SLOVENSKI STANDARD
SIST ISO 5351-1:1996

01-april-1996

Razredčena raztopina celuloze - Določanje mejne viskoznosti - 1. del: Metoda v raztopini bakrovega-etilen-diamina (CED)

Cellulose in dilute solutions -- Determination of limiting viscosity number -- Part 1: Method in cupri-ethylene-diamine (CED) solution

Cellulose en solutions diluées -- Détermination de l'indice de viscosité limite -- Partie 1: Méthode utilisant une solution de cupri-éthylène-diamine (CED)

Ta slovenski standard je istoveten z: **ISO 5351-1:1981**

ICS:

85.040 Vlaknine Pulps

SIST ISO 5351-1:1996 **en**

International Standard 5351/1

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

Cellulose in dilute solutions — Determination of limiting viscosity number — Part 1 : Method in cupri-ethylene-diamine (CED) solution

Cellulose en solutions diluées — Détermination de l'indice de viscosité limite — Partie 1 : Méthode utilisant une solution de cupri-éthylène-diamine (CED)

First edition — 1981-12-01

UDC 676.1 : 532.13 **Ref. No. ISO 5351/1-1981 (E)**

Descriptors : cellulose, tests, determination, viscosity, viscosity index.

Price based on 11 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 5351/1 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and was circulated to the member bodies in January 1980.

It has been approved by the member bodies of the following countries :

Australia	France	Philippines
Austria	Germany, F.R.	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Canada	Italy	Spain
Chile	Kenya	Sweden
China	Korea, Rep. of	Switzerland
Czechoslovakia	New Zealand	Turkey
Finland	Norway	United Kingdom

No member body expressed disapproval of the document.

Printed in Switzerland

Contents

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Cellulose in dilute solutions — Determination of limiting viscosity number — Part 1 : Method in cupri-ethylene-diamine (CED) solution

0 Introduction

The viscosity (or dynamic viscosity), symbol η, of a fluid is defined by the Newtonian equation

$$\tau = \eta \cdot \dot{\gamma}$$

where

τ is the shear stress;

η is the viscosity;

$\dot{\gamma} = \frac{dv}{dz}$ is the velocity gradient (v is the velocity of one plane relative to the other, and z the co-ordinate perpendicular to the two planes).

In non-Newtonian behaviour, normally the case with high-polymer solutions such as cellulose, the ratio of the shear stress to the velocity gradient varies with the shear stress.

The data required for evaluation of the limiting viscosity number of cellulose in dilute solutions (for definitions and symbols, see clause 4) are derived by means of a capillary-tube viscometer. the results of these measurements are seriously affected by the shear rate. The solution to the problem that thereby arises can be sought either by determination of the viscometric properties at so low a concentration of cellulose that the effect of shear rate is small, or by determination at a shear rate closely reproducible in different laboratories. In this document, both alternatives are given, as it has been proved that the results they provide are equal so long as the limiting viscosity number is less than 1 000 ml/g. For values which exceed this, alternative procedure B gives somewhat higher results by reason of the lower shear rate.

In **alternative A**, the concentration c of cellulose is chosen that if multiplied by the limiting viscosity number it gives a product of $[\eta] \cdot c$ = 1,0 to 1,5, corresponding to viscosity ratio $\frac{\eta}{\eta_0}$ equal to 2,3 to 3,4. At this low concentration, the effect of shear rate can be ignored, and determinations of the efflux times of the solution and diluted solvent can be made in the same viscometer.

In **alternative B**, the concentration c of cellulose is so chosen that if multiplied by the limiting viscosity number it gives a product of $[\eta] \cdot c = 3{,}0 \pm 0{,}4$, corresponding to viscosity ratio $\frac{\eta}{\eta_0}$ equal to 6 to 10. The determination shall then be carried out at a reproducible shear rate of 200 ± 30 s^{-1}; this involves the employment of two viscometers, one for the diluted solvent, and one for the solution. This alternative should be applied when there is reason to expect that the narrow capillary specified in alternative A will become clogged by undissolved particles, or when difficulty is experienced in attaining the prescribed degree of accuracy in weighing the small amounts of sample implied by alternative A in some cases.

1 Scope and field of application

This part of ISO 5351 specifies a method for the determination of the limiting viscosity number of cellulose in dilute cupri-ethylene-diamine (CED) solution.

This method is applicable to CED-soluble samples of cellulose, for instance in pulps and textiles.

NOTE — The viscosity test is a means for determining the extent of cellulose degradation produced by cooking and bleaching. This degradation greatly affects the suitability of pulp for dissolving purposes and papermaking.

ISO 5351/2 specifies a method for the determination of the limiting viscosity number of cellulose in dilute iron(III) sodium tartrate complex ($EWNN_{mod\ NaCl}$) solution.

