International Standard 302

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Pulps — Determination of Kappa number

Pâtes — Détermination de l'indice Kappa

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 302 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and was circulated to the member bodies in August 1979.

It has been approved by the member bodies of the following countries :

Australia	France	Norway
Austria	Germany, F. R.	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Canada	Italy	Spain
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

This International Standard cancels and replaces ISO Recommendation R 302-1963, of which it constitutes a technical revision.

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Pulps — Determination of Kappa number

1 Scope and field of application

This International Standard specifies a method for the determination of the Kappa number of pulp. The Kappa number is an indication of the lignin content (hardness) or bleachability of pulp.

It should be noted that there is no general and unambiguous relationship between the Kappa number and the lignin content of a pulp. The relationship varies according to the wood species and delignification procedure. If the Kappa number is to be used to derive an index of pulp lignin content, specific relationships will have to be developed for each pulp type. Kraft pulp values above 125 and sulphite pulp values above 100 should be interpreted with caution.

This method is applicable to all kinds of unbleached pulp obtained in yields under approximately 60 % (mass/mass). The degree of delignification of pulps produced at higher yields should be determined by methods which do not involve permanganate oxidation, such as ISO 3260.

The method is also applicable for determinations on semi-bleached pulps. Using the maximum 10 g test specimen, the practical lower limit of this procedure is a Kappa number of 5.

Variations of the standard procedure which may be used for process control work are given in the annex.

2 References

ISO 638, *Pulps — Determination of dry matter content.*

ISO 3260, *Pulps — Determination of chlorine consumption (degree of delignification).*

3 Definition

For the purpose of this International Standard, the following definition applies.

Kappa number of pulp : The number of millilitres of 0,02 mol/l potassium permanganate solution consumed under the specified conditions by 1 g of pulp (calculated on an oven-dry basis). The results are corrected to a value corresponding to that obtained when 50 % (mass/mass) of the permanganate is consumed in the test.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Sulphuric acid, 2,0 mol/l solution, containing 196,0 g of sulphuric acid (H_2SO_4, ϱ = 1,84 g/ml) per litre.

4.2 Potassium iodide, 1 mol/l solution, containing 166,0 g of potassium iodide (KI) per litre.

4.3 Potassium permanganate, standard volumetric solution, 0,02 ± 0,001 mol/l, containing 3,161 g of potassium permanganate ($KMnO_4$) per litre.

4.4 Sodium thiosulphate, about 0,2 mol/l solution, the concentration of which is known with an accuracy of ± 0,000 5 mol/l, 0,2 mol/l corresponding to 49,64 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) per litre.

4.5 Starch indicator, 2 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Agitator, of propeller type, made of glass or other non-corrosive material (a plastics- or glass-covered magnetic stirrer may be used instead).

5.2 Wet-disintegration apparatus, high-speed mixer, capable of disintegrating the pulp completely with minimum damage to the fibres.

5.3 Constant-temperature bath, capable of maintaining a temperature of 25,0 ± 0,2 °C in the reaction vessel.

5.4 Timing device, capable of measuring 10 min to the nearest 1 s.

6 Preparation of sample

Tear or cut 3 to 10 g of a representative sample[1] of the air-dry pulp into small pieces.

Prior to weighing the test specimen, condition the sample for not less than 20 min in the atmosphere near the balance.

1) Sampling of pulps will form the subject of a future International Standard.

7 Procedure

Weigh, to the nearest 0,001 g, that amount of pulp which will consume approximately 50 % (mass/mass) of the potassium permanganate solution (4.3). The permanganate consumption shall be between 30 and 70 % (mass/mass). At the same time, weigh out a separate test specimen for dry matter determination in accordance with ISO 638.

Disintegrate the test specimen in not more than 500 ml of distilled water until free from fibre clots and from large fibre bundles. Avoid methods of disintegration which involve extensive cutting of the fibres. Transfer the disintegrated test specimen to a 1 500 ml reaction beaker and rinse out the disintegrator (5.2) with sufficient water to bring the total volume to 790 ml.

Place the beaker in the constant-temperature bath (5.3), adjusted so that the reaction temperature stays at 25,0 ± 0,2 °C during the entire reaction. Adjust the agitator (5.1) to obtain a vortex approximately 25 mm deep in the solution.

With a pipette, add 100,0 ± 0,1 ml of the potassium permanganate solution (4.3) and 100 ml of the sulphuric acid (4.1) to a 250 ml beaker. Bring this mixture to 25 °C, add it quickly to the disintegrated test specimen and simultaneously start the timing device (5.4). Rinse out the 250 ml beaker using at least 10 ml of distilled water, and add the washings to the reaction mixture. The total volume shall be 1 000 ml. At the end of exactly 10,0 min, terminate the reaction by adding 20 ml of the potassium iodide solution (4.2) from a graduated cylinder.

Immediately after mixing, but without filtering out the fibres, titrate the free iodine with the sodium thiosulphate solution (4.4). Add a few drops of the starch indicator solution (4.5) toward the end of the titration. (See the note.)

NOTE — Iodine volatilization has been found to be an important variable in the determination of Kappa number. The time between the addition of potassium iodide solution to terminate the reaction and the completion of the subsequent titration should be as short as possible, particularly when titrating the blank.

Carry out a blank test, using exactly the same procedure as above, but omitting the pulp. The potassium iodide solution may be added immediately after the potassium permanganate and sulphuric acid addition.

Make two determinations, the results of which shall, for Kappa number values above 20, fall within ± 1,0 % of the mean Kappa number value. If the difference between duplicate determinations is greater than 2 %, include a third determination in the calculation of the mean Kappa number value. (See 8.1.)

