INTERNATIONAL STANDARD

ISO 302

Second edition
2004-07-01

Pulps — Determination of Kappa number

Pâtes — Détermination de l'indice Kappa

Reference number
ISO 302:2004(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword ... iv
1 Scope ... 1
2 Normative references ... 1
3 Terms and definitions ... 1
4 Principle ... 2
5 Reagents and materials ... 2
6 Apparatus and equipment ... 3
7 Sampling and preparation of sample ... 3
7.1 Sampling ... 3
7.2 Sample preparation ... 3
8 Procedure ... 3
8.1 General ... 3
8.2 Blank ... 4
8.3 Determination ... 4
9 Calculations ... 6
9.1 Kappa number 5 to 100 ... 6
9.2 Kappa number 1 to 5 ... 7
9.3 Expression of results ... 8
9.4 Example of calculation ... 8
10 Precision ... 8
10.1 Reference pulp ... 8
10.2 Repeatability ... 8
10.3 Reproducibility ... 9
11 Test report ... 9
Bibliography ... 10

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

This second edition cancels and replaces the first edition (ISO 302:1981), which has been technically revised.

Pulps — Determination of Kappa number

1 Scope

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 or bleachability of pulp.

This International Standard is applicable to all kinds of chemical pulps and semi-chemical pulps within the Kappa number range 1 to 100. For pulps with a Kappa number exceeding 100, use the chlorine-consumption procedure (ISO 3260) to describe the degree of delignification.

To achieve the greatest precision and accuracy, the sample size should be adjusted so that the consumption of permanganate falls between 20 % and 60 % of the amount added.

NOTE There is no general and unambiguous relationship between the Kappa number and the lignin content of pulp. The relationship varies according to the wood species and delignification procedure. All compounds oxidized by $KMnO_4$, not only lignin, will increase the consumption of $KMnO_4$, and thereby increase the Kappa number (see [7]). 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.

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 7213, *Pulps — Sampling for testing*

3 Terms and definitions

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

3.1
oxidation capacity
relative amount of permanganate oxidized (expressed as MnO_2) of the total oxidation capacity

3.2
total oxidation capacity
oxidation capacity (permanganate consumption) when all permanganate is oxidized into Mn^{2+}

3.3.
Kappa number of pulp
number of millilitres of 0,02 mol/l potassium permanganate solution consumed under the specified conditions by one gram of pulp (calculated on an oven-dry basis)

NOTE The results are corrected to a value corresponding to that obtained when 50 % of the total oxidation capacity of the permanganate is consumed in the test at a temperature of 25 °C.

4 Principle

Disintegrated pulp is allowed to react with a specified amount of potassium permanganate solution for a given time. The amount of pulp is chosen so that about 50 % of the total oxidation capacity of the permanganate is left unconsumed at the end of the reaction time.

The main reactions are as follows:

1) Residual lignin + other oxidable compounds + MnO_4^- + $4H^+$ → oxidized lignin + other oxidized compounds + excess MnO_4^- + MnO_2 + $2H_2O$

2) $2MnO_4^-$ + $10I^-$ + $16H^+$ → $2Mn^{2+}$ + $5I_2$ + $8H_2O$

3) MnO_2 + $4H^+$ + $2I^-$ → Mn^{2+} + $2H_2O$ + I_2

4) $2S_2O_3^{2-}$ + I_2 → $S_4O_6^{2-}$ + $2I^-$

NOTE By theoretical calculation and experimental observation, a consumption of 60 % (mass/mass) is actually the endpoint of the consumption for the permanganate ions, at which point the ions have been reduced to MnO_2. Further oxidation, performed by means of MnO_2 should be considered as "out of range". By adding potassium iodide solution, the reaction is terminated and the free iodine is titrated with sodium thiosulfate solution. The value so obtained is corrected to 50 % consumption of the total oxidation capacity of permanganate.

5 Reagents and materials

Use only chemicals of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sulfuric acid, $c(H_2SO_4)$ = 2,0 mol/l.

