INTERNATIONAL STANDARD

ISO 5263-3

First edition
2004-09-01

Pulps — Laboratory wet disintegration —

Part 3:
Disintegration of mechanical pulps at $\geqslant$ 85 °C

Pâtes — Désintégration humide en laboratoire —

Partie 3: Désintégration des pâtes mécaniques à $\geqslant$ 85 °C

Reference number
ISO 5263-3:2004(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 5263-3 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

This first edition cancels and partially replaces ISO 5263:1995 which has been technically revised. In the revision, ISO 5263 has been divided into three parts; Part 1 which is applicable to chemical pulps, Part 2 which is applicable to mechanical pulps without latency and Part 3 which is applicable to mechanical pulps exhibiting latency. In Part 3, an informative Annex has been inserted describing the effect of latency in mechanical pulps.

ISO 5263 consists of the following parts, under the general title *Pulps — Laboratory wet disintegration*:

— *Part 1: Disintegration of chemical pulps*

— *Part 2: Disintegration of mechanical pulps at 20 °C*

— *Part 3: Disintegration of mechanical pulps at ⩾ 85 °C*

Pulps — Laboratory wet disintegration —

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

1 Scope

This part of ISO 5263 specifies an apparatus and the procedures for the laboratory wet disintegration of mechanical pulps that exhibit latency. This apparatus and procedure are required for preparation of the test portion in a number of other International Standards dealing with pulps.

ISO 5263-3 is applicable to all kind of mechanical pulps (i.e. mechanical, semi-chemical and chemi-mechanical pulps) exhibiting latency. Mechanical pulps not exhibiting latency shall be disintegrated according to ISO 5263-2.

The procedure specified in ISO 5263-2 should be used to disintegrate all mechanical pulps to be measured for brightness.

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

2 Normative references

The following referenced documents are indispensable for the application 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, *Pulps — Determination of dry matter content*

ISO 4119, *Pulps — Determination of stock concentration*

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

3 Definition

For the purposes of this part of ISO 5263, the following definitions apply.

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 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 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 In this part of ISO 5263, the temperature for latency removal is stated to be not less than 85 °C.

4 Apparatus

Ordinary laboratory apparatus and the following:

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 [6]), the disintegrator used must be reported.

4.1.1 Standard disintegrator, constructed as described in 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.

NOTE 1 The procedure for checking the Standard disintegrator is given in Annex B.

NOTE 2 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, constructed as described in Annex C. The disintegrator shall have a supply of water of a temperature of between 90 °C and 95 °C.

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

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

4.3 Standard water, for physical testing, as specified in ISO 14487.

5 Preparation of test portion

If the pulp is wet or air-dry, determine the dry matter content in accordance with ISO 638. 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 × 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.

NOTE 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 Disintegration and latency removal

WARNING — Since the hot-disintegration procedure involves the treatment of the test portion at a temperature exceeding 85 °C, cautions must 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.

6.2 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 ⩾ 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, dilute the pulp suspension using cold water of the same quality as used in Clause 5, to a concentration not less than 3 g/l. If necessary, cool the suspension to approximately 20 °C.

6.3 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 (2,0 ± 0,1) min. 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.

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

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 (2,0 ± 0,1) min. Use caution when removing the lid, since pressure might 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, cool the pulp suspension to approximately 20 °C.

7 Test report

The test report shall include the following particulars:

a) reference to this part of ISO 5263 (e.g., ISO 5263-3:2004);

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 International Standard, 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.