8 Calculation and expression of results

8.1 Calculation

The Kappa number X expressed as a numerical value only, is given by the formulae

$$V_1 = \frac{(V_2 - V_3)\, c}{0{,}02 \times 5}$$

$$X = \frac{V_1\, d}{m}$$

where

V_1 is the volume of the potassium permanganate solution (4.3) consumed in the determination, in millilitres;

V_2 is the volume of the sodium thiosulphate solution (4.4) consumed in the blank test, in millilitres;

V_3 is the volume of the sodium thiosulphate solution (4.4) consumed in the determination, in millilitres;

c is the concentration of the sodium thiosulphate solution (4.4), expressed in moles per litre;

d is the correction factor to 50 % (mass/mass) permanganate consumption; d is dependent on the value of V_1 (see the table);

m is the oven-dry mass of the test specimen, in grams.

8.2 Expression of results

Report the Kappa number of the pulp as the mean of two determinations to the following precision :

Kappa number ⩽ 50 : to the nearest 0,1;

50 < Kappa number ⩽ 100 : to the nearest 0,5;

Kappa number > 100 : without decimals.

Table — Correction factor d, expressed as a function of V_1

V_1 ml	d									
	0	1	2	3	4	5	6	7	8	9
30	0,958	0,960	0,962	0,964	0,966	0,968	0,970	0,973	0,975	0,977
40	0,979	0,981	0,983	0,985	0,987	0,989	0,991	0,994	0,996	0,998
50	1,000	1,002	1,004	1,006	1,009	1,011	1,013	1,015	1,017	1,019
60	1,022	1,024	1,026	1,028	1,030	1,033	1,035	1,037	1,039	1,042
70	1,044									

NOTE — The correction factor d is based on an experimental study which gave the following formula :

$$\log_{10} X = \log_{10} \frac{V_1}{m} + 0{,}000\,93\,(V_1 - 50)$$

8.3 Example of calculation

Air-dry mass of the test specimen 2,200 g

Dry matter content of the sample 91,5 % (mass/mass)

Oven-dry mass of the test specimen $\frac{91,5}{100} \times 2,200\ g = 2,013\ g$

Volume of the sodium thiosulphate solution (4.4) consumed in the blank test, V_2 52,4 ml

Volume of the sodium thiosulphate solution (4.4) consumed in the determination, V_3 21,0 ml

Concentration of the sodium thiosulphate solution (4.4), c 0,191 0 mol/l

Volume of the potassium permanganate solution (4.3) consumed in the test, V_1 $\frac{(52,4 - 21,0) \times 0,191\,0}{0,1} = 60,0$ ml

Correction factor, d 1,022

Kappa number, X $\frac{60,0 \times 1,022}{2,013} = 30,5$

9 Test report

The test report shall include the following particulars :

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

b) reference to this International Standard;

c) the number of determinations, where this is greater than 2;

d) the variation of standard procedure, if applied;

e) the results, expressed as a numerical value only;

f) any unusual features observed during the course of the test;

g) any operation not specified in this International Standard, or in the International Standards to which reference is made, or regarded as optional, which might have affected the results.

Annex

Variations of the standard procedure

(This annex forms part of the standard.)

The variations of the standard procedure given in this annex shall be used only for process control purposes.

The results obtained using these variations should be similar to those obtained by the standard method, but cannot be considered as complying with the standard procedure.

The fact that the variations have been used shall be stated in the test report.

A.1 Preparation of sample

A.1.1 Screened slush pulps

Obtain a representative sample of the pulp, and make a 3 to 10 g air-dry pad by filtering on a Buchner funnel, avoiding any loss of fibres or fines. Air-dry or oven-dry the pad at a temperature not exceeding 105 °C, and tear it into small pieces.

A.1.2 Unscreened pulp which is normally screened before bleaching or other processing

Remove the shives and knots from the sample by screening. The method of screening shall be stated in the test report, and shall be chosen to give results similar to those obtained by the industrial screening of the pulp. Complete the preparation of the screened pulps as specified in A.1.1.

A.1.3 Unscreened pulp which is not normally screened before further processing

Wash the pulp thoroughly before completing the preparation of the sample as specified for screened pulp in A.1.1.

A.2 Variations of procedure

The procedure should be as specified in clause 7, except that the following variations are permitted :

A.2.1 Use of smaller quantities

A suggested control method for chemical pulps uses 50 ml of the potassium permanganate solution (4.3), 50 ml of the sulphuric acid (4.1), 400 ml of distilled water and the appropriate amount of pulp. In this case, when only half the volumes are used, the permanganate consumption V_1 in the table (8.1) should be changed to $2V_1$. Thus, if V_1 = 25 ml, then $2V_1$ = 50 ml, and the correction factor d is 1,000. The method follows the standard procedure in all other respects.

A.2.2 Disintegration in the reaction beaker

Easily disintegrated pulps can be slurried directly in the reaction beaker. For the variant using smaller quantities (see A.2.1), a 1-litre beaker of the type used in kitchen mixers is recommended as a combined disintegrator and reaction beaker, provided that disintegration is not continued during the reaction.

A.2.3 Correction for reaction temperature

When a constant-temperature bath is not available, measure the temperature of the pulp mixture after the reaction has been taking place for 5 min, and assume this to be the average reaction temperature throughout the test. If this temperature is not higher than 30 °C or lower than 20 °C, correct the Kappa number X as follows :

$$X = \frac{V_1 d}{m} [1 + 0{,}013 (25 - t)]$$

where t is the average reaction temperature, in degrees Celsius.

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