INTERNATIONAL STANDARD

ISO 14453

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Pulps — Determination of acetone-soluble matter

Pâtes — Détermination des matières solubles dans l'acétone

Reference number
ISO 14453:1997(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14453 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

It cancels and replaces ISO 624:1974 of which it constitutes a technical revision.

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Introduction

This International Standard has been developed as a replacement for ISO 624:1974, *Pulps — Determination of dichloromethane soluble matter*, the previous solvent extraction method for pulps. The reason for the replacement is the potential health hazard using dichloromethane, the use of which is restricted by law in some countries.

For some pulps the results obtained according to this International Standard are on a higher level than those obtained with ISO 624.

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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, except pulps made entirely or partly from waste paper. The lower limit of the determination is about 0,05 %.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7213:1981, *Pulps — Sampling for testing.*

ISO 638:1978, *Pulps — Determination of dry matter content.*

3 Definition

For the purposes of this International Standard, the following definition applies.

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

4 Principle

The sample is extracted with acetone in a Soxhlet apparatus. After at least 16 extraction cycles the solvent is evaporated and the residue is dried at 105 °C for 2 h and weighed.

5 Reagent

5.1 Acetone (CH_3COCH_3), analytical reagent grade.

WARNING — Acetone is inflammable. Keep away from open fire. Do not use gas heaters. Follow pertinent safety regulations.

6 Apparatus

Ordinary laboratory equipment and

6.1 Soxhlet extraction apparatus, with ground-glass joints, having a 300 ml to 500 ml flask, a Soxhlet extractor and a water-cooled condenser.

6.2 Cellulose extraction thimbles, cleaned by pre-extraction with acetone.

6.3 Glass fibre, cleaned by extraction with acetone.

6.4 Heater, for the extraction apparatus (hot-water bath or equivalent).

6.5 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.

7 Sampling and preparation of the sample

The procedure to be followed when sampling depends on the particular circumstances in each case. For sampling from lots and consignments of market pulp, the instructions in ISO 7213 are recommended.

Use protective gloves whenever handling the sample. Keep samples protected from ambient air in polyethylene bags or in packages of aluminium foil.

Dry wet pulp at a temperature not exceeding 40 °C.

Cut samples of pulp in sheet form into narrow strips to fit the extractor. Tear flash-dried pulp into suitable pieces no more than 15 mm wide.

8 Procedure

Allow the sample to attain moisture equilibrium with the atmosphere near the balance. Weigh two samples of about 10 g to the nearest 1 mg and a separate sample for determination of the dry matter content as described in ISO 638.

Carry out the following extraction procedure in duplicate.

Introduce into the draining tube of the extractor (6.1) a small wad of glass fibre (6.3).

Place the pulp in the extractor (6.1). Use a cellulose extraction thimble (6.2) or place a porous porcelain disc or a wad of glass fibre (6.3) over the sample to prevent any loss. Check that no part of the sample extends above the top of the draining tube. Always use an extraction thimble (6.2) if the pulp is shredded or flash dried.

Add a sufficient volume of acetone (5.1) to ensure that there are at least 50 ml of solvent remaining in the flask when the extractor is ready for siphoning. Assemble the extractor.

Bring the solvent to boiling and continue the extraction until the extractor has emptied at least 16 times. The extraction time shall not be less than 3 h and preferably not more than 4 h.

NOTE — If there is a tendency for the solvent to become super-heated, resulting in uneven boiling, it is advisable to use glass beads or boiling chips in the extraction flasks.

To facilitate the recovery of the acetone, stop the extraction just before the extractor is ready to empty.

Check the extract for the presence of fibres and other visible solids. If necessary, filter the extract through a fritted glass filter of medium pore width.

With the aid of small portions of acetone (5.1) transfer the extract quantitatively to a tared weighing dish (6.5).

WARNING — In order to eliminate potential explosion hazards in the drying oven (next step), allow the remaining acetone to evaporate completely at a temperature not exceeding 40 °C.

Finally dry the extraction residue in a drying oven at (105 ± 2) °C for 2 h.

Allow the dish to cool to room temperature in a desiccator. Weigh it to the nearest 0,1 mg and calculate the mass of the residue.

9 Blanks

Check at regular intervals that the blank value is negligible in comparison with the test result. Run the entire procedure exactly as usual, but without any sample.

If properly done, this test should result in no significant blank. If a significant blank value is obtained, try to eliminate the source, which, for example, may be impurities in the acetone used.

10 Expression of results

For each single extraction, calculate the acetone-soluble matter content, expressed as a percentage of the oven-dry mass of the pulp sample, using the expression

$$x = \frac{m_2}{m_1} \times 100$$

where

x is the acetone-soluble matter content, expressed as a percentage by mass;

m_1 is the mass of the test portion, calculated on an oven-dry basis;

m_2 is the mass of the residue obtained after extraction by acetone and drying in the oven.

Then calculate the mean result of parallel determinations and report it with two decimals. If the mean falls below 0,05 %, report the result as “less than 0,05 %”.

11 Precision

Four samples were analysed by 17 different laboratories distributed worldwide. Each laboratory was asked to follow the procedure in this International Standard exactly and, for each pulp, to report both values obtained. The results have been summarized as follows:

11.1 Repeatability

The average deviation between duplicates was between 3 % and 5 % of the average result for each particular pulp sample.

11.2 Reproducibility

The mean and the coefficient of variation of the reported results (each time the mean of duplicates) were as shown in table 1.

Table 1

Pulp	Mean value %	Coeff. of variation %
Bleached mechanical	0,68	9,9
Birch sulfate, TCF	0,18	8,5
Pine sulfate, ECF	0,15	9,9
Hardwood sulfite	0,24	9,7

TCF, totally chlorine free.
ECF, elemental chlorine free.

12 Test report

The test report shall include the following particulars:

a) reference to this International Standard;

b) date and place of testing;

c) complete identification of the samples tested;

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

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

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