INTERNATIONAL STANDARD

ISO 5263-3

Second edition
2023-02

Pulps — Laboratory wet disintegration —

Part 3: Disintegration of mechanical pulps at ≥85°C

Pâtes — Désintégration humide en laboratoire —

Partie 3: Désintégration des pâtes mécaniques à une température supérieure ou égale à 85 °C

Reference number
ISO 5263-3:2023(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 (see 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 (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 172, *Pulp, paper and board,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 5263-3:2004), which has been technically revised.

The main changes are as follows:

— the requirement to reduce the temperature of the suspension immediately following disintegration has been made explicit.

A list of all parts in the ISO 5263 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.

Pulps — Laboratory wet disintegration —

Part 3: Disintegration of mechanical pulps at ≥85°C

1 Scope

This document specifies an apparatus and the procedures for the laboratory wet disintegration of mechanical pulps that exhibit latency except when brightness is measured. This apparatus and procedure can be used for preparation of the test portion in other International Standards dealing with pulps.

This document is applicable to all kind of mechanical pulps (i.e. mechanical, semi-chemical and chemi-mechanical pulps) exhibiting latency.

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

ISO 4119, *Pulps — Determination of stock concentration*

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 terminology databases for use in standardization at the following addresses:

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

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

3.1
disintegration of mechanical pulp
mechanical treatment in water so that interlaced fibres, which were free in the pulp stock, are again separated from one another without appreciably changing their structural properties

3.2
latency
condition of a mechanical pulp in which some of its properties are inhibited and require disintegration of the pulp at elevated temperature to be developed

Note 1 to entry: Latency is due to the distorted form of the fibres acquired in mechanical processing, especially at high consistency, and subsequently preserved upon cooling at high consistency. It is assumed that its preservation is caused by the hardening of the lignin.

Note 2 to entry: The degree of latency in a pulp is generally related to the consistency and energy applied during the mechanical processing.

3.3
latency removal
procedure using a combination of mechanical treatment, i.e. disintegration, and heat treatment using a temperature exceeding the softening temperature of lignin

Note 1 to entry: In this document, the temperature for latency removal is stated to be not less than 85 °C.

4 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

4.1 Disintegrator with heat supply or hot-water supply capable of maintaining a temperature of ≥85 °C in the suspension during the entire disintegration procedure. Use either the standard disintegrator (4.1.1) or the circulation disintegrator (4.1.2).

Since the two types of disintegrator do not treat the fibres in the same way (see Reference [5]), the disintegrator used shall be reported.

4.1.1 Standard disintegrator, in accordance with Annex A. For latency removal, the standard disintegrator shall be equipped with an electrically heated container or shall have a supply of hot water, capable of keeping the temperature of the fibre-water suspension at the temperature stated in 4.1.

The procedure for checking the standard disintegrator is given in Annex B.

For safety reasons, it is not recommended to use a standard disintegrator intended for disintegration at 20 °C and an electric hot plate for boiling water.

4.1.2 Circulation disintegrator, of Domtar type, in accordance with Annex C. The disintegrator shall have a supply of water of a temperature of between 90 °C and 95 °C.

There shall be a means of running the pump for short intervals of time. To prevent damage to the pump, the pump shall not be run dry for more than 3 s.

4.2 Balance, capable of weighing with an accuracy of 0,2 g.

4.3 Water, standard water, as specified in ISO 14487, distilled water or tap water, depending on the requirement in the procedure for which the disintegrated pulp will be used.

5 Preparation of test portion

If the pulp is wet or air-dry, determine the dry matter content in accordance with ISO 638-1. If the pulp is in slush form, determine the dry matter content in accordance with ISO 4119.

If the concentration of a slush pulp sample is less than 1,5 % by mass, thicken it to the appropriate volume, carefully avoiding the loss of fines. This is most easily done by sedimentation of the suspension and removing a part of the water phase or by dewatering through a filter paper on a Büchner funnel.

Use standard water (4.3) for disintegrating pulps to be used in tests where drainability properties are of importance. In all cases, use water of the same quality as required in the procedure for which the pulp is disintegrated.

