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

Second edition — 1982-11-15

UDC 676.014.362 **Ref. No. ISO 699-1982 (E)**

Descriptors : paper pulps, tests, chemical resistance, solubility, sodium hydroxide, alkali resistance.

Price based on 3 pages

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.

International Standard ISO 699 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. 699-1974), which had been 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	USA
Egypt, Arab Rep. of	New Zealand	USSR
Finland	Norway	Yugoslavia
France	Poland	
Germany, F. R.	Portugal	

No member body had expressed disapproval of the document.

Printed in Switzerland

Pulps — Determination of alkali resistance

0 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.

1 Scope and field of application

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 Reference

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

3 Definitions

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.

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

4 Principle

Defibering 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 defibering, 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.

NOTES

The sodium hydroxide solution may 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.

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

Ordinary laboratory apparatus, and

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 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 × 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.

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.

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 notes 1 and 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 2 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.

Sixty 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.

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 defibering. The volume of alkali for the second addition has then to be increased to 35 or 30 ml respectively.

2 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.

9 Expression of results

9.1 Method of calculation and formula

The alkali resistance, R_c, expressed as a percentage by mass, is given by the formula

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

where

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

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

9.2 Precision and expression of results

The results of the parallel determinations should agree to within 0,3 %.

Report the mean alkali resistance to one 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) determined by the method for alkali solubility of pulps, described in ISO 692.

10 Test report

The test report shall include the following particulars :

a) a reference to this International Standard;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

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

This page intentionally left blank

This page intentionally left blank

This page intentionally left blank