INTERNATIONAL STANDARD

ISO 699

Third edition
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Pulps — Determination of alkali resistance

Pâtes — Détermination de la résistance aux solutions d'hydroxyde de sodium

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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 699:1982), of which it constitutes a minor revision.

Introduction

The object of both this International Standard and ISO 692, *Pulps — Determination of alkali solubility,* is to permit the study of the behaviour of pulps in the presence of alkali solutions, but their fields of application are different. While this International Standard describes the gravimetric determination of the alkali insoluble constituents of the pulp and applies to all categories of pulps, ISO 692 describes the volumetric determination of the alkali soluble constituents of the pulp and is applied preferably to the control of bleached pulps.

Pulps — Determination of alkali resistance

WARNING — The method specified in this International Standard involves the use of hazardous chemicals. Appropriate precautions are to be taken to ensure the proper use and disposal of these chemicals.

1 Scope

This International Standard specifies a method for the determination of the alkali-insoluble fraction of pulps using sodium hydroxide solution of fixed concentration. The sodium hydroxide concentrations most frequently used are 18, 10, and 5 % (*m*/*m*).

The method is applicable to all kinds of pulp.

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 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

3 Terms and definitions

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

3.1
***R*-value**
alkali resistance; the insoluble fraction expressed as a percentage by mass of the oven-dry pulp

3.2
R_{18} , R_{10} , R_5, or R_c
R-values in which the indices 18, 10, 5, or *c* refer to the chosen concentration in grams of sodium hydroxide per 100 g of solution

4 Principle

Defibreing of the pulp under specified conditions in sodium hydroxide solution of chosen concentration.

Filtering of the insoluble fraction, washing with sodium hydroxide solution of the same concentration, and temperature as that used for defibreing and acidification, washing, drying, and weighing.

5 Reagents

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

5.1 Sodium hydroxide, solution of known concentration containing less than 1 g of sodium carbonate per litre (see the notes), for example:

— (5,39 ± 0,03) mol/l solution, containing (18,0 ± 0,1) g of sodium hydroxide per 100 g of solution (ρ_{20} = 1,197 2 g/ml), equivalent to (215,5 ± 1,0) g of sodium hydroxide per litre;

- (2,77 ± 0,03) mol/l solution, containing (10,0 ± 0,1) g of sodium hydroxide per 100 g of solution (ρ_{20} = 1,108 9 g/ml), equivalent to (110,9 ± 1,0) g of sodium hydroxide per litre;
- (1,31 ± 0,03) mol/l solution, containing (5,0 ± 0,1) g of sodium hydroxide per 100 g of solution (ρ_{20} = 1,053 8 g/ml), equivalent to (52,7 ± 1,0) g of sodium hydroxide per litre.

NOTE 1 The sodium hydroxide solution can be conveniently prepared as follows: dissolve a quantity of solid sodium hydroxide in an equal mass of water and allow the suspended sodium carbonate to settle. Decant the supernatant liquid and dilute with carbon dioxide-free water to the appropriate concentration. Check by titration with standard acid solution.

NOTE 2 Although sodium hydroxide solution generally possesses the maximum dissolving power at a concentration of about 10 % *(m/m)*, certain pulps show maximum solubility at some lower or higher alkali concentration. If the *R*-value of an unknown pulp or of a new type of pulp is to be determined with the sodium hydroxide solution of maximum dissolving power for this pulp, it is necessary to establish a solubility diagram using several different concentrations in order to determine the sodium hydroxide concentration of maximum dissolving power.

5.2 Acetic acid, 1,7 mol/l solution, corresponding to 100 ml of CH_3COOH (ρ_{20} = 1,055 to 1,058 g/ml) per litre.

6 Apparatus

6.1 Beaker, of capacity 250 ml, flat-bottomed, made of alkali-resistant material.

6.2 Stirring rod, of diameter 15 mm, with a flat end, made of a non-brittle alkali-resistant material, preferably hard plastics.

6.3 Filtering device, of capacity 80 to 100 ml, internal diameter about 30 mm, with a bottom made of sintered glass, of porosity grade P 250 (porosity 0) in accordance with ISO 4793.

