
**Pulps — Determination of water retention
value (WRV)**

Pâtes — Détermination de la valeur de rétention d'eau (VRE)

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Published in Switzerland

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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 23714 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

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Introduction

The water retention value (WRV) is an empirical measure of the capacity of a test pad of fibres to hold water. The WRV-value increases with increasing beating because of internal fibrillation, a widening of the small internal pores and delaminations, which has been called “swelling” and which occurs concurrently with the development of external fibrils, which also serve to hold additional water.

This test may be useful for the pulp producer as a guide to the influence of the pulping process on the fibre produced and the papermaking potential of the pulp. It is also useful for the papermaker as a measure of the efficiency of pulping and refining.

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Pulps — Determination of water retention value (WRV)

1 Scope

This International Standard specifies a procedure for the determination of the water retention value (WRV) of all kinds of pulp.

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 5263-1, *Pulps — Laboratory wet disintegration — Part 1: Disintegration of chemical pulps*

ISO 5263-2, *Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 °C*

ISO 5263-3, *Pulps — Laboratory wet disintegration — Part 3: Disintegration of mechanical pulps at ≥ 85 °C*

ISO 7213, *Pulps — Sampling for testing*

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

3 Terms and definitions

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

3.1

water retention value

ratio of the mass of water retained after centrifugation under specified conditions by a wet pulp sample to the oven-dry mass of the same pulp sample

4 Principle

A test pad consisting of pulp fibres is formed by dewatering a pulp suspension on a glass-fibre filter. The test pad is centrifuged under a specified centrifugal force for a specified time, weighed, dried and weighed again. The water retention value (WRV) is calculated from the wet mass of the centrifuged test pad and the dry mass of the test pad.

NOTE The results obtained from a rewetted pulp will not be the same as those from a corresponding never-dried pulp.

5 Reagents

5.1 Standard water, as specified in ISO 14487.

If any other type of water is used, this shall be reported.

6 Apparatus

6.1 Laboratory centrifuge with a swing-out head and buckets, made of an inert material such as steel or anodized aluminium, about 100 ml in volume and about 45 mm in inner diameter. The centrifugal force shall be $(3\,000 \pm 50) g$ (g is the acceleration due to gravity, i.e. 9.81 m/s^2) at the position of the test pad, approximately 15 mm from the bottom of the bucket. The centrifuge shall be equipped with a timer and a brake.

It is desirable that the centrifuge should maintain a working temperature of $(23 \pm 3) ^\circ\text{C}$.

NOTE If g is the acceleration due to gravity, i.e. 9.81 m/s^2 , then the rotational frequency is obtained using the equation:

$$N = \left(\frac{Z \times 896}{r} \right)^{\frac{1}{2}} \quad (1)$$

where

N is the rotational frequency, in min^{-1} or r/min ;

Z is the centrifugal force, $(3\,000 \pm 50) g$;

r is the distance between the centre of rotation and the filtration plane of the glass filter (radius of gyration), in metres.

6.2 Büchner funnel or similar funnel, made of non-corrosive material, the perforated bottom of which shall be flat, and having an internal diameter larger than 30 mm.

6.3 Wire cloth of glass-fibre filter, e.g. Whatman grade GF/C filter or equivalent.

6.4 Suction flask, connected to a water suction pump or similar device for vacuum filtration applied to the Büchner funnel.

6.5 Set of test-pad-holding units. The holding unit is a metal tube, $(30 \pm 5) \text{ mm}$ in inner diameter, with a phosphor-bronze wire screen attached to one end. The wire-screen shall have a nominal size of aperture of $125 \mu\text{m}$ and a preferred diameter of the wire of $90 \mu\text{m}$. The centrifuge tube should be capped with a hole in the centre to prevent moisture loss.

The design of the holding unit depends on the design of the centrifuge and is therefore not specified in this International Standard. The unit shall fit into the centrifuge buckets in such a way that the test pad in the test-pad-holding unit will not be rewetted after centrifugation. Two examples of test-pad-holding units are described in Annex A.

The number of test-pad-holding units required depends on the design of the centrifuge. All units shall have the same mass.

6.6 Weighing bottles with lids, having a volume of 25 ml.

6.7 Oven, capable of maintaining $(105 \pm 2) ^\circ\text{C}$.

7 Sampling

If the test is being carried out to evaluate a pulp lot, the sample shall be selected in accordance with ISO 7213. If the test is made on another type of sample, report the source of the sample and, if possible, the sampling procedure used.

Make sure that the test portions taken are representative of the pulp sample.

8 Preparation of sample

If the pulp sample is in dried form, disintegrate the pulp in standard water (5.1) in accordance with ISO 5263-1 or ISO 5263-2.

If a mechanical pulp has been disintegrated at a temperature higher than 85 °C, in accordance with ISO 5263-3 to remove latency effects, then before proceeding further, the diluted stock sample must be allowed to cool to (23 ± 3) °C.

If the sample has been disintegrated, the disintegration method shall be stated in the test report.

Dilute the stock in standard water (5.1) to give a concentration in the range from 2 g/l to 5 g/l. For samples that drain slowly, use the highest concentration.

