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Plastics — Polyamides — Determination of viscosity number

Plastiques — Polyamides — Détermination de l'indice de viscosité

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Foreword

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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 sixth edition cancels and replaces the fifth edition (ISO 307:2007), which has been technically revised to update Clause 9. It also incorporates the Amendment ISO 307:2007/Amd.1:2013.

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

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 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 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 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 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 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 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*

ISO 16396-1, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 1: Designation system, marking of products and basis for specifications*

3 Terms and definitions

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

3.1
viscosity number
<polymer> value calculated by the following formula for flow times long enough so that no kinetic energy correction need be applied:

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

where

η is the viscosity of a solution of the polymer in a specified solvent, in Pa/ s or N/m2·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 g/ml, of the polymer in the solution;

VN is the viscosity number, expressed in ml/g

Note 1 to entry: The formula is applicable to the viscometers of the suspended level Ubbelohde type, complying with the requirements of ISO 3105.

Note 2 to entry: 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:

$$\frac{\eta}{\eta_0}$$

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

Note 3 to entry: 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 ≥0,15 %.

Note 4 to entry: The flow time of a liquid is related to its viscosity by the formula:

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

where

η is the viscosity of a solution of the polymer in a specified solvent, in Pa/ s or N/m2·s;

v is the viscosity/density ratio, in metres squared per second;

ρ is the density of the liquid, in kilograms per cubic metre;

C is the constant of the viscometer, in metres squared per second squared;

t is the flow time, in seconds;

A is the parameter of the kinetic correction in metres squared seconds.

Note 5 to entry: For a particular viscometer used, with substantially equal densities of the solvent and solution and a given kinetic factor, the viscosity ratio

$$\frac{\eta}{\eta_0}$$

is given by the flow time ratio for the solution concentration in this document, each flow time reduced with the so-called Hagenbach correction (in seconds) given by the manufacturer for the viscometer as a function of the flow time.

4 Principle

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.

5 Reagents and materials

5.1 Solvents and reagents

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

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

5.1.1 Sulfuric acid, 96,00 % ± 0,20 % (by mass) solution.

For the determination of the concentration of commercial sulfuric acid (95 % to 98 %) and adjustment to 96,00 %, is referred to Annexes A and B.

5.1.2 Formic acid, 90,00 % ± 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 % ± 0,15 %, is referred to Annexes A and B.

5.1.3 *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.

5.1.4 Phenol, 99 % (by mass) min.

5.1.5 1,1,2,2-tetrachloroethane, 99,5 % (by mass) min.

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

Weigh out 6 parts by mass of phenol (5.1.4) and dissolve in 4 parts by mass of 1,1,2,2-tetrachloroethane (5.1.5). Work to an accuracy of 1 % or better in the weighings. Stir the mixture in its original container at 23 °C to prevent crystallization.

5.1.7 Orthophosphoric acid, 85 % (by mass), density 1,71 g/ml.

5.1.8 *m*-Cresol/phosphoric acid.

Transfer 50 ml of *m*-cresol (5.1.3) into a weighing flask (6.4) and add with a glass pipette (6.5) 0,14 ml of orthophosphoric acid (5.1.7). Close the flask and stir with a magnetic stirrer for 30 min at 100 °C. Add the solution to approximately 800 ml of *m*-cresol in a volumetric flask while continuously stirring. Rinse the weighing flask several times with *m*-cresol and add 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 sulfuric acid (96 %, ρ_{20} = 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 Vacuum drying cabinet, with pressure less than 100 kPa.

6.2 Balance, accurate to 0,1 mg.

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

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

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

6.6 Shaking apparatus or magnetic stirrer.

6.7 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 mm^2.

