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Tissue paper and tissue products —

Part 17: Determination of disintegration in water

Papier tissue et produits tissue —

Partie 17: Détermination du délitage (désintégration dans l'eau)

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 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6 *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board*.

A list of all parts in the ISO 12625 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes a test method to determine the disintegration in water for tissue paper and tissue products after agitation time periods of 30 s, 2 min and 10 min.

Tissue paper and tissue products —

Part 17:
Determination of disintegration in water

1 Scope

This document specifies a method to assess the disintegration of tissue paper and tissue products when subjected to mechanical agitation in water.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 186, *Paper and board — Sampling to determine average quality*

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 638-1[1)], *Paper, board, pulps and cellulosic nanomaterials — Determination of dry matter content by oven-drying method — Part 1: Materials in solid form*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 14487, *Pulps — Standard water for physical testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
disintegration
process that is characterized by a material breaking into small pieces in water under specified conditions

4 Principle

A test piece of tissue paper or tissue product is placed in a beaker and is agitated in water using an impeller rotating at a constant speed for time durations of 30 s, 2 min and 10 min. After agitation, the beaker contents are poured into a specified sieve, and if there are remains on the sieve, they are

1) Under preparation. Stage at the time of publication: ISO/FDIS 638-1.

collected and analysed gravimetrically to determine the percentages of disintegration after each agitation time period. Precision data are available in Annex D.

5 Reagents

5.1 Deionized water at 23 °C ± 0,5 °C, with a conductivity ≤ 0,25 mS/m at 25 °C, in accordance with ISO 14487.

6 Apparatus

6.1 Transparent beaker with a flat bottom, internal diameter 98 mm ± 5 mm, total height ≥ 150 mm.

6.2 Graduated cylinder, beaker, or other suitable container, having enough capacity to accurately measure and contain 600 ml ± 10 ml of water.

6.3 Stopwatch, with an accuracy of 0,1 s.

6.4 Agitating device with a rotation speed of 800 r/min ± 20 r/min, equipped with a polytetrafluoroethylene (PTFE) screw-propeller. The technical description of the propeller is given in Annex A, Figure A.1.

6.5 Stainless-steel test sieve, conforming to ISO 3310-2, 200 mm in diameter and height 50 mm, with perforated plate with round holes 12,5 mm in diameter.

NOTE Example and picture of typical, suitable and commercially available sieve is given in Annex B, Figure B.4.

6.6 Drying oven capable of maintaining a constant air temperature of 105 °C ± 2 °C, suitably ventilated, and capable of maintaining 80 °C ± 2 °C when test pieces are submitted to accelerated ageing.

6.7 Analytical balance with an accuracy of 0,001 g.

6.8 Drying pans.

6.9 Forceps.

6.10 Desiccator.

6.11 Shower head of diameter 70 mm ± 5 mm, having 50 ± 5 holes of diameter 1,0 mm ± 0,2 mm, attached to a faucet (tap) with a regulator adjusted to deliver 4,0 l/min ± 0,5 l/min.

NOTE Examples and pictures of typical, suitable and commercially available shower head types are given in Annex B.

7 Conditioning of test pieces

Conditioning shall be done prior to the preparation of test pieces, according to ISO 187.

8 Preparation of test pieces

8.1 General

If the tests are being made to evaluate a lot, the sample shall be selected in accordance with ISO 186.

If the tests are being made on another type of sample, make sure that the specimens taken are representative of the sample. Each test piece shall be free from faults not normally inherent to the tissue product.

For converted multi-ply tissue products, testing shall be done on the product as received, with no ply separation.

Tissue papers that have not been converted into finished products shall be tested as single plies, unless otherwise agreed between the parties concerned.

8.2 Accelerated ageing (optional)

The wet strength of tissue paper is frequently enhanced by addition of a wet strength agent. An accelerated ageing with heat, also called curing, is frequently used to develop the maximum wet strength that a tissue paper or tissue product will achieve after a period of natural ageing at ambient conditions, which may vary from a few days to several weeks depending on the wet strength agent used. Accelerated ageing is not a requirement of this document, but is an allowed option.

NOTE 1 The decision of whether or not to use accelerated ageing is determined by the user of this document, based upon the information about the tissue paper or tissue product sample being tested.

There is no rule for determining whether to rapidly age or not, but the following principles are generally applied.

Tissue product which has not left the manufacturing environment is usually subjected to accelerated ageing. It is recommended that this be carried out by heating the samples in an oven at 80 °C ± 2 °C for 30 min and then placing them in the standard atmosphere specified in ISO 187 for at least 1 h prior to testing.

NOTE 2 For quality control of the production process, where test results are required quickly, accelerated ageing conditions of 105 °C ± 2 °C for 15 min can be used, and shall be recorded with the reported results.

Test pieces which have been delivered into the marketing chain, and especially those available for sale to the consumer, are generally not subjected to accelerated ageing.

The test report shall state whether the sample was subjected to accelerated ageing, and if so, the procedure used.

8.3 Mass and shaping of test pieces

Cut samples in the cross direction to provide test pieces with a mass of 1,0 g ± 0,1 g.

If the sample is in continuous sheet form, cut a continuous length of material in the cross direction of such dimensions as to achieve the required mass.

If the sample is in the form of separate units, such as boxed tissue, take sufficient units and, if necessary, cut a portion of a unit in the cross direction as to achieve the required mass.