6.4 Weighing bottle, with a lid.

6.5 Constant temperature bath, capable of maintaining a temperature of (20 ± 0,2) °C.

7 Preparation of the test sample

If the pulp is in slush form, remove the water by suction taking precautions to avoid the loss of fine fibres, press between blotters, and dry at a maximum temperature of 60 °C.

If the pulp is in the form of wet sheets or rolls, dry the sample at a maximum temperature of 60 °C.

Tear the sample into pieces of size approximately 5 mm x 5 mm. If the pulp is difficult to defibre, split the sample by means of tweezers (see Note 1).

Check that the pulp contains not more than 0,1 % ash (see Note 2). Before weighing, condition, the sample for not less than 20 min in the atmosphere near the balance.

NOTE 1 Dry disintegration, for example with a Wiley-mill, or wet disintegration, for example with a high-speed stirrer, are not permitted.

NOTE 2 If the pulp to be tested contains more than 0,1 % ash, determine the ash content of the alkali-insoluble fraction. Calculate the *R*-value on the basis of the ash-free pulp and the ash-free insoluble fraction.

8 Procedure

8.1 Test portion

Weigh, to the nearest 1 mg, about 2,5 g of the test sample. Then immediately weigh two separate test portions for the determination of the dry matter content in accordance with ISO 638.

8.2 Determination

Transfer the test portion to the beaker (6.1), add 25 ml of the sodium hydroxide solution (5.1) adjusted to (20 ± 0,2) °C (see 5.1, Note 1 and Note 2), place the beaker in the constant temperature bath (6.5), and allow the pulp to swell for 3 min.

Thoroughly defibre the pulp by stirring and macerating with the stirring rod (6.2) for at least 3 min, macerating at a rate of two strokes per second. Add another 25 ml of the sodium hydroxide solution at 20 °C, stir until the suspension is uniform, and dilute by adding 100 ml of the sodium hydroxide solution at 20 °C. Cover the beaker with a watch-glass and leave in the constant temperature bath.

60 minutes after the first addition of the sodium hydroxide solution, stir the fibre suspension again and transfer it to the filtering device (6.3) fitted on a dry suction flask, adjusted to a temperature of (20 ± 0,2) °C in the constant temperature bath.

Apply suction only as long as the fibre mat is still covered with liquid in order that no air is sucked through the mat. Use the filtrate for rinsing the beaker and filter again through the slightly pressed fibre mat in order to collect all fibres. Finally, apply full suction briefly. The time for filtering and washing shall not exceed 20 min.

Compact the fibre mat, especially at the edges, cover with the acetic acid (5.2) and allow 200 ml to pass through slowly without suction. Drain completely and wash with hot water until the filtrate is free from acid.

Cover the filtering device with the hand during the last washing in order that a vacuum is formed above the fibre mat. Then quickly release the vacuum in the suction flask in order to lift the fibre mat. Transfer the fibre mat together with any remaining fibres adhering to the filtering device by means of stainless steel tweezers to the weighing bottle (6.4).

Place the open weighing bottle together with its lid in a drying oven and dry to constant mass at a temperature of (105 ± 2) °C (normally for 6 h). Allow the closed weighing bottle to cool in a desiccator and determine the mass of the alkali-insoluble fraction to the nearest 1 mg, after briefly raising the lid to allow equalization of pressure.

Carry out at least two parallel determinations on each test sample.

NOTE 1 In certain cases, for example, straw pulps, it is advisable to add initially only 15 ml or 20 ml of the sodium hydroxide solution to the pulp in order to facilitate defibreing. The volume of alkali for the second addition has then to be increased to 35 ml or 30 ml respectively.

The solubility in 18 % (*m*/*m*) sodium hydroxide solution is not affected by variations of a few degrees in temperature. At this concentration, the temperature may be kept at (20 ± 2) °C.

The solubility in sodium hydroxide solution of lower concentration (for example, 10 % (*m*/*m*)) is much more dependent on temperature. At this lower concentration, the temperature should be kept at (20 ± 0,2) °C.