
Pulps — Determination of acetone-soluble matter

Pâtes — Détermination des matières solubles dans l'acétone

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

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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.

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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 (CH₃COCH₃), 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

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 $(105 \pm 2) ^\circ\text{C}$.

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 14 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.