

## Chemical pulp

# Water Retention Value

## 0 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.

## 1 Scope

This SCAN-test Method describes a procedure for the determination of the Water Retention Value (WRV) of chemical pulp.

*Note* – The method is not applicable to mechanical pulps or pulps from recycled fibre.

## 2 References

- ISO 638 Pulps – Determination of dry matter content (EN 20638)
- ISO 4119 Pulps – Determination of stock concentration (EN ISO 4119)
- ISO 5263-1 Pulps – Laboratory wet disintegration - Part 1: Disintegration of chemical pulps (EN ISO 5236)
- ISO 14487 Pulps – Standard water for physical testing

*Note* – SCAN-test has withdrawn a number of test methods and refers instead to the corresponding ISO and/or EN Standards.

## 3 Definition

For the purpose of this Method, the following definition applies:

3.1 *Water Retention Value* – The ratio of the mass (weight) of water retained after centrifugation under specified conditions by a wet pulp sample to the oven-dry mass (weight) of the same pulp sample.

*Note* – The temperature of the test pad during the centrifugation influences the result and is therefore specified to be  $(23 \pm 3)$  °C.

## 4 Principle

A test pad consisting of pulp fibres is formed by dewatering a pulp suspension on a wire screen or on a filter paper or alternatively on a test-pad former. The test pad is centrifuged under a specified centrifugal force for a specified time, weighed, dried and weighed again. The Water Retention Value is calculated from the wet mass (weight) of the centrifuged test pad and the dry mass (weight) of the test pad.

*Note 1* – The two alternative procedures of forming the test pad, described in this method, will give different results, see Annex B. Thus the procedure used for forming the test pad must be reported.

*Note 2* – Neither of the two alternative procedures is considered to be more correct than the other.

**5 Reagents and chemicals**

5.1 *Standard water* as specified in ISO 14487.

*Note* – If any other kind of water is used, this must be reported.

**6 Apparatus**

6.1 *Laboratory centrifuge* with a swing-out head and buckets, made of steel or anodized aluminium, about 100 ml in volume and about 45 mm in inner diameter. The centrifugal force shall be  $(3000 \pm 50)g$  at the position of the test pad, approximately 15 mm from the bottom of the bucket. The centrifuge shall be able to maintain a working temperature of  $(23 \pm 3)^\circ\text{C}$  and be equipped with a timer and an electric brake.

*Note 1* –  $g$  is the acceleration of free fall, i.e.  $9,81 \text{ m/s}^2$ .

6.2 *Büchner funnel* or similar of non-corrosive material, the perforated bottom of which shall be flat, and having an internal diameter of between 65 mm and 95 mm.

6.3 A *wire cloth* of nylon with an aperture size of  $(71 \pm 10) \mu\text{m}$  or Munktell filter paper no 3.

*Note 2* – If the test pad is formed using a test-pad-holding unit (6.5), the equipment described in 6.2 and 6.3 is not necessary.

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

6.5 A *set of test-pad-holding units*. Each unit must include a test-pad former. The former 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 the aperture of  $125 \mu\text{m}$  and a preferred diameter of the wire of  $90 \mu\text{m}$ .

The design of the holding unit depends on the design of the centrifuge and is therefore not specified in this Method. The unit shall fit into the centrifuge buckets in such a way that the test pad in the pad former 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.

**7 Sampling and preparation of sample**

The sampling procedure is not covered by this Method. Make sure that the samples taken are representative of the gross sample received.

If the pulp sample is in dried form, disintegrate the pulp in standard water (5.1).

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 higher concentration.

**8 Procedure**

From the diluted and well-stirred stock, take samples and carry out the WRV determination in duplicate. This method describes two different procedures of forming the test pad.

*Note 1* – The two different procedures of forming the test pad will give different results, see Annex B, but neither of the two alternative procedures is considered to be more correct than the other.

**8.1 Alternative 1 – Forming in a Büchner funnel**

Connect the Büchner funnel (6.2) to the suction flask (6.4). Put the wire or the filter paper in the funnel, moisten it 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  $(1700 \pm 100) \text{ g/m}^2$ , when placed in the test-pad-holding unit.

If necessary (if the retention of fines is too low), stop the suction and bring back about 100 ml of the first filtrate. Inspect the water in the suction flask to ensure that the retention of fines is not too low.