For the standard disintegrator (4.1.1), take a test portion corresponding to a mass of (50 ± 5) g of oven-dry pulp. For the circulation disintegrator (4.1.2), take a test portion corresponding to a mass of (56 ± 1) g of oven-dry pulp. If the sample is in sheet-form, do not cut the sheets and avoid taking cut edges.

If the dry matter comprises 20 % or more of the sample, soak the test portion in 1 l to 1,5 l of water (standard water or other) at (20 ± 5) °C for at least the minimum soaking time specified in Table 1.

If the pulp is in the form of sheets or slabs, after soaking tear the sample into pieces of dimensions approximately 25 mm x 25 mm. Soaking for a time longer than the minimum specified, for example overnight, has been found not to have any significant effect on the results. The soaking time shall, however, never be longer than 24 h for any pulp grade.

Flash-dried mechanical pulp needs to be soaked for a minimum of 10 min.

Table 1 — Recommended soaking time for mechanical pulps

Dry matter content of the pulp % by mass	Minimum soaking time
<20	0 min
20 to 60	30 min
>60	4 h

Where necessary for climatic reasons, a temperature of between 25 °C and 30 °C may be used, provided that this is stated in the test report.

6 Procedure

6.1 General

This procedure shall be used to remove the latency from all pulps that exhibit latency, except when brightness is to be measured, in which case the pulp shall be disintegrated in accordance with ISO 5263-2. Mechanical pulps not exhibiting latency shall be disintegrated in accordance with ISO 5263-2.

NOTE Brightness is not significantly altered by the presence of latency; however, hot disintegration of mechanical pulps can lead to significant loss of brightness.

6.2 Disintegration and latency removal

WARNING — Since the hot-disintegration procedure involves the treatment of the test portion at a temperature equal to or exceeding 85 °C, caution shall be taken to prevent scalding.

Information regarding the effect of latency in mechanical pulps (or in pulps having a high lignin content) is given in Annex D.

6.3 Standard disintegrator

Transfer the test portion, after preparing as described in Clause 5, into the container of the standard disintegrator (4.1.1).

Add water of the same quality as used in Clause 5 to give a total volume of (2 500 ± 25) ml. Use the heater to heat the mixture to a temperature ≥85 °C. Set the revolution counter to zero. Switch the motor on and allow the propeller to make 30 000 revolutions. Stop the propeller and check visually that the pulp is completely disintegrated, for instance by diluting a small portion from the disintegrator with water in a glass cylinder and inspecting it under transmitted light. If it is not completely disintegrated, continue the disintegration until complete separation of fibres is achieved and/or the fibre bundles and fragments are separated to the extent expected in the pulp at the time of manufacture. At the end of the disintegration, the temperature shall not be less than 85 °C. If for any reason a different pulp charge or a different number of revolutions is used, this shall be stated in the test report.

Immediately after disintegration, and as quickly as possible, dilute the pulp suspension using cold water of the same quality as used in Clause 5, to a concentration not less than 0,3 % and not more than 1,5 %

and a temperature of (20 ± 2) °C. Ice that meets the requirements specified in 4.3 may also be used if necessary. For freeness testing, the stock concentration required is (0,3 ± 0,02) %.

NOTE The hot disintegration step described is usually sufficient to release the latent properties. It is possible that some latency returns if the stock is cooled slowly and without dilution. For this reason, the dilution and cooling to 20 °C is done as quickly as possible.

6.4 Circulation disintegrator

To heat the circulation disintegrator (4.1.2), fill the disintegrator container with water at a temperature between 90 °C and 95 °C to within 4 cm of the top of the container. Close the lid tightly. Start the recirculation pump and allow it to operate for (120 ± 5) s. After the pump has stopped, open the drain of the container slightly and measure the temperature of the water leaving the container. Drain the water completely and repeat this cycle until this temperature exceeds 90 °C.

