides, steryl esters and waxes.

In addition, acetone extracts of mechanical pulps may also contain phenolic compounds such as lignans. In the case of incompletely washed chemical pulps, the acetone extracts will also include dissolved kraft lignin.

Metal soaps of fatty and resin acids, such as those present in unwashed or deinked pulp, are not extracted under the conditions specified in this International Standard.

Pulps — Determination of acetone-soluble matter

1 Scope

This International Standard describes the determination of acetone-soluble matter in pulp.

It is applicable to all types of pulp. The lower limit of the determination is about 0,05 %. This limit can be lowered by increasing the amount of sample analysed.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7213, *Pulps — Sampling for testing*

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.

3.1
acetone-soluble matter
amount of material that can be extracted with acetone from a sample of pulp by the method specified in this International Standard

4 Principle

A pulp sample is extracted with acetone in a Soxhlet apparatus (Option A) or in a Soxtec®[1] apparatus or similar extraction equipment (Option B).

NOTE Extraction with a Soxtec® apparatus is carried out with boiling solvent instead of condensed solvent as is the case with the Soxhlet extraction. The main advantages of the Soxtec® system are much shorter extraction times and recovery of the main portion of the solvent[1] [2]. Other extraction methods, such as Accelerated Solvent Extraction (ASE), can produce different results and are not within the scope of this International Standard[2]. As reported in Reference [2] and based on several interlaboratory studies[3], including that reported in Annex A, no statistically significant differences were found between the two extraction methods.

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 pulp.

5 Reagent

5.1 Acetone (CH_3COCH_3), analytical reagent grade.

WARNING — Acetone is a highly flammable material; therefore only approved electric or steam heating shall be used. The entire procedure must be performed in a chemical fume hood and the

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1) Soxtec® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

extraction should not be left unattended for any length of time. Caution should be taken to avoid open flame. Care should be taken to avoid inhalation, ingestion, or body contact.

6 Apparatus

Ordinary laboratory equipment and the following.

6.1 Option A

6.1.1 Soxhlet extraction apparatus, with ground-glass joints, consisting of a Soxhlet extraction flask of 250 ml capacity, Soxhlet extraction tube, and a Graham or Allihn-type condenser.

NOTE For pulp samples with a low content of acetone-soluble matter, an extractor with a larger volume may be needed.

6.1.2 Electric heater, with a suitable capacity, giving an extraction rate of at least 4 cycles per hour. A hot water bath can also be used.

6.2 Option B

6.2.1 Extraction apparatus of the Soxtec® type, or equivalent.

6.2.2 Aluminium or glass cups for extraction.

6.3 Options A and B

6.3.1 Extraction thimbles, alundum (aluminium oxide), porosity RA 98; fritted glass, coarse porosity; or cellulose pre-extracted with acetone (5.1).

6.3.2 Boiling beads, made of porcelain or similar material, pre-extracted with acetone (5.1).

6.3.3 Glass fibre wool, pre-extracted with acetone.

6.3.4 Glass filter, of porosity 3.

6.3.5 Weighing dishes, of aluminium or other lightweight material. Check that the dishes do not lose or gain mass when subjected to the drying cycle described in Clause 7.

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

6.3.7 Balance, with a precision of 0,1 mg.

7 Sampling and preparation of the sample

Obtain a representative sample of air-dry pulp sufficient to provide two 10 g test specimens and two specimens for determination of dry matter content. If it is found (or known) that the mass of the extract from 10 g does not exceed 5 mg, a greater mass of sample will be required. For sampling from lots and consignments of market pulp, the sample shall be taken in accordance with ISO 7213. If the analysis is made on another type of sample, report the origin of the sample and, if possible, the sampling procedure, and ensure that the specimens taken in Clause 8 are representative of the sample received.

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

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

NOTE Freeze-drying instead of air-drying or oven drying is recommended if the extracts are to be analysed for their chemical composition, in order to avoid possible oxidation of fatty and resin acids.

Cut or tear the test portions into small pieces, 1 cm by 1 cm. Tear flash-dried pulp into suitable pieces no more than 15 mm wide.

8 Procedure

8.1 Extraction with acetone

8.1.1 Option A

Carry out the extraction procedure in duplicate.

Allow the sample to attain moisture equilibrium with the atmosphere near the balance. Weigh two specimens of about 10 g each to the nearest 1 mg. At the same time, weigh two separate specimens for the determination of the dry matter content as described in ISO 638. The mass of the test portion should be adjusted so that the extract mass will exceed 5 mg. For samples with a low content of acetone-soluble matter, an extractor with a larger volume can be used 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.