If the test piece is larger than 12 cm in either direction (machine direction or cross direction), then it shall be folded in middle until it is 12 cm or less in both directions. Only fold the minimum number of times needed to achieve dimensions of 12 cm or less.

Prepare nine test pieces.

Additionally, at least 4 g of sample is required for determination of dry matter content.

9 Test procedure

9.1 General

9.1.1 Perform the whole test procedure at an ambient temperature of 23 °C ± 1 °C.

9.1.2 Determine the dry matter content (X) of the sample according to ISO 638-1.

9.1.3 Prepare 6 l of dionized water at 23,0 °C ± 0,5 °C, and record its temperature to the nearest 0,1 °C.

9.2 Test at 30 s

9.2.1 Take a test piece (that has been conditioned according to ISO 187) and record its mass ($m_{0,\,30\,s}$) to the nearest 0,001 g.

9.2.2 Place the test piece on the bottom of an empty, dry, transparent beaker allowing the paper to bend upwards on the beaker walls no more than 40 mm high.

9.2.3 Put the screw-propeller of the agitating device in a centred position, 50 mm ± 5 mm from the bottom of the beaker as given in Figure A.2. The screw-propeller should not contact the test piece.

9.2.4 Prior to adding water, set the screw-propeller rotating at 800 r/min. Pour 600 ml of deionized water quickly into the beaker in a time less than 3 s, along the rotational axis of the screw-propeller (in order to minimize perturbations due to contact between the test piece and the water). Verify that the test piece is driven under the screw-propeller.

9.2.5 Start the timer as soon as all the water is completely poured into the container.

9.2.6 Stop the agitating device after 30 s.

9.2.7 5 s ± 1 s after stopping the agitation device, start pouring the beaker contents by constantly moving the beaker over the entire surface of the sieve, with the beaker lip 10 cm ± 2 cm from the sieve surface, evenly pouring the entire contents in 5 s ± 1 s.

If pouring is not started within time frame of 5 s ± 1 s, a glass stir rod shall be used to re-suspend the material in the beaker, then poured within 5 s ± 1 s into the sieve. If the beaker contents are not poured into the sieve within the required time frame and using the procedure described above, then the test shall be considered invalid.

9.2.8 Turn on the shower head water flow and adjust it to a flow rate of 4,0 l/min.

9.2.9 Hold the showerhead spray nozzle between 10 cm to 15 cm above the top surface of the sieve, constantly moving the spray over the entire sieve surface with a continuous circular movement of 60 ± 5 rotations per minute, without stopping or concentrating the spray on any specific area. Do not purposely force the passage of any material through the sieve by concentrating the spray on one location.

9.2.10 Stop the rinsing after 1 min.

9.2.11 Collect quantitatively all the remains from both sides of the sieve, by hand and/or by using forceps and/or by backwashing the material into a fine mesh sieve (a 125 µm mesh sieve according to ISO 3310-1 or finer is suitable), and transfer this material into a labelled drying pan.

9.2.12 Repeat 9.2.1 to 9.2.11 on two other test pieces.

9.2.13 Set the oven at a temperature of 105 °C ± 2 °C and dry the three collected remains according to the procedure described in ISO 638-1.

9.2.14 Weigh the dry remains (according to ISO 638-1) and record their mass ($m_{d,\ 30s}$) to the nearest 0,001 g.

9.3 Test at 2 min and 10 min

Repeat the procedure described in 9.2 applying agitation times of 2 min and 10 min respectively (using three test pieces each).

It is permitted not to perform testing at 2 min agitation if no residue has been collected in any replicate in test at 30 s. In this case, the average percentage of material passing through the 12,5 mm sieve after 2 min agitation time in water is considered to be 100 % (since the average result at 30 s is 100 %).

In the same manner, it is permitted not to perform testing at 10 min agitation if no residue has been collected in any replicate in the 2 min testing. In this case, the average percentage of material passing through the 12,5 mm sieve after 10 min agitation time in water is considered to be 100 % (since the average result at 2 min is 100 %).

NOTE Additional agitation time periods can be assessed as an option (see Annex C).

10 Calculation and expression of the results

For each agitation time T and each test piece, calculate the percentage of material passing through the 12,5 mm sieve using the following equation:

$$P_T = 100 \times \left(1 - \frac{m_{d,T}}{m_{0,T} \times X/100} \right)$$

where

P_T is the percentage of material passing through the 12,5 mm sieve for each test piece after an agitation time in water T (30 s, 2 min, 10 min);

$m_{d,T}$ is the dry mass of the collected remains corresponding to each test piece after an agitation time ($_T$) in water, recorded to the nearest 0,001 g;

$m_{0,T}$ is the initial mass of each test piece used for the test at time T (shall be 1 g ± 0,1 g) recorded to the nearest 0,001 g;

X is the dry matter content of the sample, determined according to ISO 638-1, recorded to 3 significant figures.

Calculate and record the average and standard deviation of the three replicates of the percentage of material passing through the sieve at each of the three time periods ($\overline{p}_{30s}$, $\overline{p}_{2min}$, $\overline{p}_{10\ min}$) to three significant figures.

NOTE Percentages of material passing through the sieve can be 100 % for one or more of the replicates. For example, if two of the three replicates are 100 %, and one is 94,0 %, the reported results is 98,0 % with a standard deviation of 3,46 %.

11 Test report

The test report shall include at least the following information:

a) Reference to this document, i.e. ISO 12625-17:2021;