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ISO RECOMMENDATION

R 699

PULPS

DETERMINATION OF ALKALI RESISTANCE

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BRIEF HISTORY

The ISO Recommendation R 699, *Pulps – Determination of alkali resistance,* was drawn up by Technical Committee ISO/TC 6, *Paper, board and pulps,* the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1962 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In January 1966, this Draft ISO Recommendation (No. 898) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Iran	South Africa, Rep. of
Australia	Israel	Spain
Austria	Italy	Sweden
Belgium	Japan	Switzerland
Brazil	Korea, Rep. of	Turkey
Bulgaria	Mexico	U.A.R.
Chile	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Finland	Norway	U.S.S.R.
France	Poland	Yugoslavia
Germany	Portugal	
India	Romania	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 699 March 1968

PULPS

DETERMINATION OF ALKALI RESISTANCE

FOREWORD

The scope of both this ISO Recommendation and of ISO Recommendation R 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 ISO Recommendation describes the gravimetric determination of the alkali insoluble constituents and applies to all categories of pulp, ISO Recommendation R 692 describes the volumetric determination of the alkali soluble constituents of the pulp and is applicable preferably for the control of bleached pulps.

1. SCOPE

This ISO Recommendation describes a method for the determination of the alkali - insoluble fraction of pulp using sodium hydroxide solution of a fixed concentration. The concentrations of sodium hydroxide most frequently used are 18, 10 and 5 % (m/m).

2. FIELD OF APPLICATION

This method is applicable to all kinds of pulp.

3. PRINCIPLE OF THE METHOD

The pulp is defibred under specified conditions in sodium hydroxide solution of the chosen concentration.

The insoluble fraction is filtered off, washed with sodium hydroxide solution of the same concentration and temperature as that used for the processing itself, and is acidified, washed, dried and weighed.

4. DEFINITION OF TERMS

4.1 *R-value.* Alkali resistance : the insoluble fraction expressed as a percentage of the oven-dry pulp mass.

4.2 R_{18}, R_{10}, R_5 or R_C. The indices 18, 10, 5 or *c* refer to the chosen concentration in grammes of sodium hydroxide per 100 g of solution.

5. REAGENTS

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

- 5.39 M ± 0.03 M, containing 18.0 ± 0.1 g of NaOH per 100 g of solution (ρ_{20} = 1.1972 g/cm^3) equivalent to 215.5 ± 1.0 g of NaOH per litre,
- 2.77 M ± 0.03 M, containing 10.0 ± 0.1 g of NaOH per 100 g of solution (ρ_{20} = 1.1089 g/cm^3) equivalent to 110.9 ± 1.0 g of NaOH per litre,
- 1.31 M ± 0.03 M, containing 5.0 ± 0.1 g of NaOH per 100 g of solution (ρ_{20} = 1.0538 g/cm^3) equivalent to 52.7 ± 1.0 g of NaOH per litre.

NOTES

1. *Preparation of the sodium hydroxide solution.* The sodium hydroxide solution is conveniently prepared as follows :

   Dissolve a quantity of solid sodium hydroxide in an equal mass of distilled water and allow the suspended sodium carbonate to settle. Decant the supernatant liquid and dilute with carbon dioxide-free distilled water to appropriate concentration. Check by titration with standard acid.

2. *Strength of alkali of maximum dissolving power.* Although sodium hydroxide solution generally possesses the maximum dissolving power at a concentration of about 10 %, 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 with several different concentrations in order to find the sodium hydroxide concentration of maximum dissolving power.

5.2 *Acetic acid,* 1.7 M, 100 ml of CH_3COOH (ρ_{20} = 1.055 to 1.058 g/cm^3) per litre.

6. APPARATUS

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

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

6.3 *Filtering device,* capacity 80 to 100 ml, with a perforated bottom, inner diameter about 30 mm, made of alkali-resistant material.

6.4 *Stainless steel gauze,* to fit the filtering device, pore size about 0.3 mm, soldered along the rim.

6.5 *Weighing bottle,* provided with a lid.

6.6 *Constant temperature bath,* capable of maintaining a temperature of 20 ° ± 0.2 °C.

7. PREPARATION OF 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 approximately 5 mm × 5 mm. If the pulp is difficult to defibre, split the sample by means of tweezers (see Note 1 below).

Check that the pulp contains not more than 0.1 % ash (see Note 2 below). Before weighing, condition the sample for not less than 20 minutes in the atmosphere near the balance.

NOTES

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

2. *Pulps, containing more than 0.1 % ash.* 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

Weigh about 2.5 g of the sample to the nearest 1 mg. Then immediately weigh out two separate samples for the determination of dry matter content according to ISO Recommendation R 638, *Pulps – Determination of dry matter content.*

Transfer the weighed sample to a 250 ml beaker, add 25 ml of sodium hydroxide solution adjusted to 20 ° ± 0.2 °C (see Notes 1 and 2 below), place the beaker in the constant temperature bath and allow the pulp to swell for 3 minutes.

Thoroughly defibre the pulp by stirring and macerating with the stirring rod for at least 3 minutes until completely defibred, with a macerating rate of 2 strokes per second. Add another 25 ml of sodium hydroxide solution at 20 °C, stir until the suspension is uniform and dilute finally by adding 100 ml sodium hydroxide solution at 20 °C. Cover the beaker with a watch-glass and leave it in the constant temperature bath.

60 minutes after the first addition of sodium hydroxide solution stir the fibre suspension again and transfer it to the filter funnel, 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.

Then wash the fibre mat with two 25 ml portions of sodium hydroxide solution of the same concentration - at 20 ° ± 0.2 °C - using only slight suction and without allowing air to penetrate into the fibre mat. Finally apply full suction briefly. The time for filtering and washing should not exceed 20 minutes.

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

Cover the funnel 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 or the gauze, by means of stainless steel tweezers to a weighing bottle provided with a lid.

Place the open weighing bottle together with the lid in the drying oven and dry to constant mass at a temperature of 103 ° ± 2 °C (normally for 6 hours). 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 for pressure equalization.

NOTES

1. *Dry pulps, which can be defibred only with difficulty.* In certain cases, for example, straw pulps, it is advisable to add initially only 15 or 20 ml of sodium hydroxide solution to the pulp in order to facilitate the defibering. The second addition of alkali has then to be raised to 35 or 30 ml respectively.

2. *Temperature.* The solubility in 18 % sodium hydroxide solution is not affected by variations of a few degrees in temperature. For this reason the temperature may be kept at 20 ° ± 2 °C.

   The solubility in weaker alkali (for example, 10 %) is much more dependent on temperature. At this lower concentration, the temperature of the mixture should be kept at 20 ° ± 0.2 °C.