Add with caution 112 ml of sulfuric acid, H_2SO_4, of density 1,84 g/ml, to about 600 ml of water. Allow to cool and dilute to 1 litre with water.

5.2 Potassium iodide, c(KI) = 1 mol/l.

Dissolve 166 g of potassium iodide, KI, in a 1 000 ml volumetric flask and fill up to the mark with water.

5.3 Potassium permanganate, $c(KMnO_4)$ = (0,020 ± 0,001) mol/l.

Dissolve 3,161 g of potassium permanganate, $KMnO_4$, in a 1 000 ml volumetric flask and fill up to the mark with water.

NOTE Fresh solution is stable for at least 6 months if stored in a dark bottle.

5.4 Sodium thiosulfate, $c(Na_2S_2O_3)$ = (0,200 0 ± 0,000 5) mol/l.

Dissolve 49,65 g of sodium thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$, in a 1 000 ml volumetric flask and fill up to the mark with water.

5.5 Starch indicator, 2 g/l solution.

NOTE Commercially available standard solutions may be used.

6 Apparatus and equipment

Ordinary laboratory equipment and the following.

6.1 Agitator, of the propeller type, made of glass or other noncorrosive material (a plastic- or glass-covered magnetic stirrer may be used instead).

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

6.3 Water bath, capable of maintaining a temperature of (25,0 ± 0,2) °C in the reaction vessel (see 8.3 regarding temperature correction).

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

NOTE Automatic Kappa number analysers can be used if they follow this International Standard and give the same results.

7 Sampling and preparation of sample

7.1 Sampling

If the test is being made 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.

As the presence of small amounts of spent cooking liquor affects the Kappa number, ensure that the sample is well washed.

7.2 Sample preparation

Prepare the test material according to one of the following procedures.

7.2.1 Air-dry pulp. Tear or cut the pulp into small pieces.

7.2.2 Screened slush pulp. Dewater the pulp sample by filtering on a Büchner funnel or by centrifuging, avoiding any loss of fibres or fines. Air-dry the pulp sample, or dry it at a temperature not exceeding 105 °C, and tear it into small pieces.

7.2.3 Unscreened pulp. If the sample is taken from unscreened pulp, which is normally screened before bleaching or other processing, remove the shives and knots from the sample by screening. Choose a procedure that gives results similar to those obtained by industrial screening. State the method of screening in the test report. Continue the sample preparation as described in 7.2.2.

NOTE If the pulp sample contains a considerable amount of shives, the screening procedure may give rise to incorrect results. A more reliable value may be obtained by defibrating the pulp sample before the determination. State the method of defibration in the test report.

8 Procedure

8.1 General

This International Standard includes two different procedures. One is used in the Kappa number range 5 to 100 and the other in the Kappa number range 1 to 5.

The main difference between the procedures is the added amounts of pulp and of $KMnO_4$, and the calculation. Due to stirring problems when determining Kappa numbers below 5, decrease the amounts of pulp and permanganate.

Use 8.2 and 8.3 in both procedures. The separate procedures are described in 8.3.1 and 8.3.2.

Run the determination in duplicate.

NOTE Experimental work in Nordic countries has shown that the Kappa number determination according to the procedure for Kappa number 5 to 100 gives results equivalent to those obtained by the procedure for Kappa number 1 to 5, within the Kappa number range from 4 to 6.

8.2 Blank

Carry out a blank determination using exactly the procedure described in 8.3, but without the pulp. Read off the volume, V_1, to the nearest 0,1 ml, of the sodium thiosulfate (5.4) consumed at the inflection point. The consumption of sodium thiosulfate solution may vary by at most ± 1 % from its theoretical value (25,0 ml).

Divide the blank value of the Kappa number determination in the range 5 to 100 by two, for use in the Kappa number determination in the 1 to 5 range.

8.3 Determination

Condition the test specimens for at least 20 min, or until constant weight has been reached, in an atmosphere near the balance prior to weighing the samples for determination of Kappa number and dry matter content.

Weigh, to the nearest 0,001 g, the amount of pulp which will consume approximately 50 % of the potassium permanganate (5.3). Examples of suitable amounts of pulps are given in Tables 1 and 2. Ensure that the consumption of permanganate is between 20 % and 60 % (mass/mass) of the amount added (see [10]).

