
Polimerni materiali - Poliamidi - Ugotavljanje števila viskoznosti (ISO/DIS 307:2026)

Plastics - Polyamides - Determination of viscosity number (ISO/DIS 307:2026)

Kunststoffe - Polyamide - Bestimmung der Viskositätszahl (ISO/DIS 307:2026)

Plastiques - Polyamides - Détermination de l'indice de viscosité (ISO/DIS 307:2026)

Ta slovenski standard je istoveten z: prEN ISO 307

ICS:

83.080.20

Plastomeri

Thermoplastic materials

oSIST prEN ISO 307:2026

en,fr,de

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DRAFT International Standard

ISO/DIS 307

Plastics — Polyamides — Determination of viscosity number

Plastiques — Polyamides — Détermination de l'indice de viscosité

ICS: 83.080.20

ISO/TC 61/SC 9

Secretariat: **KATS**

Voting begins on:
2026-06-12

Voting terminates on:
2026-09-04

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This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 307:2026(en)

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Published in Switzerland

ISO/DIS 307:2026(en)

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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.

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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

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

This seventh edition cancels and replaces the sixth edition (ISO 307:2007), which has been technically revised.

The main change is the incorporation of an alternative procedure, the differential pressure method (following same change in ISO 1628-1:2024), based on comparing the differential pressure in capillary tubing due to the flow of polymer solution and neat solvent simultaneously.

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.

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Introduction

This document 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 are intended to be completely soluble in the solvents mentioned. Additives contained in them, like glass and carbon fibres, are to 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 document, two specific glass capillary 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.

The differential pressure method general principles are described in ISO 1628-1:2024 and can be used as an alternative to the efflux time method with equivalent results.

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Plastics — Polyamides — Determination of viscosity number

1 Scope

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

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 16396-1, as well as to co-polyamides, 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 1628-1:2024, observing the particular conditions specified in this document.

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 1628-1:2024, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 25337:2010, *Plastics — Production quality control — Statistical method for using single measurements*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1628-1:2024 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

4.1 Efflux time method

The times of flow of a solvent and a solution of the polyamide at a concentration of 0,005 g/ml in the solvent are measured at 25 °C, the same viscometer being used for both measurements. The viscosity number is calculated from these measurements and from the known concentration of the solution.

4.2 Differential pressure method

The differential pressure across each of the 2 capillaries connected in series, one of them receiving the solvent having the differential pressure Δp_0 , the other receiving the solution of the polyamide at a concentration of

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0,005 g/ml in the solvent having the differential pressure Δp , are measured at 25°C. The viscosity number is calculated from these measurements and from the known concentration of the solution.

5 Reagents and materials

5.1 Solvents and reagents

5.1.1 General

Only reagents of recognised analytical grade and only distilled water or water of equivalent purity shall be used.

When using an automated instrument to apply the differential pressure method, use only solvents compatible with the specific instrument used. In particular, some components of the equipment are not compatible with acids.[AC1] Furthermore, any solvent used in the equipment shall be filtered through a 0.45 μm membrane to prevent clogging of the tubing.

WARNING — Persons using this document should be familiar with laboratory practice. This document does not purport to address all of the safety concerns associated with its use. Some chemicals, for example 1,1,2,2-tetrachloroethane, are prohibited in some countries. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

WARNING — WARNING — Avoid contact with the skin and inhalation of any vapours of the solvents and cleaning liquids.

5.1.2 Sulphuric acid, 96,00 % $\pm$ 0,20 % (by mass) solution.

For the determination of the concentration of commercial sulphuric acid (95 % to 98 %) and adjustment to 96,00 %, is referred to [Annex A](#) and [Annex B](#).

5.1.3 Formic acid, 90,00 % $\pm$ 0,15 % (by mass) solution.

The solvent shall be stored in a brown glass bottle. Its concentration shall be checked at least every 2 weeks. It shall not contain more than 0,2 % acetic acid or methyl formate.

For the determination of the concentration of commercial formic acid (90 %) and adjustment to 90,00 % $\pm$ 0,15 %, is referred to [Annex A](#) and [Annex B](#).

5.1.4 *m*-Cresol, meeting the following specifications:

- appearance: clear and colourless;
- *m*-cresol content: 99 % (by mass) min.;
- *o*-cresol content: 0,3 % (by mass) max.;
- water content: 0,13 % (by mass) max.

m-Cresol of the required purity may be obtained by distillation of chemically pure *m*-cresol, preferably in vacuum. If distillation is used, nitrogen shall be used for pressure compensation to avoid oxidation. Its purity may be checked by gas chromatography. The solvent shall be stored in a brown glass bottle.

5.1.5 Phenol, 99 % (by mass) min.

5.1.6 1,1,2,2-tetrachloroethane, 99,5 % (by mass) min.

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5.1.7 Phenol/1,1,2,2-tetrachloroethane.

6 parts by mass of phenol (5.1.5) shall be weighed and dissolved in 4 parts by mass of 1,1,2,2-tetrachloroethane (5.1.6). Work to an accuracy of 1 % or better in the weighing. The mixture shall be stirred in its original container at 23 °C to prevent crystallization.