Immediately add water of the same quality as used in Clause 5, at a temperature between 90 °C and 95 °C until the container is approximately half-full. Transfer the test portion, after preparing as described in Clause 5, to the circulation-disintegrator container and add water at a temperature between 90 °C and 95 °C to within 4 cm of the top of the container. Close the lid tightly and start the timer to operate the pump for (120 ± 5) s. Use caution when removing the lid since pressure can build up during the operation.

Measure the temperature and repeat the disintegration if the temperature is below 85 °C. If fibre bundles and fragments are not separated to the extent expected in the pulp at the time of manufacture, continue the disintegration.

When the disintegration is completed, open the drain valve and run the pump for a brief interval to drain the test portion into a collection pail. Open the lid and, with the drain valve closed, add about 4 l of hot water into the container. Close the lid, run the pump for 2 s, then open the drain valve flushing the remainder of the test portion from the system. Never run the pump for more than 3 s without liquid in it to avoid damaging it.

Immediately after disintegration, and as quickly as possible, dilute the pulp suspension using cold water of the same quality as used in Clause 5, to a concentration not less than 0,3 % and not more than 1,5 % and a temperature of (20 ± 2) °C. Ice that meets the requirements specified in 4.3, may also be used if necessary.

NOTE 1 The preliminary step with hot water is done to heat the container, piping and pump.

NOTE 2 The hot disintegration step described is usually sufficient to release the latent properties. It is possible that some latency returns if the stock is cooled slowly and without dilution. To minimize any such tendency, the dilution and cooling to 20 °C are done as quickly as possible.

NOTE 3 If the water to which the stock is added is at a temperature above 20 °C, the temperature after dilution will be considerably higher than 20 °C. To overcome this, ice made from the same quality water can form part of the dilution water. The volume and temperature of the dilution water can be so chosen as to bring the stock concentration and temperature to within (0,30 ± 0,02) % and (20 ± 2) °C respectively for freeness testing.

7 Test report

The test report shall include the following particulars:

a) reference to this document (e.g. ISO 5263-3:2023);

b) all the information necessary for complete identification of the sample;

c) the water grade (standard water, distilled water or tap water) used;

d) the soaking time;

e) the dry matter content of the sample;

f) the method used for the hot disintegration;

g) any unusual features observed in the course of the test;

h) any operations not specified in this document, or that are regarded as optional, which might have affected the results.

Annex A
(normative)

Construction of the standard disintegrator

A.1 Materials

All components that come into contact with pulp suspensions shall be resistant to water and to dilute acids and alkalis. Stainless steel is normally used.

A.2 Standard disintegrator

The cylindrical container, Figure A.1, is fitted with four equally spaced spiral baffles extending between 32 mm from the bottom and 57 mm from the lid, each baffle traversing half the internal circumference of the container. The baffles spiral downwards in a clockwise direction. There is a fillet of radius 13 mm around the inside of the base of the container. The three-bladed propeller is mounted on a vertical shaft centrally in the container, at a fixed distance above the bottom. It is driven at the specified speed in the stock and a counter is fitted to record the number of revolutions. The counter should preferably be of the pre-set type, which will switch off the disintegrator after the required number of revolutions. Viewed from above, the propeller rotates in a clockwise direction.

The container is provided with a lid, which in most disintegrators is fitted to the propeller/motor assembly.

The container is fixed firmly in position during operation of the disintegrator, but it is capable of being removed and replaced easily and quickly.

A.3 Dimensions

Table A.1 — Standard disintegrator part dimensions

Part	Dimension	Specified Value (unless otherwise stated)	Tolerances
container	internal height	191 mm	±2 mm
	internal diameter	152 mm	±2 mm
	radius of fillet	13 mm	±2 mm
baffles	square section	6,5 mm	±1 mm
	height from container base	32 mm	±1 mm
	distance from rim	57 mm	±1 mm
	ends radiused	3 mm	±0,5 mm
	edges radiused	0,4 mm	±0,1 mm
	spacing (centres)	51 mm	±1 mm
propeller	diameter of swept circle at tip of blades	90 mm	±0,5 mm
	diameter of hub	≥22 mm	-
	distance between propeller blades and container base (lowest point)	25 mm	±2 mm
propeller blades	width at hub	18,2 mm	±0,5 mm