

Methods for

Determination of limiting viscosity number of cellulose in dilute solutions —

Part 1: Cupri-ethylene-diamine (CED) method

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

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National foreword

This Part of this British Standard has been prepared under the direction of the Paper Standards Committee and is identical with ISO 5351-1:1981 “*Cellulose in dilute solutions — Determination of limiting viscosity number — Part 1: Method in cupri-ethylene-diamine (CED) solution*” published by the International Organization for Standardization (ISO). Part 2 of this standard is identical with ISO 5351-2:1981 “*Cellulose in dilute solutions — Determination of limiting viscosity number — Part 2: Method in iron (III) sodium tartrate complex (EWNN_{mod NaCl}) solution*”.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following. The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker. Wherever the words “part of ISO 5351” and “International Standard” appear, referring to this standard, they should be read as “Part of BS 6306” and “British Standard” respectively.

Cross-references

International Standards	Corresponding British Standards
ISO 638:1978	BS 4502:1980 <i>Method for determination of dry-matter content of pulps</i> (Identical)
ISO 5089:1977	BS 4658:1978 <i>Methods of test for textiles — Preparation of laboratory test samples and test specimens for chemical testing</i> (Identical)
ISO/TR 5090:1977	BS Handbook 11:1974 <i>Methods of test for textiles, Section 5, pages 5/3 to 5/7. The removal of non-fibrous matter prior to quantitative analysis</i> (Technically equivalent)

The Technical Committee has reviewed the provisions of ISO 1833, to which reference is made in clause 2 and 5.2.1, and has decided that they are acceptable for use in conjunction with this standard. ISO 1833 is related to BS 4407:1975 “*Methods of test. Quantitative analysis of fibre mixtures*”.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 14, an inside back cover and a back cover. This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 $200 \pm 30 \text{ s}^{-1}$; 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:

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

its unit is the millilitre per gram¹⁾

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.

5 Pretreatment of test samples

5.1 Pulp samples

Take a sample corresponding to approximately 10 g of overndry 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.