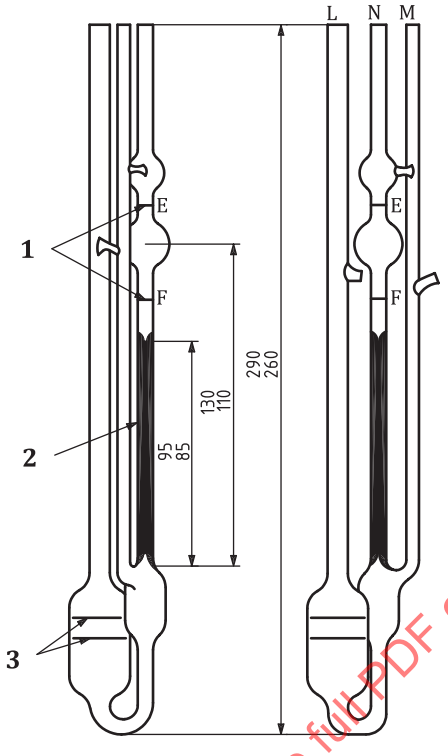
6.8 Viscometer, of the suspended-level Ubbelohde type, complying with the requirements of ISO 3105. The essential dimensions of the viscometer are shown in Figure 1. For use with the formic acid solution (5.1.2), the inside diameter of the capillary shall be 0,58 mm ± 2 % (complying with the requirements of size No. 1 of ISO 3105). For use with the sulfuric acid solution (5.1.1) or *m*-cresol (5.1.3), 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.

ISO 1628-1 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. 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 ml ± 0,2 ml

2 capillary tube of diameter 0,58 mm ± 2 % for formic acid; 1,03 mm ± 2 % for sulfuric acid and *m*-cresol

3 filling marks

Figure 1 — Ubbelohde viscometer

6.9 Thermometer, a liquid-in-glass, "total immersion" thermometer, reading to 0,05 °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.

6.10 Thermostatic bath, capable of being maintained and controlled at 25,00 °C ± 0,05 °C.

6.11 Time device, for example a stop-watch, accurate to 0,1 s.

6.12 Centrifuge.

7 Preparation of test samples

7.1 General

Polyamide test samples for the determination of the viscosity number shall be soluble in the solvents mentioned, except for additives present, such as reinforcement or fillers.

The sample should contain less than 0,28 % moisture. If it contains more than 0,28 % moisture the sample should be dried. Generally, drying at 70 °C in a vacuum for 4 h to 6 h is adequate.

NOTE The dissolution time of some samples might be too long for adequate production control. In these cases, the material can be ground in order to shorten the dissolution time, provided that the results are demonstrated to be equivalent.

7.2 Samples containing less than 98 % (by mass) polyamide

For samples containing more than 2 % additives, the amount of additives shall be either determined by a specifically developed method or taken from the recipe. The method of determination shall be mentioned in the report.

The water content of the sample shall be determined according to ISO 15512. The ash content shall be determined according to ISO 3451-4.

The correct amount of polyamide sample to be weighed out is calculated using Formula (1).

Some additives, such as antimony trioxide and zinc sulfide, are completely volatilized during the calcination according to ISO 3451-4. Materials reinforced with glass fibre contain flame-retardant antimony trioxide and/or other volatilizable additives. If the total content of additives is more than 2 %, these shall be brought into account by the formulation of the sample for calculating the exact test portion.

NOTE For production quality control purposes, the laboratory response time for determination of the additives might be too long for adequate production control. In these cases, the additive(s) content in the production recipe can be used for calculating the amount of sample, if the total variation of the polymer content is less than 4 % (by mass), e.g. 65 % PA would range from 63 % to 67 %.

8 Calculation of test portion

Calculate the mass m_c, in milligrams, of the test portion according to Formula (1):

$$m_c = \frac{250}{1 - \frac{w_1 + w_2 + w_3}{100}} \tag{1}$$

where

w_1 is the water content of the sample, expressed as a percentage by mass, determined in accordance with ISO 15512;

w_2 is the content of inorganic materials (for example fillers or glass fibres) in the sample, expressed as a percentage by mass, determined in accordance with ISO 3451-4;

w_3 is the content of other materials (for example other polymers, such as polyolefins, or additives, such as flame-retardants), expressed as a percentage by mass, determined by appropriate methods.