2 References

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

ISO 1833, *Textiles — Binary fibre mixtures — Quantitative chemical analysis.*

ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing.*

ISO/TR 5090, *Textiles — Methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures.*

3 Principle

Measurement of the times of efflux of the diluted solvent and solution of cellulose through a capillary-tube viscometer at a specified concentration at 25 °C. Calculation by Martin's formula of the limiting viscosity number from these measurements, and from the known concentration of the solution.

4 Definitions

For the purpose of this International Standard, the following definitions apply.

4.1 shear rate, G : The velocity gradient of a fluid layer, parallel to the direction of flow, at the periphery of the capillary, defined by the equation

$$G = \frac{4\,V}{\pi\, r^3 t_f}$$

where

V is the volume between two arbitrary calibration marks of the viscometer, in millilitres;

r is the radius of the capillary tube, in centimetres;

t_f is the efflux time of the fluid, in seconds.

4.2 viscosity ratio : The ratio of the viscosities η and η_0 of the polymer solution of stated concentration and of the solvent respectively at the same temperature :

$$\frac{\eta}{\eta_0}$$

This ratio, as a number, is dimensionless.

4.3 viscosity relative increment : The viscosity ratio (4.2) minus one :

$$\frac{\eta}{\eta_0} - 1 = \frac{\eta - \eta_0}{\eta_0}$$

As a number, it is dimensionless.

4.4 viscosity number : The ratio of the viscosity relative increment (4.3) to the polymer concentration c in the solution :

$$\frac{\eta - \eta_0}{\eta_0 \cdot c}$$

Its unit is the millilitre per gram.

4.5 limiting viscosity number (L.V.N.) $[\eta]$: The limiting value of the viscosity number (4.4) at infinite dilution :

$$[\eta] = \lim_{c \to 0} \left(\frac{\eta - \eta_0}{\eta_0 \cdot c} \right)$$

Its unit is the millilitre per gram.[1)]

5 Pretreatment of test samples

5.1 Pulp samples

Take a sample corresponding to approximately 10 g of oven-dry mass. Split and tear the pulp into small pieces. If it is expected that the pulp will not disintegrate easily on being shaken in distilled water with copper pieces (6.2.3), disintegrate the sample in water in a suitable apparatus, and form thin sheets on a Büchner funnel. Dry the sheets at a temperature below 60 °C.

5.2 Textile samples

5.2.1 Generally

Prepare the sample as specified in ISO 5089.

Extract a sample of at least 3 g of air-dry mass in a Soxhlet apparatus with light petroleum for 1 h at a minimum rate of 6 cycles per hour. Allow the light petroleum to evaporate from the sample; soak the specimen in cold distilled or deionized water for 1 h, and then in a fresh portion of water at 65 ± 5 °C for a further 1 h. In both cases, use a liquor/specimen ration of 100 : 1. Agitate the liquor from time to time. Remove the excess water from the sample by squeezing, suction or by means of a centrifuge and then allow the sample to become air-dry. For additional information, see ISO 1833 and ISO/TR 5090.

5.2.2 For bast fibres

Prepare the sample as described in annex D.

6 Alternative A — Determination of limiting viscosity number at low concentration of cellulose

6.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled or deionized water.

6.1.1 Cupri-ethylene-diamine (CED) solution, solution of cupri-ethylene-diamine saturated with copper(II) hydroxide, for convenience referred to as cupri-ethylene-diamine solution.

The solution contains 1,0 mol of copper, and 2,0 mol of ethylene-diamine per litre. It is commercially available, or prepared and analysed as described in annex A. When a commercial solution is used, check the conformity of the copper and ethylene-diamine contents by determination (as indicated in clause A.7 of annex A).

If the copper and the ethylene-diamine concentrations differ by more than ± 2 % from the values given above a new solution has to be prepared.

NOTE — By reason of allergens, avoid contact of skin with CED and ethylene-diamine solutions. Ethylene-diamine is volatile and repeated exposure may lead to severe respiratory allergic reactions with subsequent sensitization. Cupri-ethylene-diamine solutions should not be pipetted by mouth.

6.1.2 Diluted cupri-ethylene-diamine solution, 50 % (V/V).

With a pipette, using a pipette filler, measure 25,0 ml of the cupri-ethylene-diamine solution (6.1.1) into the dissolving

1) The SI unit of $[\eta]$ is the cubic metre per kilogram. Information from ISO/TC 61, *Plastics*, indicates plans in TC 61 to adopt this unit in practice.

flask (6.2.2). Add, with a pipette, 25,0 ml of distilled or deionized water. Shake until dissolved.