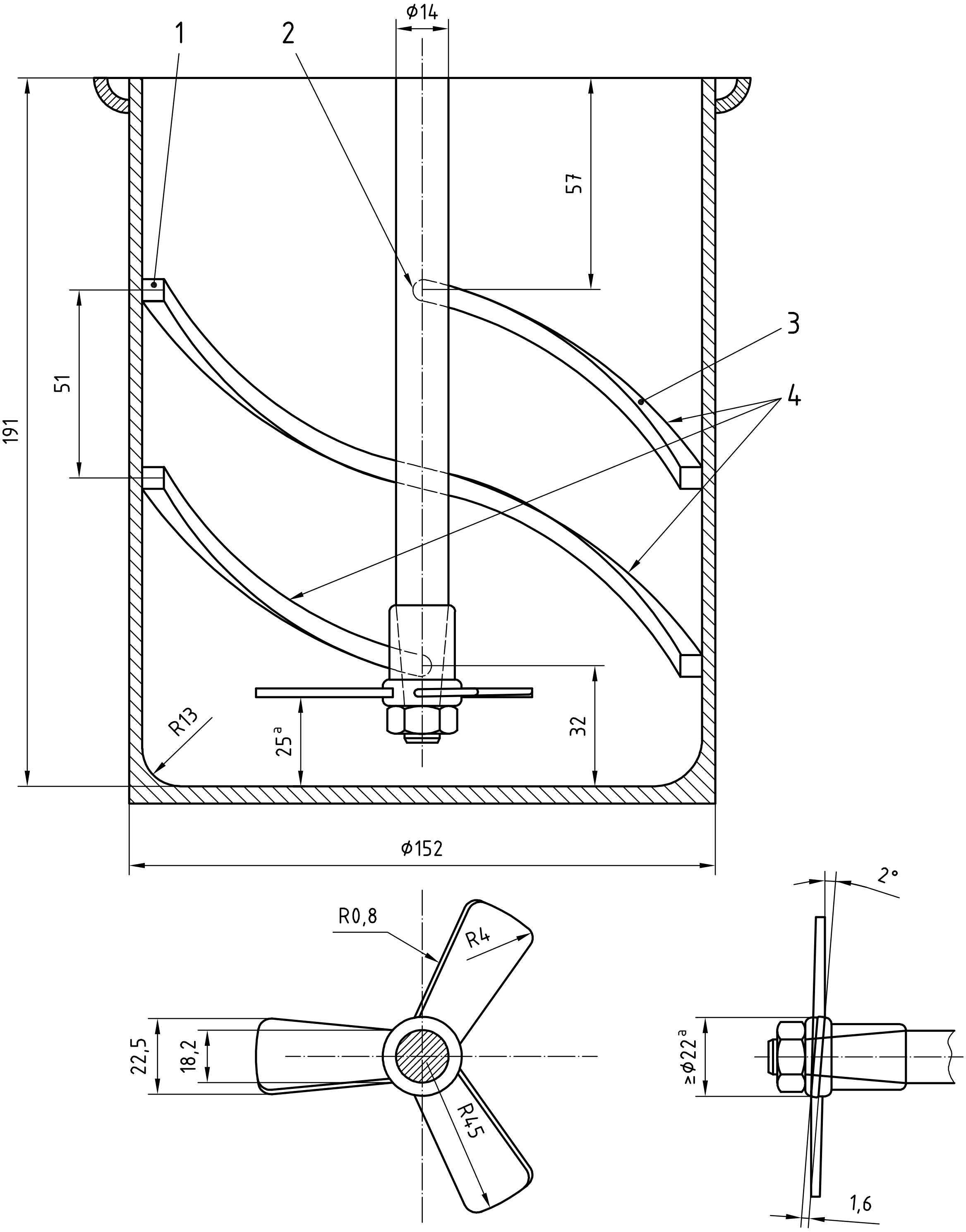
A.3 Dimensions

Part	Dimension	Specified value (unless otherwise stated)	Tolerance
container	internal height internal diameter radius of fillet	191 mm 152 mm 13 mm	± 2 mm ± 2 mm ± 2 mm
baffles	square section height from container base distance from rim ends radiused edges radiused spacing (centres)	6,5 mm 32 mm 57 mm 3 mm 0,4 mm 51 mm	± 1 mm ± 1 mm ± 1 mm ± 0,5 mm ± 0,1 mm ± 1 mm
propeller	diameter of swept circle at tip of blades diameter of hub distance between propeller blades and container base (lowest point)	90 mm ⩾ 22 mm 25 mm	± 0,5 mm — ± 2 mm
propeller blades	width at hub maximum width thickness edges radiused ends radiused pitch	18,2 mm 22,5 mm 1,6 mm 0,8 mm 4 mm 2°	± 0,5 mm ± 0,5 mm ± 0,5 mm ± 0,2 mm ± 1 mm ± 15'
propeller shaft	diameter end taper	⩽ 20 mm to fit any propeller hub	—

A.4 Rotational frequency

The rotational frequency of the propeller shaft is (49 ± 1,5) s^{-1}.

Dimensions in millimetres



Key

1 6,5 mm × 6,5 mm section

2 end R 3

3 round edges R 0,4

4 four baffles, each a half-turn around the container (three shown)

a Not to scale.

Figure A.1 — Standard disintegrator details

Annex B
(normative)

Checking the Standard disintegrator

Check the Standard disintegrator regularly. Special care shall be taken to ensure that

a) the propeller shaft rotates smoothly and is always centred with respect to the container;

b) the propeller runs at the specified rotational frequency;

c) the propeller blades are set as specified (this may be checked by means of a propeller gauge);

d) the dimensions of the propeller blades are as specified (see A.3) and the propeller blades are not damaged.

If the apparatus is used properly, the other dimensions of the Standard disintegrator should remain constant; they shall, however, be checked at intervals.

Annex C
(normative)

Circulation disintegrator

The Circulation disintegrator, of Domtar type, consists of a container, a centrifugal pump and a timing device.

C.1 Container, of stainless steel having a diameter of 0,1 m and equipped with a closed system with a total volume of 3 l.

To avoid cavitation at the pump inlet, the fibre suspension shall enter and leave the container tangentially. The container shall have a tightly fitting lid. To release steam and to prevent pressure build-up, the lid contains a vent hole 8 mm in diameter in the centre. A concentric baffle on the lid, one-half of the container diameter, prevents splashing through the vent hole see [5] and [6].

NOTE A larger container (having a diameter of 0,2 m) with a water volume of 7 l is also available. In this case, the pulp charge is (136 ± 2) g of oven-dry pulp.