Transfer one specimen to the extractor (6.1.1). Use a cellulose extraction thimble (6.3.1) or place a porous porcelain disc or a wad of glass fibre wool (6.3.3) over the specimen to prevent any loss. Check that no part of the specimen extends above the top of the draining tube. Always use an extraction thimble if the pulp is shredded or flash dried.

To the extraction 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 a boil and adjust the rate of emptying the extractor to at least 4 times per hour. The total extraction time shall be no less than 4 h.

8.1.2 Option B

Operate the automated extraction apparatus according to the manufacturer's instructions

Carry out the extraction procedure in duplicate.

Allow the sample to attain moisture equilibrium with the atmosphere near the balance.

Weigh two specimens of about 5 g to 10 g each to the nearest 1 mg. The mass of the specimens depends on the extractives level and on the capacity of the extractor. The mass of the test portion should be adjusted so that the extract mass will exceed 5 mg. If the mass of extract is less than 5 mg, two or more sample portions can be extracted and the solvent portions can be combined before evaporation. At the same time weigh two separate specimens for the determination of the dry matter content as described in ISO 638. Transfer the specimen to an extraction thimble (6.3.1).

Add between 50 ml and 100 ml of acetone (5.1) to the extraction cup (6.2.2) and start the boiling. The test portion shall be covered with acetone. Select the temperature for extraction as recommended by the equipment manufacturer.

Allow the test portion to boil for at least 15 min (see Note), and then move the extraction thimble to the rinsing position and rinse for 1 h.

NOTE A boiling period of 15 min is normally sufficient for samples with low extractives content (below 0,5 %), such as bleached chemical pulps. However, for samples with a relatively higher (above 0,5 %) level of extractives, including unbleached pulps and mechanical pulps, a longer extraction period of up to 1 h may be necessary to ensure complete extraction. It is recommended that, for such samples, or for new types of samples, to establish first that there are no significant differences with a 1 h boiling period before adopting a shorter boiling period.

8.2 Blanks

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.

8.3 Evaporation of the solvent and drying of the extraction residue

The solvent can be partially evaporated directly in the Soxhlet (Option A) or in the Soxtec® (Option B) apparatus. Alternatively, the extract can be transferred to a Zymark®[2] tube and evaporated in a Zymark®[2] apparatus at 40 °C with nitrogen.

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

NOTE 1 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 cup with 3 × 5 ml acetone (5.1) and transfer the rinsings to the aluminium dish.

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 hood and finally dry the extraction residue in a drying oven (6.3.7) at 105 °C to constant mass, about 30 min.

NOTE 2 If the extract is to be analysed further for its chemical composition, drying should be carried out at a lower temperature e.g. at 40 °C for 2 h, or preferably by freeze-drying.

Allow the extraction residue in the aluminium dish to cool to room temperature in a desiccator and weigh it on a balance (6.3.1) to the nearest 0,1 mg, *a*.

9 Expression of results

Calculate the content of acetone-soluble matter according to:

$$X = \frac{(a-b)100}{m}$$

where

X is the content of acetone-soluble matter of the sample, in percent;

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.

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[2] Examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) date and place of testing;

c) complete identification of the sample tested;

d) test equipment used: Soxhlet apparatus (Option A) or Soxtec® or similar extraction equipment (Option B);

e) mass of specimen extracted for each determination and extraction time;

f) the results, expressed as a percentage of the oven-dry mass of the pulp sample;

g) any departure from the standard procedure that may have affected the result.

Annex A
(informative)

Precision

A.1 General

This precision statement was obtained from a 2002 round-robin study, with participation from laboratories in Sweden, Finland, Norway, South Africa, and Thailand.

The precision data are based on two pulp samples, a hardwood kraft, and a TMP, tested by 21 different laboratories, according to this International Standard, with the exception that a 15-min extraction time was used for both samples for the Soxtec® method. Since the extractives content in the TMP sample was found to be greater than 0,5 %, a longer extraction time should have been used in this case. For the Soxhlet method, participating laboratories used the same extraction conditions as those specified in this method. Six laboratories used the Soxhlet method, and 15 laboratories used the Soxtec® method (14 for the TMP).

The calculations have been made according to ISO/TR 24498 [4].

The repeatability and reproducibility limits 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 $2,77 = 1,96\sqrt{2}$.

The pooled repeatability and reproducibility data are presented in Tables A.1 and A.2, for the Soxhlet method, and in Tables A.3 and A.4 for the Soxtec® method. The higher coefficient of variation content for the TMP sample by the Soxtec® method is probably due to the fact that a 15-min extraction time was insufficient in this case, since the extractives content was greater than 0,5 %.