For the content of the additive(s) which cannot be determined, the content according to the product recipe shall be used.

9 Selection of solvent

The value of the viscosity number of a polyamide depends on the solvent used.

The solvent or solvents to be used for a particular polyamide are specified below.

a) For PA 6, PA 46, PA 66, PA 69, PA 610, PA MXD6 and corresponding copolyamides, formic acid solution or sulfuric acid shall be used as solvent. For polyamides containing additives that liberate gases in acidic solvents, *m*-cresol shall be used as the solvent. In cases of dispute, formic acid shall be used as a solvent.

b) For PA 612, the sulfuric acid solution or *m*-cresol shall be used as solvent. In cases of dispute, *m*-cresol shall be used.

c) For PA 11, PA 12, PA 11/12 copolymers, *m*-cresol shall be used as a solvent. In cases of dispute about ammonium carboxylate influencing viscosity through the formation of end-group associations, additional measurements shall be made using *m*-cresol/phosphoric acid solution as a solvent (5.1.8).

d) For PA 6T/66, PA 6I/66, PA 6I/6T, PA 6T/6I/66, PA 6T/6I, PA 6I/6T/66, *m*-cresol, phenol/1,1,2,2-tetrachloroethane or sulfuric acid shall be used as solvent. In cases of dispute, *m*-cresol shall be used.

e) For other polyamides, any of the mentioned solvents may be used.

NOTE 1 In the future revision of this document, it is the intention that for a given PA only one solvent will be allowed.

NOTE 2 Viscosity numbers of polyamides not containing additives that interfere with the viscosity measurement can be converted from one solvent to another by a general interconversion formula. Graphs for interconversion are mentioned in Clause 13 and presented in Annex E. The reliability of the conversions is discussed in Annex E.

10 Procedure

10.1 Cleaning of the viscometer

Clean the viscometer (6.8) prior to the first use, again after discordant readings (for example, when two successive determinations of the efflux time of the solvent differ by more than 0,4 s) and, further, at intervals during regular use. For this purpose, allow it to stand for at least 12 h filled with a cleaning agent (5.2), for example chromic acid solution (5.2.1). Remove the cleaning agent, rinse the viscometer with water then with acetone (5.2.2) and dry, for example by a slow stream of filtered air or in the vacuum drying cabinet (6.1).

After each determination, drain the viscometer, rinse with the solvent, then with water, followed by, for example, acetone (5.2.2) and dry as described above.

However, if the next solution to be measured is of a polyamide of the same type and of a similar viscosity, it is permissible to drain the viscometer, wash it with the solution to be measured, and then fill it with this solution.

NOTE In the case of, for example, production control and automated flow time measurement the viscometer can be filled with the solvent in anticipation of the next sample.

10.2 Preparation of test solution

10.2.1 General

Three different methods for preparing the test solution are described in this document. The first volumetric method (see 10.2.2), without correction for the volume of insoluble additives in the test portion, is equal to the method described in the previous version of this document (ISO 307:2007). For practical reasons, test portion masses of (m_c ± 5) mg are allowed. For pure polyamide, this results in a concentration range of 0,004 9 g/ml to 0,005 1 g/ml. The actual polymer concentration is taken into account in the calculation of the viscosity number. For samples containing insoluble additives, a test portion of exactly the calculated mass will give a solution that is almost equal to 0,005 g/ml.

The second volumetric method (see 10.2.3) and the gravimetric method (see 10.2.4) take into account the insoluble additives and the polyamide volume. The latter two methods are often used in combination with (semi-)automatic viscosity measurement equipment.

NOTE For polyamide samples containing only insoluble additives, the concentration of the solution prepared according to the volumetric or gravimetric method will be exactly 5 mg/ml.

10.2.2 Volumetric method

Weigh, to the nearest 0,2 mg, a test portion of mass m_t mg, where m_t lies in the range (m_c ± 5) mg where m_c is the mass calculated in accordance with Formula (1), working rapidly to minimize moisture pick-up by the polymer. If the weighing takes more than 2 min, reject the material and begin another weighing.

