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Vlaknine - Določanje števila Kappa

Pulps -- Determination of Kappa number

Pâtes -- Détermination de l'indice Kappa

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Pulps — Determination of Kappa number

Pâtes — Détermination de l'indice Kappa

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 302:2004), 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 has to 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.[8] 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

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 potassium permanganate reduced (expressed as MnO_2) of the total oxidation capacity

3.2
total oxidation capacity
oxidation capacity (permanganate consumption) when all permanganate is reduced 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 1 to entry: 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:

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

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

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

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

NOTE By theoretical calculation and experimental observation, a consumption of 60 % (mass/mass) is actually the end point 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 8.3.2 and 8.3.3.

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.

