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Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — ~~Part 1: General principles~~

Part 1:
General principles

Plastiques — Détermination de la viscosité des polymères en solution diluée à l'aide de viscosimètres à capillaires —

Partie 1: Principes généraux

FDIS stage

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 1628~~-~~-1:2021), which has been technically revised.

The main changes are as follows:

— ~~—~~an introduction section has been added in relation to the new procedure;

— ~~—~~the calculation of K-value was moved to ~~9.2;~~9.2;

— ~~—~~an alternative procedure has been incorporated, the differential pressure method~~,~~, (see Annex B), based on comparing the differential pressure in capillary tubing due to the flow of polymer solution and neat solvent simultaneously.

A list of all parts in the ISO 1628 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Two methods are described in this document to determine the viscosity of polymer solutions, the efflux time method and the differential pressure method. The results of both methods are equivalent. Differences may be found due to different conditions for the determination, such as concentration, solvent or shear rate.

The differential pressure method which has been incorporated in this document has the important advantage for industry that it is more easily adapted to automation, leading to improved efficiency, higher throughput, and enhanced safety for the operator. The new added method can help in the reduction of solvents use due to the lower requirement for washing of the capillaries.

Another advantage of the new alternative differential pressure method is that it can be integrated within existing polymer characterization workflows, as part of existing or new polymer analysis instrumental setups.

Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers —

Part 1:
General principles

1 Scope

This document specifies the general conditions for the determination of the reduced viscosity, intrinsic viscosity and K~~-~~-value of organic polymers in dilute solution. It specifies the standard parameters that are applied to viscosity measurement.

This document is applicable to develop standards for measuring the viscosities in solution of individual types of polymer. It is also applicable to measure and report the viscosities of polymers in solution for which no separate standards exist.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 80000~~-~~-1, *Quantities and units — Part 1: General*

ISO 80000~~-~~-4, *Quantities and units — Part 4: Mechanics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000~~-~~-1, ISO 80000~~-~~-4 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ~~—~~ISO Online browsing platform: available at ~~https://www.iso.org/obp~~https://www.iso.org/obp

— ~~—~~IEC Electropedia: available at ~~https://www.electropedia.org/~~https://www.electropedia.org/

3.1 Terms related to any liquid

3.1.1
viscosity
property of a fluid sheared between two parallel plates, one of which moves relative to the other in uniform rectilinear motion in its own plane, defined by the Newton formula

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

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

where

τ is the shear stress;

~~η~~η is the viscosity;

~~$\dot{\gamma}$~~$\dot{\gamma}$ is the velocity gradient or rate of shear, given by $\frac{dv}{dz}$ where v is the velocity of one plane relative to the other and z the coordinate perpendicular to the two planes~~.~~

Note 1 to entry: The units of viscosity are Pa·s.

Note 2 to entry: Viscosity is usually taken to mean “Newtonian viscosity”, in which case the ratio of shearing stress to velocity gradient is constant. In non-Newtonian behaviour, which is the usual case with solutions of polymers with high molar masses, the ratio varies with the shear rate. Such ratios are often called “apparent viscosities” at the corresponding shear rate.

3.1.2

viscosity/density ratio

kinematic viscosity

v

ratio defined by the formula

~~$v = \frac{\eta}{\rho}$~~

$$v = \frac{\eta}{\rho}$$

where ρ is the density of the fluid at the temperature at which the viscosity is measured~~.~~

Note 1 to entry: The units of kinematic viscosity are $m^2 \cdot s^{-1}$.

3.2 Terms related to polymer solutions

3.2.1

relative viscosity

viscosity ratio

η**r**

ratio of the viscosity of the polymer solution (of stated concentration) η and the viscosity of the solvent η_0, at the same temperature

~~$\eta_r = \frac{\eta}{\eta_0}$~~

$$\eta_r = \frac{\eta}{\eta_0}$$

Note 1 to entry: The ratio has no dimensions.

3.2.2

relative viscosity increment

viscosity ratio increment and specific viscosity

η**sp**

viscosity ratio minus one

~~$\eta_{sp} = \left(\frac{\eta}{\eta_0}\right) - 1 = \frac{\eta - \eta_0}{\eta_0}$~~

$$\eta_{sp} = \left(\frac{\eta}{\eta_0}\right) - 1 = \frac{\eta - \eta_0}{\eta_0}$$

Note 1 to entry: The increment has no dimensions.

3.2.3
reduced viscosity
viscosity number
I
ratio of the relative viscosity increment to the polymer concentration c in the solution

~~$I = \frac{\eta - \eta_0}{\eta_0 c}$~~

$$I = \frac{\eta - \eta_0}{\eta_0 c}$$

Note 1 to entry: The units of reduced viscosity are m^3/kg.

Note 2 to entry: The reduced viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations can be necessary.

3.2.4
inherent viscosity
logarithmic viscosity number
η**inh**
ratio of the natural logarithm of the viscosity ratio to the polymer concentration in the solution

~~$\eta_{inh} = \frac{\ln\left(\frac{\eta}{\eta_0}\right)}{c}$~~

$$\eta_{inh} = \frac{\ln\left(\frac{\eta}{\eta_0}\right)}{c}$$

Note 1 to entry: The dimensions and units are the same as those given in ~~3.2.3.~~3.2.3.