5.1.8 Ortho-phosphoric acid, 85 % (by mass), density 1,71 g/ml.**5.1.9 *m*-Cresol/phosphoric acid.**

50 ml of *m*-cresol (5.1.4) shall be transferred into a weighing flask (6.1.4) and added with a glass pipette (6.1.5) 0,14 ml of Ortho-phosphoric acid (5.1.8). The flask shall be closed and stirred with a magnetic stirrer for 30 min at 100 °C. The solution shall be added to approximately 800 ml of *m*-cresol in a volumetric flask while continuously stirring. The weighing flask shall be rinsed several times with *m*-cresol adding this to the *m*-cresol solution. Remove the magnetic stirrer and dilute to the mark. Stir the solution for 30 min.

5.2 Cleaning liquids

5.2.1 Chromic acid solution, prepared by mixing equal volumes of sulphuric acid (96 %, $\rho_0 = 1,84$ g/ml, industrial quality) and a saturated solution of potassium dichromate (99,5 %, industrial quality). If required, the chromic acid solution may be replaced by other equally effective cleaning liquids.

5.2.2 Acetone (99,5 %, industrial quality), or any water-soluble low-boiling-point solvent (industrial quality).

6 Apparatus**6.1 Efflux time method**

6.1.1 Vacuum drying cabinet, with pressure less than 100 k Pa.

6.1.2 Balance, accurate to 0,1 mg.

6.1.3 Volumetric flask, of capacity 50 ml or 100 ml, complying with the requirements of ISO 1042, fitted with a ground-glass stopper.

6.1.4 Weighing flask, 100 ml, fitted with a ground-glass stopper.

6.1.5 Pipette, 0,2 ml, readable to 0,01 ml.

6.1.6 Shaking apparatus or magnetic stirrer.

6.1.7 Sintered-glass filter, with a pore size between 40 µm and 100 µm (grade P 100), or stainless-steel sieve, with apertures of about 0,075 mm².

6.1.8 Viscometer, of the suspended-level Ubbelohde type, complying with the requirements of ISO 3105. The essential dimensions of the viscometer are shown in Figure 1. For use with the formic acid solution (5.1.3), the inside diameter of the capillary shall be 0,58 mm ± 2 % (complying with the requirements of size No. 1 of ISO 3105). For use with the sulphuric acid solution (5.1.2) or *m*-cresol (5.1.4), the inside diameter of the capillary shall be 1,03 mm ± 2 % (complying with the requirements of size No. 2 of ISO 3105).

Other types of viscometer listed in ISO 3105 may be used, provided that the results are demonstrated to be equivalent to those of the Ubbelohde viscometers specified above. In cases of dispute, the recommended viscometer shall be used.

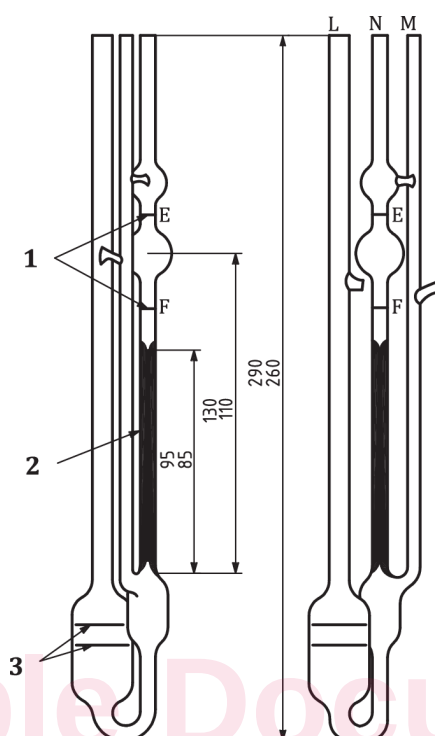
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ISO 1628-1:2024 shall be followed on selecting other type(s) of viscometer.

In this document, the No. 1 and No. 2 Ubbelohde viscometers according to ISO 3105 are recommended.

NOTE It is to be expected that at the next 5 year revision only these two viscometers will be allowed.

Dimensions in millimetres



Key

- 1 graduation marks for a volume of $4 \text{ ml} \pm 0,2 \text{ ml}$
- 2 capillary tube of diameter $0,58 \text{ mm} \pm 2 \%$ for formic acid; $1,03 \text{ mm} \pm 2 \%$ for sulphuric acid and *m*-cresol
- 3 filling marks

Figure 1 — Ubbelohde viscometer

6.1.9 Thermometer, a liquid-in-glass, “total immersion” thermometer, reading to $0,05 \text{ }^{\circ}\text{C}$ in the range to be used and in a known state of calibration, is suitable. Other thermometric devices of at least equal precision may be used.

6.1.10 Thermostatic bath, capable of being maintained and controlled at $25,00 \text{ }^{\circ}\text{C} \pm 0,05 \text{ }^{\circ}\text{C}$.

6.1.11 Time device, for example a stop-watch, accurate to $0,1 \text{ s}$.

6.1.12 Centrifuge.

6.2 Differential pressure method

6.2.1 Analytical balance, accurate to $0,1 \text{ mg}$.

6.2.2 A schematic diagram of a **relative viscometer** used in this method is depicted in ISO 1628-1:2024.

The reference/solvent capillary and the measurement/polymer solution capillary shall be kept at the same temperature of $25 \text{ }^{\circ}\text{C}$, enclosed in a proper temperature-controlled device with variations from the specified