

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO 1628-1:2024

https://standards.iteh.ai/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: +41 22 749 01 11 Email: copyright@iso.org Website: www.iso.org Published in Switzerland

Contents

Forew	vord	iv
Introd	luction	v
1	Scope	
2	Normative references	
3	Terms and definitions3.1Terms related to any liquid3.2Terms related to polymer solutions	1
4	Principle 4.1 General 4.2 Method A — Efflux time method 4.3 Method B — Differential pressure method	3
5	Apparatus 5.1 Efflux time method 5.2 Differential pressure method	5
6	Solutions 6.1 Preparation 6.2 Concentration	9
7	Temperature of measurement	
8	Procedure 8.1 Efflux time method 8.1.1 General 8.1.2 Preparing and charging the viscometer 8.1.3 Efflux time measurement. 8.2 Differential pressure method 8.2.1 General 8.2.2 Collection of viscosity ratio increment signal	10 10 10 10 10 11 11 11
9 https:/	Expression of resultsISO 1628-1:20249.1Reduced viscosity and intrinsic viscosity9.2K-value	1-20212
10	Test report	
Annex	A (informative) Efflux time method — Notes on sources of error	
Annex	B (informative) Differential pressure method — Notes on sources of error	
Annex	c (normative) Efflux time method — Cleaning of apparatus	
Biblio	graphy	23

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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. $\frac{|SO | 628 - 1:2024}{|SO | 628 - 1:2024}$

https://standards.iteh.ai/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024 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 <u>9.2;</u>
- an alternative procedure has been incorporated, the differential pressure method (see <u>4.3</u>), 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 <u>www.iso.org/members.html</u>.

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.

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO 1628-1:2024

https://standards.iteh.ai/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024

iTeh Standards (https://standards.iteh.ai) Document Preview

<u>ISO 1628-1:2024</u> https://standards.iteh.ai/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024

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

ISO 1628-1:2024

3 ttp Terms and definitions standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024

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
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

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

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

where

- τ is the shear stress;
- η is the viscosity;
- $\dot{\gamma}$ 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

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

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 m²·s⁻¹.

3.2 Terms related to polymer solutions

3.2.1 relative viscosity viscosity ratio

Document Preview

ratio of the viscosity of the polymer solution (of stated concentration) η and the viscosity of the solvent η_0 , at the same temperature i/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024

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

Note 1 to entry: The ratio has no dimensions.

3.2.2

 η_r

relative viscosity increment viscosity ratio increment and specific viscosity

 η_{sp} viscosity ratio minus one

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

Note 1 to entry: The increment has no dimensions.

```
3.2.3
reduced viscosity
viscosity number
l
```

ISO 1628-1:2024(en)

ratio of the relative viscosity increment to the polymer concentration *c* in the solution

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

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/m³, i.e. 0,005 g/cm³), 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

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

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

Note 2 to entry: The inherent viscosity is usually determined at low concentration (less than 5 kg/m³, 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



limiting value of the reduced viscosity or of the *inherent viscosity* (3.2.4) at infinite dilution

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

$$Document Preview$$

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

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

Note 1 to entry: The dimensions and units are the same as those given in <u>3.2.3</u>.

Note 2 to entry: The effect of the shear rate on the functions defined in 3.2.1 to 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 m³/kg, i.e. 500 cm³/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 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):

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

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

$$v = \frac{\eta}{\rho} = Ct$$
 (https://standards.iteh.ai) (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 $\frac{\eta}{\eta_0}$ will be given by the so-called "efflux time ratio" $\frac{t}{t_0}$. The need for these constraints, and the consequences of not observing them, is described in <u>Annex A</u>.

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 <u>Formula (3)</u>:

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

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;

ISO 1628-1:2024(en)

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

$$\frac{\eta}{\eta_0} = K_v \cdot \frac{\Delta p}{\Delta p_0} \tag{4}$$

with

$$K_{v} = \frac{r^{4} l_{0}}{r_{0}^{4} l} \tag{5}$$

where

- *l* is the polymer solution capillary tubing length;
- *r* is the polymer solution capillary tubing radius;
- l_0 is the solvent capillary tubing length;
- r_0 is the solvent capillary tubing radius.

The instrumental constant K_v can easily be measured by flowing solvent through both capillaries given that the viscosity ratio $\frac{\eta}{\eta_0}$ is 1,0 for solvent, and therefore solving in Formula (4):

$$K_v = \frac{\Delta p_0}{\Delta p}$$
 Document Previo

Possible sources of errors for this method are described in <u>Annex B</u>.

<u>ISO 1628-1:2024</u>

bttps://standards.iteh.ai/catalog/standards/iso/a52d47d2-9c09-4c16-8156-f37c063a9b6b/iso-1628-1-2024 5 Apparatus

5.1 Efflux time method

5.1.1 Capillary viscometer, of the suspended-level Ubbelohde type

The use of a viscometer having the dimensions given in <u>Figure 1</u> or <u>Figure 2</u> is strongly recommended. Furthermore, it is strongly recommended that the size of the viscometer be chosen from among those listed in <u>Table 1</u>. The choice is determined by the viscosity/density ratio of the solvent at the temperature of the measurement, as indicated in <u>Table 1</u>. The next-smaller viscometer may also be used.

Other types of viscometer listed in ISO 3105 may be used, provided they give results equivalent to those given by the particular size of Ubbelohde viscometer chosen on the basis of the criteria specified in the preceding paragraph. In cases of dispute, an Ubbelohde viscometer shall be used.

With automated apparatuses, fitted with special timing devices, equivalent results with larger sizes of capillaries than those listed for the appropriate solvent viscosity/density ratio in <u>Table 1</u> can be obtained. Some modified viscometer excluding parts P and L can be used, and the measuring part of the viscometer meets the recommendations of the <u>Table 1</u>.

5.1.2 Viscometer holder, suitable to hold the viscometer firmly in the thermostatic bath (5.1.3) in the vertical position.