6.2 Apparatus

Ordinary laboratory apparatus and

6.2.1 Constant-temperature bath, capable of being controlled at 25 ± 0,1 °C, accommodating the dissolving flask (6.2.2) and provided with a pump for water circulation through the jacket of the viscometer (6.2.6 and 7.2.1).

6.2.2 Dissolving flask, so constructed that the remaining air can be expelled when the flask is filled with 50 ml of test solution.

NOTE — A polyethene flask with screw cap and rubber gasket can be used. Some practice will enable the analyst to expel the air and lock the flask with the screw cap in one operation. If the pulp does not dissolve readily, use a flat-sided bottle. The air may also be expelled by a current of nitrogen.

6.2.3 Copper pieces, made of electrolytic copper.

6.2.4 Balance, accurate to ± 0,1 mg.

6.2.5 Timing device, capable of being read to the nearest 0,1 s.

6.2.6 Capillary-tube viscometer, with water jacket, connected to the constant-temperature bath (6.2.1) and having an efflux time of about 40 s for the diluted solvent (6.1.2), and a shear rate (4.1) of about 400 s^{-1} for a concentration of cellulose such that $[\eta] \cdot c = 1{,}5$ and $\frac{\eta}{\eta_0} = 3{,}4$.

A suitable viscometer is shown in figure 1.

Dimensions in millimetres

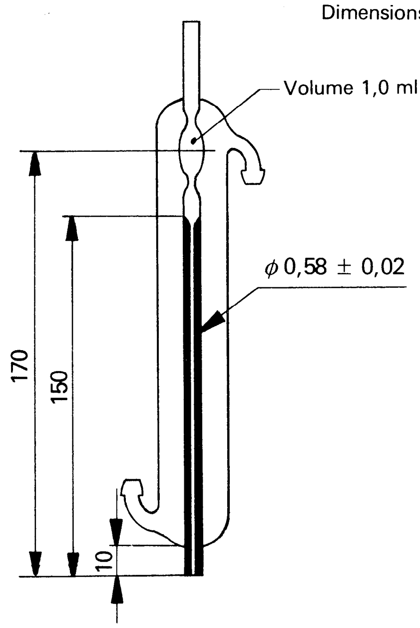

Figure 1 — Viscometer suitable for determination of the limiting viscosity number in accordance with alternative A

6.2.7 Shaking device.

6.3 Procedure

6.3.1 Choice of concentration of solution (see also annex C)

6.3.1.1 If the approximate limiting viscosity number of the sample is known, choose the concentration from table 1.

Table 1 — Concentration c to be used, as a function of the limiting viscosity number $[\eta]$ which will be measured

Limiting viscosity number $[\eta]$	Quantity of sample	Concentration c
ml/g	mg/50 ml	g/ml
< 200	250	0,005
201 to 400	200	0,004
401 to 600	125	0,002 5
601 to 900	80	0,001 6
901 to 1 200	60	0,001 2
1 201 to 1 500	45	0,000 9

6.3.1.2 If the approximate value of the limiting viscosity number of the sample is not known, test a sample of 125 mg/50 ml. If the limiting viscosity number so obtained is not within the range prescribed by table 1 for that concentration, make the test by choice of the correct concentration according to the value of the limiting viscosity number so derived.

6.3.2 Weighing of sample

Weigh the chosen amount of sample to an accuracy of ± 0,5 mg into the dissolving flask (6.2.2). At the same time, weigh out a separate sample for the determination of dry matter in accordance with ISO 638 or ISO 1833, sub-clause 1.7.

6.3.3 Preparation of test solution

With a pipette, add 25,0 ml of distilled or deionized water to the sample, together with some copper pieces (6.2.3). Close the flask, and shake it continuously until the sample has been completely disintegrated. With a pipette, using a pipette filler, add 25,0 ml of the CED solution (6.1.1) and expel all of the remaining air. Re-close the flask, shake in the shaking device (6.2.7) for 2 h, with the flat-sided bottles placed in the direction of movement of the device (see note 1). Immerse the flask in the constant-temperature bath (6.2.1) until a temperature of 25 ± 0,1 °C has been reached.

NOTES

1 Cold-alkali-treated pulps, and unbleached pulps of high viscosity, may sometimes be difficult to dissolve; this is effected more easily if swelling is prevented by first dissolving the pulp in a solution of lower CED concentration. Consequently, prepare a slurry of the pulp in 25 ml of distilled or deionized water, and add 5 ml of the CED solution (6.1.1); shake, and add another portion of 5 ml of CED solution (6.1.1), until the total added volume is 25,0 ml. Minimize degradation by shaking for as short a time as possible. For low viscosity pulps, about 3 min will be enough.