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Plastics — Polyamides — Determination of viscosity number

Plastiques — Polyamides — Détermination de l'indice de viscosité

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 307 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This fifth edition cancels and replaces the fourth edition (ISO 307:2003), which has been technically revised.

Introduction

This International Standard specifies a method for the determination of the viscosity number of dilute solutions of polyamides in certain specified solvents. The determination of the viscosity number of a polyamide provides a value that depends on the molecular mass of the polymer, but does not strictly correlate with the molecular mass.

Additives such as flame-retardants and modifiers often interfere with the viscosity measurement and may have an increasing effect on the viscosity number in one solvent and a decreasing effect in another solvent. The extent of the effect depends among others on the additive, the quantity of the additive, the presence of other additives and reactions.

The viscosity number of a polyamide sample containing additives that interfere with the viscosity measurement, measured in a specific solvent, represents a specific viscosity number for the polyamide under investigation and the actual measurement conditions. The measured viscosity number cannot, in principle, be converted from one solvent to another and is only suitable for intra-product comparison.

The viscosity number of pure polyamides or polyamides containing additives that do not interfere with the viscosity measurement can be converted from one solvent to another by a general relationship for that type of polyamide.

Polyamide test samples for the determination of the viscosity number must be completely soluble in the solvents mentioned. Additives contained in them, like glass and carbon fibres, must be separated from the solution.

As it is not possible to distinguish between extractables such as caprolactam, its oligomers and other extractable additives, these are considered as an essential part of the sample and therefore included in the sample mass.

The test method is applicable for production control and intra-product comparison even if the polyamide contains additives that do interfere with the viscosity measurement. However, it should be realised that deviations of the viscosity number can be caused by either the polyamide itself, effects caused by the additives present, or a combination of these.

The interference of additives with the viscosity determination can be checked by comparing the viscosity results of dry blend mixtures and regular production samples at several concentrations of the additive under investigation and in the solvents concerned. It should be noted that the other additives present also could influence the viscosity result.

The repeatability and reproducibility of the test method are strongly influenced by the correctness of the solvent concentration, the use of the Hagenbach correction if applicable and the temperature of the solvent on diluting the sample solution.

In this International Standard two specific viscometers are recommended. Furthermore, other types of viscometers listed in ISO 3105 may also be used, provided that the results are demonstrated to be equivalent to those measured with the recommended viscometers. It is to be expected that in the next revision the use of the other types of viscometers will be excluded.

Plastics — Polyamides — Determination of viscosity number

1 Scope

This International Standard specifies a method for the determination of the viscosity number of dilute solutions of polyamides in certain specified solvents.

Polyamide samples must be completely soluble in the solvents mentioned. Additives such as flame-retardants and modifiers often interfere with the viscosity measurement, having an increasing effect on the viscosity number in formic acid and a decreasing effect on the viscosity number in sulfuric acid. The extent of the effect for polyamide compounds depends on the additive, the quantity of the additive, the presence of other additives and the compounding conditions.

For pure polyamides or polyamides containing additives that do not interfere with the viscosity measurement, the determination of the viscosity number of a polyamide provides a measure of the molecular mass of the polymer. The viscosity number of pure polyamides or polyamides which contain additives that do not interfere with the viscosity measurement can be converted from one solvent to another.

The viscosity number of polyamides containing additives that do interfere with the viscosity measurement is specific to the solvent used and the material composition. In this case, the measured viscosity number cannot be converted from one solvent to another.

The method is applicable to the polyamides designated PA 46, PA 6, PA 66, PA 69, PA 610, PA 612, PA 11, PA 12, PA 6T/66, PA 6I/6T, PA 6T/6I/66, PA 6T/6I, PA 6I/6T/66 and PA MXD6 as defined in ISO 1874-1, as well as to copolyamides, compounds of polyamides and other polyamides that are soluble in one of the specified solvents under the specified conditions.

The method is not applicable to polyamides produced by anionic polymerization of lactams or produced with cross-linking agents; such polyamides are normally insoluble in the specified solvents.

The viscosity number is determined by the general procedure specified in ISO 1628-1, observing the particular conditions specified in this International Standard.

2 Normative references

The following referenced documents are indispensable for the application 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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1628-1, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 1874-1, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 1: Designation*

ISO 3105, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 3451-4, *Plastics — Determination of ash — Part 4: Polyamides*

ISO 15512, *Plastics — Determination of water content*

ASTM D789, *Standard test methods for determination of relative viscosity of polyamide (PA)*

JIS K 6920-2:2000, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1628-1 and the following apply.

3.1
viscosity number of a polymer
number calculated by the following formula for the viscometers mentioned in this International Standard and flow times long enough so that no kinetic energy correction need be applied:

$$\mathrm{VN} = \left(\frac{\eta}{\eta_0} - 1 \right) \times \frac{1}{c} \tag{1}$$

where

η is the viscosity of a solution of the polymer in a specified solvent, in Pascal seconds or N/m^2·s;

η_0 is the viscosity of the solvent, expressed in the same units as η;

$\frac{\eta}{\eta_0}$ is the relative viscosity of a solution of the polymer in a specified solvent;

c is the concentration, in grams per millilitre, of the polymer in the solution;

VN is the viscosity number, expressed in millilitres per gram.

NOTE 1 For a particular viscometer used and with substantially equal densities of the solvent and solution, the viscosity ratio is given by the flow time ratio for the solution concentration:

$$\frac{\eta}{\eta_0} \tag{2}$$

where

$\frac{\eta}{\eta_0}$ is the relative viscosity of a solution of the polymer in a specified solvent.

NOTE 2 As mentioned in ISO 3105, in case of flow times below 200 s and 60 s, for type 1 and type 2 Ubbelohde viscometers respectively, a correction for kinetic correction has to be applied: the so-called Hagenbach correction. For other types of viscometers, the kinetic energy correction has to be applied if the correction is $\geqslant$ 0,15 %.

NOTE 3 The flow time of a liquid is related to its viscosity by the formula

$$v = \frac{\eta}{\rho} = C \times t - \left(\frac{A}{t^2} \right) \tag{3}$$