Note 2 to entry: The inherent viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations can be necessary.

3.2.5
intrinsic viscosity
limiting viscosity number
η
limiting value of the reduced viscosity or of the *inherent viscosity* ~~(3.2.4)~~(3.2.4) at infinite dilution

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

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

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

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

Note 1~~-~~ to entry:~~-~~ The dimensions and units are the same as those given in ~~3.2.3.~~3.2.3.

Note 2~~-~~ to entry:~~-~~ The effect of the shear rate on the functions defined in ~~3.2.1~~3.2.1 to ~~3.2.5~~3.2.5 has been neglected, since this effect is usually negligible for values of the reduced viscosity, inherent viscosity and intrinsic viscosity less than 0,5 m^3/kg, i.e. 500 cm^3/g. Strictly speaking, all these functions can be defined at the limiting (preferably infinitely small) value of the shear rate.

3.2.6

***K*~~-~~-value**

empirical parameter related to the relative viscosity and concentration used to estimate the viscosity average of the molecular mass of polymers

Note 1~~-~~ to~~-~~ entry: For constant measurement parameters such as type of solvent, concentration and temperature, the K-value depends only on the viscosity average of the molecular mass distribution.

4 Principle

4.1 General

The data needed for the evaluation of the functions defined in ~~3.2~~3.2 are obtained comparing viscosity measurements of a polymer solution and the solvent.

4.2 Method A ~~–~~— Efflux time method

The data are obtained by means of a capillary-tube viscometer. The efflux times of a given volume of solvent t_0 and of solution t are measured at fixed temperature and atmospheric-pressure conditions in the same viscometer. The efflux time of a liquid is related to its viscosity by the Poiseuille-Hagenbach-Couette formula as shown in ~~Formula (1).~~Formula (1):

$$\cancel{v = \frac{\eta}{\rho} = Ct\left(\frac{A}{t^2}\right)} \quad v = \frac{\eta}{\rho} = Ct - \left(\frac{A}{t^2}\right) \qquad (1)$$

where

v is the viscosity/density ratio;

C is a constant of the viscometer;

A is a parameter of the kinetic-energy correction;

ρ is the density of the liquid;

η is the viscosity of the liquid;

t is the efflux time.

For the purposes of this document, the kinetic energy correction ~~$\left(\frac{A}{t^2}\right)$~~$(\frac{A}{t^2})$ shall be regarded as negligible when it is less than 3 % of the viscosity of the solvent. Hence, ~~Formula (1)~~Formula (1) can be reduced to ~~Formula (2):~~Formula (2):

~~$v = \frac{\eta}{\rho} = Ct$~~ $v = \frac{\eta}{\rho} = Ct$ (2)

Moreover, if the solution concentrations are limited so that the solvent density ρ_0 and that of the solution ρ differ by less than 0,5 %, the viscosity ratio ~~$\frac{\eta}{\eta_0}$~~ $\frac{\eta}{\eta_0}$ will be given by the so-called “efflux time ratio” ~~$\frac{t}{t_0}$.~~ $\frac{t}{t_0}$.

The need for these constraints, and the consequences of not observing them, is described in ~~Annex A.~~Annex A.

4.3 Method B ~~–~~— Differential pressure method

The data are obtained by means of a 2-capillary relative viscometer. The differential pressure across each of the 2 capillaries connected in series, one of them receiving the solvent Δp_0, the other receiving the polymer solution Δp, are measured at a fixed temperature while applying a forced flow through them.

The differential pressure across a capillary tubing is related to the viscosity of the flowing liquid under laminar flow regime, by the Poiseuille formula as shown in ~~Formula (3):~~Formula (3):

~~$\Delta p = \frac{8q_v l}{\pi r^4}\eta$~~ $\Delta p = \frac{8q_v l}{\pi r^4}\eta$ (3)

where

Δp is the differential pressure in a capillary tubing;

q_v is the liquid flow rate;

l is the capillary tubing length;

r is the capillary tubing radius;

η is the viscosity of the liquid;

As in a 2~~-~~-capillaries serial configuration viscometer (see ~~5.2)~~5.2) the same flow rate q_v is maintained for the solvent and the polymer solution, the viscosity ratio ~~$\frac{\eta}{\eta_0}$~~ $\frac{\eta}{\eta_0}$ is proportional to the pressure ratio ~~$\frac{\Delta p}{\Delta p_0}$.~~ $\frac{\Delta p}{\Delta p_0}$. By introducing the instrumental constant K_v the viscosity ratio ~~$\frac{\eta}{\eta_0}$~~ $\frac{\eta}{\eta_0}$ can be calculated from the pressure ratio as seen in ~~Formula (4)~~Formula (4) and ~~Formula (5):~~Formula (5):

$$\frac{\eta}{\eta_0} = K_v \cdot \frac{\Delta p}{\Delta p_0} \qquad (4)$$

with