It shall be stated in the test report whether the sample is rewetted or never-dried, since there could be significant differences in the results.

9 Procedure

9.1 General

From the diluted and well-stirred stock, take samples and carry out WRV determinations at (23 ± 3) °C in duplicate. Carry out all determinations as soon as possible after sampling.

If, for practical reasons, WRV determinations are carried out a day or more after sampling, the WRV-value will be somewhat higher (normally less than 0,03 g/g) than it would have been if the determination had been carried out the same day. Any significant delay in carrying out the WRV tests shall be reported.

9.2 Forming in a Büchner funnel

Connect the Büchner funnel (6.2) to the suction flask (6.4). Put the glass-fibre filter in the funnel, moisten and then start the suction. Add to the Büchner funnel a volume of the stock, so chosen that the test pad formed will have an oven-dry grammage of $(1\,700 \pm 100)$ g/m², when placed in the test-pad-holding unit (6.5).

For the volume of stock chosen, 100 ml of this volume should be filtered and then the suction stopped. If necessary (if the retention of fines is too low), add this initial filtrate to the remainder of the stock volume to be filtered.

Stop the suction when all the surface water has directly disappeared. The dry-matter content of the test pad should then be between 5 % and 15 %.

Remove the pad from the Büchner funnel and place it in a test-pad holder.

9.3 Centrifuging the test pad

Place the complete test-pad-holding unit with the test pad in a centrifuge bucket (see 6.1). Centrifuge at a speed that gives a centrifugal force of $(3\,000 \pm 50) g$ at the bottom of the pad for $30 \text{ min} \pm 30 \text{ s}$. This period shall include neither acceleration nor retardation.

The temperature of the test pad during the centrifugation may influence the result, and it is therefore recommended that the centrifuge be able to maintain a working temperature of $(23 \pm 3) ^\circ\text{C}$. Otherwise, the temperature of centrifugation should be included in the report. If multiple centrifuging tests are to be run, then some time should be allowed for the centrifuge to cool between each run, to be within the proper temperature range.

Immediately after the centrifuge has stopped, transfer the test pad to a preweighed weighing bottle (6.6). Weigh it to the nearest 1 mg. Place the open weighing bottle together with its lid in a drying oven (6.7) and dry to constant mass at a temperature of $(105 \pm 2) ^\circ\text{C}$ (normally overnight). Close the weighing bottle and allow it to cool in a desiccator, raise the lid briefly to achieve pressure equalization and weigh the closed bottle to the nearest 1 mg.

10 Calculation

Calculate the water retention value (WRV), in grams per gram, using the expression:

$$\text{WRV} = \frac{m_1}{m_2} - 1 \quad (2)$$

where

m_1 is the mass of the centrifuged wet test pad, in grams;

m_2 is the mass of the dry test pad, in grams.

Calculate the mean of the duplicates and report the result to two decimal place.

The results of the parallel determinations shall not differ by more than 5 % from their mean.

11 Precision

11.1 Repeatability

Different kinds of pulp samples were tested in one laboratory in accordance with this International Standard. Five to ten determinations were made in each case. Mean values and coefficients of variation for each sample are shown in Table 1.

Table 1 — Repeatability for determination of water retention value (WRV)

Sample	WRV g/g	Coefficient of variation %
Bleached softwood	1,02	2,6
Bleached hardwood	1,01	2,8
TMP ^a	1,39	5,2
CTMP ^b (hardwood)	1,22	4,5
CTMP ^b (50 % HW ^c + 50 % SW ^d)	1,24	3,8
Unbleached sulfite	1,04	2,4
Recycled pulp	1,59	9,3
Non-wood pulp	2,04	2,0
Never-dried kraft pulp	1,14	0,94
^a TMP: thermo-mechanical pulp ^b CTMP: chemi thermo-mechanical pulp ^c HW: hardwood ^d SW: softwood		

Mechanical/semi-mechanical and recycled pulps contain a high percentage of fines, as do some refined chemical pulps. The presence of fines can be seen in the repeatability results.

11.2 Reproducibility

Different kinds of pulp samples were tested in six different laboratories in accordance with this International Standard. Mean values and coefficients of variations for each sample are shown in Table 2.

Table 2 — Reproducibility for determination of water retention value (WRV)

Sample	WRV g/g	Coefficient of variation %
Bleached softwood	0,86	15,1
Bleached hardwood	0,86	16,8
TMP	1,28	17,8
CTMP (100 % HW)	1,23	* 37,7
CTMP (50 % HW + 50 % SW)	1,02	14,3
Unbleached sulfite	0,94	20,8
Recycled pulp	1,31	25,3
Non-wood pulp	1,60	17,6
[*] One result was higher than the other results, which is the reason why the coefficient of variation in that sample is not as good as in the other samples		

The quite high coefficient of variation values in reproducibility results are probably due to the deviation between laboratories, instruments, as well as pulp unevenness, including the fines content.

12 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) all information necessary for complete identification of the sample;
- c) the date and place of testing;
- d) rewetted or never-dried pulp used;
- e) the results, as specified in Clause 10;
- f) any departure from this International Standard, or any other circumstances or influences regarded as optional, that could have affected the results.

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