At the same time, weigh a separate test specimen for determination of the dry matter content in accordance with ISO 638, or any other method for determination of the dry matter content giving a similar result.

Table 1 — Suitable amounts of oven-dry pulp in the Kappa number range 5 to 100

Kappa number	Amount of sample, g
5	4,5
6	4,0
8	3,0
10	2,5
15	1,5
20	1,2
25	1,0
30	0,9
35 to 45	0,6
50 to 55	0,5
60 to 70	0,4
80 to 90	0,3
100	0,25

Table 2 — Suitable amounts of oven-dry pulp in the Kappa number range 1 to 5

Kappa number	Amount of sample, g
1	5,5
2 to 3	4,0
4	3,0
5	2,5

In order to avoid stirring problems when the Kappa number is low (i.e. 5 in the Kappa number range 5 to 100, and 1 to 2 in the Kappa number range 1 to 5), the amount of sample must be smaller than the amount corresponding to approximately 50 % of the total oxidation capacity of permanganate. However, the amount of sample should still correspond to at least 20 % of the total oxidation capacity of permanganate.

Disintegrate the test specimen in 300 ml of distilled water until it is free from fibre clots and from large fibre bundles. Avoid methods of disintegration which involve extensive cutting of the fibres. Rinse the disintegrator with approximately 90 ml of distilled water. If a combined disintegration and reaction beaker is used, perform the disintegration in 390 ml of distilled water.

Place the beaker in a water bath (6.3) adjusted to maintain a reaction temperature of (25,0 ± 0,2) °C during the entire reaction. As an alternative to using a water bath, the temperature in the specimen solution during the reaction time can be registered. However, the temperature of the specimen solution must be between 20 °C and 30 °C. Make a temperature correction if the temperature is not (25,0 ± 0,2) °C. Read the temperature after a reaction time of 5 min and take this as the mean reaction temperature.

Adjust the agitator (6.1) to produce a vortex approximately 25 mm deep in the reaction mixture. It is very important that the stirring is adequate.

8.3.1 Kappa number range 5 to 100

Pipette (50 ± 0,1) ml of the potassium permanganate solution (5.3) and 50 ml of the sulfuric acid (5.1) into a beaker. Bring this mixture to 25 °C, quickly add the mixture to the disintegrated specimen and simultaneously start the timing device (6.4). Rinse the beaker with approximately 10 ml of distilled water, and add the washings to the reaction mixture. The total volume shall be 500 ml. At the end of 10,0 min ± 15 s, terminate the reaction by adding exactly 10 ml of the potassium iodide solution (5.2).

Immediately after mixing, but without filtering out the fibres, titrate the free iodine with the sodium thiosulfate solution (5.4). Add a few drops of the starch indicator solution (5.5) toward the end of the titration (see the second to last paragraph of 8.3.2). Read off the volume, V_2, to the nearest 0,1 ml, of the sodium thiosulfate (5.4) consumed at the inflection point.

8.3.2 Kappa number range 1 to 5

Pipette (25,0 ± 0,1) ml of the potassium permanganate solution (5.3) and 50 ml of the sulfuric acid (5.1) into a beaker. Bring the mixture to 25 °C, quickly add the mixture to the disintegrated specimen and simultaneously start the timing device (6.4). Rinse the beaker with approximately 35 ml of distilled water, and add the washings to the reaction mixture. The total volume shall be 500 ml. At the end of 10,0 min ± 15 s, terminate the reaction by adding exactly 10 ml of the potassium iodide solution (5.2).

Immediately after mixing, but without filtering out the fibres, titrate the free iodine with the sodium thiosulfate solution (5.4). Add a few drops of the starch indicator solution (5.5) toward the end of the titration (see the second last paragraph of this subclause). Read off the volume, V_2, to the nearest 0,1 ml, of the sodium thiosulfate (5.4) consumed at the inflection point.

Iodine volatilization has been found to be an important variable in the determination of the 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.