C.2 Centrifugal pump, for continuous circulation of the fibre suspension, with an electric motor with a rating of between 0,56 kW and 0,75 kW, capable of delivering approx. 240 l/min at 3 450 rpm.

The pump shall have an inlet pipe of 38 mm nominal size and an outlet of 25 mm nominal size. Parts of the pump and associated piping in contact with the fibre suspension shall be of stainless steel or other corrosion-resistant material. The equipment shall have a valve to drain the system.

C.3 Timing device, to control the operation of the circulation pump to an interval of (2,0 ± 0,1) min.

Annex D
(informative)

The effect of latency in mechanical pulps

D.1 Introduction

When, in 1966, Beath *et al.*[1] established the fact that sequestered properties can be made available by simple appropriate treatment and are thus latent within the pulp, they called the phenomenon “latency”.

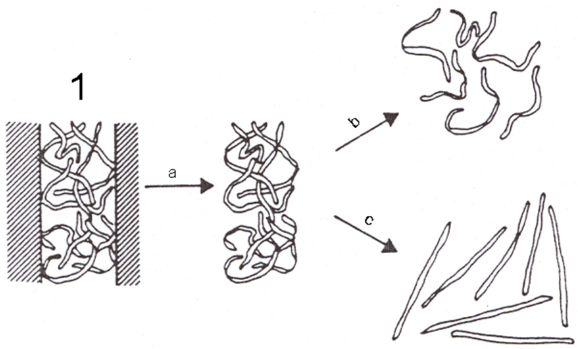
Latency may be exhibited in pulps with a high lignin content, e.g. pulps produced by mechanical fibre separation. This annex describes the phenomenon of latency and gives some examples of the changes in pulp properties that occur when latency is released from a pulp by hot disintegration.

D.2 The phenomenon called latency

In production of mechanical pulps, the fibres are separated at a temperature exceeding the softening temperature of lignin. If, in the separation procedure, the fibre material is curled and twisted, this condition may be made permanent after cooling; see Figure D.1. Latency is not released from the pulp by disintegration in water at room temperature.

To remove the latency from a pulp, it is necessary to disintegrate the fibres in a water suspension at a temperature exceeding the softening temperature of lignin, i.e. by hot disintegration at a temperature of ⩾ 85 °C.

NOTE The softening temperature of lignin is influenced by the chemical treatment and will, in turn, influence temperature at which the fibre separation occurs see [3].



Key

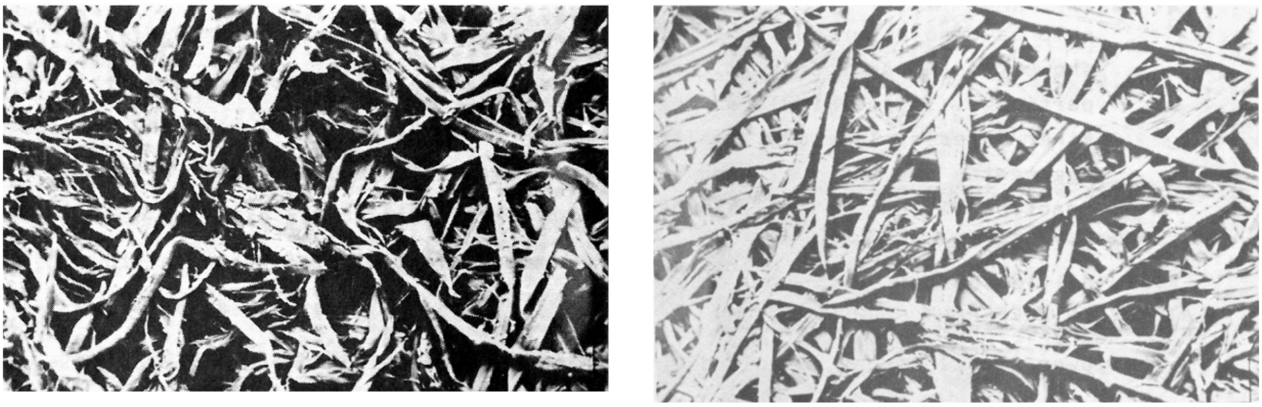
1 refiner

a Cooling.

b Cold disintegration at 25 °C.

d Hot disintegration at ⩾ 85 °C.

Figure D.1 — The effect of hot and cold disintegration on latency in mechanical pulps; Reference [2]



a) b)

Figure D.2 — SEM photographs of pulp fibres with latency (a) and without latency (b); Reference [4]. Fibre fraction, Bauer McNett, passed 30 mesh, retained 50 mesh

The crookedness of the fibres results in different properties from those the pulp would have if the fibres were straight.

D.3 The effect of latency on pulp properties

Hot disintegration, i.e. treatment at low consistency and high temperature, of groundwood pulp (SGW) and refiner mechanical pulp (RMP) will reduce the CSF-value and increase the burst strength, compared to cold disintegration, i.e. treatment at low consistency and low temperature see [1].

Tensile properties and bursting strength are the properties of thermomechanical pulp (TMP) which are most strongly influenced by latency see [2]. Bonding properties (Scott-Bond and z-directional tensile strength) and tear index are not influenced by latency.

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