**8.2 Alternative 2 – Forming in a test-pad former**

Connect the test-pad-holding unit (6.5) to the suction flask (6.4) and attach a funnel to the test-pad-former. Add to the funnel a volume of the stock, so chosen that the test pad formed will have an oven-dry grammage of  $(1700 \pm 100) \text{ g/m}^2$ .

If necessary (if the retention of fines is too low), stop the suction and bring back about 100 ml of the first filtrate. Inspect the water in the suction flask to ensure that the retention of fines not is too low.

**8.3 Alternatives 1 and 2**

Stop the suction when the dry matter content of the test pad is between 5 % and 15 %. Alternatively, stop the suction when the dry matter content of the test pad is still below 30 % and rewet it to a dry matter content between 5 % and 15 %.

*Note 2* – Dewatering to a dry matter content higher than 30 % influences the WRV, even after re-wetting. When the water surface disappears during the filtering procedure, the dry matter content of the test pad is usually about 8 %.

If alternative 1 is used, remove the pad from the wire and place it in a test-pad former.

Place the complete test-pad-holding unit with the test pad in a centrifuge bucket. Centrifuge at a speed that gives a centrifugal force of  $(3000 \pm 50)$  g at the bottom of the pad for  $15 \text{ min} \pm 30 \text{ s}$ . This period shall include neither acceleration nor retardation. The temperature in the centrifuge shall be kept at  $(23 \pm 3)$  °C.

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 and dry to constant mass (weight) at a temperature of  $(105 \pm 2)$  °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.

*Warning* – If the WRV determination is carried out the day after the stock dilution is prepared, 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 as the dilution is prepared.

## 9 Calculation

Calculate the Water Retention Value, in grams per gram, according to the expression:

$$WRV = \frac{m_1}{m_2} - 1 \quad [1]$$

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 with two decimals.

## 10 Precision

Three chemical pulps were analysed in an interlaboratory study comprising 7 laboratories.

### 10.1 Repeatability

Each beaten pulp was tested five times in each laboratory. The average coefficient of variation between replicates was 1,03 %.

### 10.2 Reproducibility

The results from the 7 laboratories had an average coefficient of variation of 3,2 %. The results were as follows.

Pulp	SR	Alternative 1		Alternative 2	
		WRV, mean g/g	CV, % between labs	WRV, mean g/g	CV, % between labs
Unbl softwood sulphate	46	1,77	5,3	1,84	3,1
Bl softwood sulphate	16	1,23	2,4	1,29	2,9
Bl softwood sulphate	25	1,48	2,5	1,53	2,9

## 11 Report

The test report shall include reference to this SCAN-test Method and the following particulars:

- date and place of the stock preparation, if this differs from the date and place of testing;
- date and place of testing;
- identification mark of the sample tested;
- procedure used for forming the test pad;
- the results, as specified in clause 9;
- any departure from the standard procedure and any other circumstances that may have affected the results.

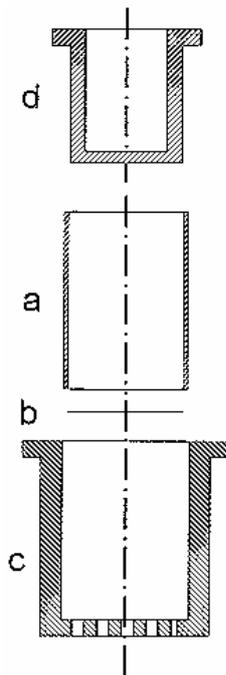
## Annex A Test-pad-holding units

This annex describes the design of two different test-pad-holding units.

**A.1** The test-pad-holding unit in *Figure 1* has four parts:

(a) is the test-pad former with its wire (b). The wire is not soldered to the tube but is fixed in position by pushing the tube (a) into the vessel (c), which has a perforated bottom. The part (d) fits into the tube (a) and forms a cover. It has a small hole to allow air to pass. All parts are machined to fit snugly to each other. During centrifugation the unit hangs on the rim of the centrifuge bucket, leaving enough space underneath to accommodate drained water.

The parts (a), (b) and (c) are assembled before filtration. The lid (d) is placed in position after filtration and before centrifugation.

