INTERNATIONAL STANDARD 699

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Pulps — Determination of alkali resistance

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

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendation; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 6 has reviewed ISO Recommendation R 699 and found it suitable for transformation. International Standard ISO 699 therefore replaces ISO Recommendation R 699-1968.

ISO Recommendation R 699 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 699 into an International Standard :

Canada

Printed in Switzerland

Pulps — Determination of alkali resistance

0 INTRODUCTION

The object of both this International Standard and of 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 and applies to all categories of pulp, ISO 692 describes the volumetric determination of the alkali soluble constituents of the pulp and is applied preferably to the control of bleached pulps.

1 SCOPE

This International Standard specifies 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 REFERENCE

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

4 PRINCIPLE

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

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

5 DEFINITION OF TERMS

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

5.2 R_{18}, R_{10}, R_5 or R_c : The indices 18, 10, 5 or *c* refer to the chosen concentration in grams of sodium hydroxide per 100 g of solution.

6 REAGENTS

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

— 5,39 ± 0,03 N, containing 18,0 ± 0,1 g of NaOH per 100 g of solution (ρ_{20} 1,197 2 g/ml) equivalent to 215,5 ± 1,0 g of NaOH per litre,

— 2,77 ± 0,03 N, containing 10,0 ± 0,1 g of NaOH per 100 g of solution (ρ_{20} 1,108 9 g/ml) equivalent to 110,9 ± 1,0 g of NaOH per litre,

— 1,31 ± 0,03 N, containing 5,0 ± 0,1 g of NaOH per 100 g of solution (ρ_{20} 1,053 8 g/ml) equivalent to 52,7 ± 1,0 g of NaOH per litre.

NOTES

1 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 the appropriate concentration. Check by titration with standard acid.

2 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.

6.2 Acetic acid, 1,7 N, 100 ml of CH_3COOH (ρ_{20} 1,055 to 1,058 g/ml) per litre.

7 APPARATUS

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

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

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

7.4 Stainless steel gauze, to fit the filtering device, pore size about 0,3 mm, soldered along the rim.

7.5 Weighing bottle, provided with a lid.

7.6 Constant temperature bath, capable of maintaining a temperature of 20 ± 0,2 °C.

8 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 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 min in the atmosphere near the balance.

NOTES

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

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.

9 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/R 638.

Transfer the weighed sample to a 250 ml beaker, add 25 ml of the sodium hydroxide solution (6.1) adjusted to 20 ± 0,2 °C (see notes 1 and 2 below), place the beaker in the constant temperature bath (7.6) and allow the pulp to swell for 3 min.

Thoroughly defibre the pulp by stirring and macerating with the stirring rod (7.2) for at least 3 min until completely defibred, with a macerating rate of 2 strokes per second. Add another 25 ml of the sodium hydroxide solution at 20 °C, stir until the suspension is uniform and dilute finally by adding 100 ml of the sodium hydroxide solution at 20 °C. Cover the beaker with a watch-glass and leave it in the constant temperature bath.

60 min after the first addition of the 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 the 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 shall not exceed 20 min.

Compact the fibre mat, especially at the edges and cover with the acetic acid (6.2) 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 the weighing bottle provided with a lid (7.5).

Place the open weighing bottle together with the lid in a drying oven and dry to constant mass at a temperature of 103 ± 0,2 °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 for pressure equalization.

Carry out at least two parallel determinations on each sample.

NOTES

1 In certain cases, for example, straw pulps, it is advisable to add initially only 15 or 20 ml of the 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 The solubility in 18 % 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 a weaker alkali solution (for example, 10 %) is much more dependent on temperature. At this lower concentration, the temperature of the mixture shall be kept at 20 ± 0,2 °C.

10 EXPRESSION OF RESULTS

Calculate the alkali-resistance, R_c, of the pulp by the formula

$$\frac{m_1 \times 100}{m_0}$$

where

m_0 is the mass, in grams, of the original pulp, calculated on an oven-dry basis;

m_1 is the oven-dry mass, in grams, of the alkali-insoluble fraction.

Parallel determinations should agree within 0,3 %.

Report the alkali-resistance as the mean of the parallel determinations to the first decimal place using the symbols R_{18}, R_{10}, etc.

NOTE — For pulps containing less than 0,1 % ash and other non-carbohydrate materials the value $(100 - R_c)$ approaches the value (S_c) found by the method of determination of alkali solubility of pulp, described in ISO 692.

11 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.