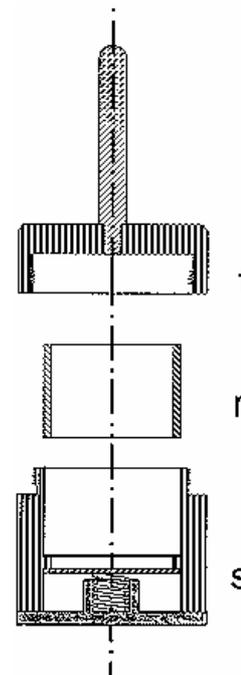


*Figure 1. Test-pad-holding unit, to hang on the rim of the centrifuge bucket.*

**A.2** The test-pad former (r) of the test-pad-holding unit in *Figure 2* has its wire-screen soldered to the tube. The former fits into the vessel (s) where it rests on a flange. A circular metal plate is pressed towards the flange from below by means of a spring. The plate acts as a non-return valve, which opens only when the centrifuge runs at full speed, thus preventing any rewetting of the pad when the centrifuge is stopped. The lid (t) fits onto the vessel (s) and has a handle to facilitate its removal.

During centrifugation, the pad holding unit rests on the bottom of the centrifuge bucket. If this has a semi-spherical bottom, a semi-spherical metal block is used as a support for the former. The main function of the metal block is to create a space under the test-pad-forming unit for the removed water.

Only the test-pad-former (r) is used in the filtration step.



*Figure 2. Test-pad-holding unit, to rest on the bottom of the centrifuge bucket.*

## Annex B

### The influence of test-pad-forming procedure on the results

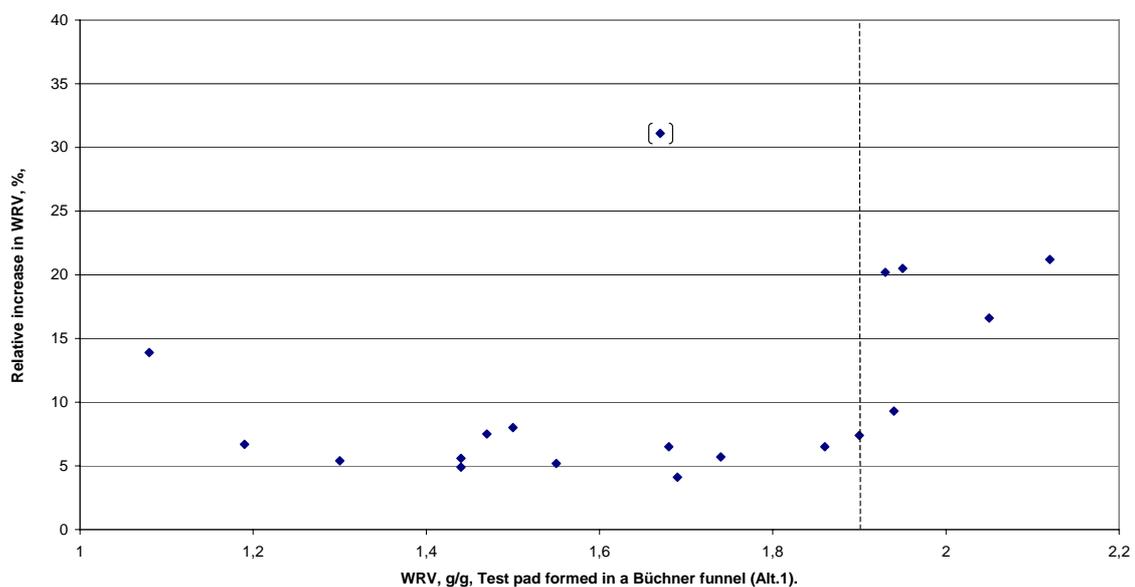
The Water Retention Value of three different pulps, bleached hardwood, bleached softwood and unbleached softwood pulps, have been measured for both unbeaten and laboratory-beaten pulp. Results where the test pad was formed in a test-pad former (Alternative 2) were compared to those where the test pad was formed in a Büchner funnel (Alternative 1).

The results showed that when the test pad was formed in a test-pad former, the WRV results were between 5 % and 10 % higher than the results obtained when the test pad was formed in a Büchner funnel. This applies to Water Retention Values in the interval 0,8 g/g to 1,9 g/g, see *Figure 3*.

*Warning* – The results also indicate that for samples with a high WRV (>1,9 g/g), the difference between the two procedures is much larger.

Neither of the two alternative procedures is considered to be more correct than the other.

*Figure 3. The increase in WRV, in %, when the test pad is formed in a test pad former (Alt. 2), compared with the results obtained when the test pad is formed in a Büchner funnel (Alt. 1).*



**SCAN-test Methods are issued and recommended by KCL, PFI and STFI-Packforsk for the pulp, paper and board industries in Finland, Norway and Sweden.  
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