Transfer the test portion to the 50 ml volumetric flask (6.3) and add about 40 ml of the solvent (see Clause 9). Close the flask and shake the contents, or stir with the magnetic stirrer (6.6), until the polymer has dissolved. This may take from approximately 0,5 h to several hours, depending on the type of polyamide and the particle size of the test portion. When sulfuric acid or formic acid solution is used as the solvent, the temperature shall not exceed 30 °C. When *m*-cresol or phenol/1,1,2,2-tetrachloroethane is used as the solvent, the temperature may be raised to 95 °C to 100 °C. If, in the latter case, dissolution takes more than 2 h, this shall be reported. For PA 6T/66, suitable conditions have been found to be 2 h at 90 °C.

When dissolution is complete, cool the solution to 25 °C ± 2 °C, dilute to the mark with the solvent and mix well. If the magnetic stirrer (6.6) is used, remove it from the solution before dilution and rinse it with the solvent, adding the rinsings to the flask before further dilution.

10.2.3 Volumetric method, in exact relation to the polymer content

Weigh, to the nearest 0,2 mg, a test portion of mass m_t mg, where m_t lies in the range (m_c ± 10 %) mg, where m_c is the mass calculated in accordance with Formula (1), working rapidly to minimize moisture pick-up by the polymer. If the weighing takes more than 2 min, reject the material and begin another weighing.

Transfer the test portion to the 100 ml volumetric flask (6.3) or weighing bottle (6.4) and add the volume of the solvent (see Clause 9) required to prepare a concentration of 0,50 g of sample per 100 ml of solution. The volume of solvent to be added is corrected for the volume of the soluble mass of the sample. The solvent shall be added by means of a suitable dosing device (e.g. a burette accurate to 0,01 ml). Close the flask and shake the contents, or stir with the magnetic stirrer (6.6), until the polymer has dissolved. This may take from approximately 0,5 h to several hours, depending on the type of polyamide and the particle size of the test portion. When sulfuric acid or formic acid solution is used as the solvent, the temperature shall not exceed 30 °C. When *m*-cresol or phenol/1,1,2,2-tetrachloroethane is used as the solvent, the temperature may be raised to 95 °C to 100 °C. If, in the latter case, dissolution takes more than 2 h, this shall be reported. For PA 6T/66, suitable conditions have been found to be 2 h at 90 °C. When dissolution is complete, cool the solution to 25 °C ± 2 °C.

EXAMPLE

Mass of polyamide in the sample	275 mg
Polyamide density	1,130 0 kg/dm^3
Volume of the polyamide mass	0,275 g/1,130 0 g/ml = 0,243 4 ml
Volume of solvent to be added	(275/250) × 50 − 0,243 4 = 54,76 ml

10.2.4 Gravimetric method, in exact relation to the polymer content

Weigh, to the nearest 0,2 mg, a test portion of mass m_t mg, where m_t lies in the range (m_c ± 10 %) mg, where m_c is the mass calculated in accordance with Formula (1), working rapidly to minimize moisture pick-up by the polymer. If the weighing takes more than 2 min, reject the material and begin another weighing.

Transfer the test portion to a 100 ml volumetric flask (6.3) or weighing bottle (6.4) and add the mass of solvent (see Clause 9) required to prepare a concentration of 0,50 g of sample per 100 ml of solution. The mass of solvent to be added is corrected for the volume of the soluble mass of the sample. Close the flask and shake the contents, or stir with the magnetic stirrer (6.6), until the polymer has dissolved. This may take from approximately 0,5 h to several hours, depending on the type of polyamide and the

particle size of the test portion. When sulfuric acid or formic acid solution is used as the solvent, the temperature shall not exceed 30 °C. When *m*-cresol or phenol/1,1,2,2-tetrachloroethane is used as the solvent, the temperature may be raised to 95 °C to 100 °C. If, in the latter case, dissolution takes more than 2 h, this shall be reported. For PA 6T/66, suitable conditions have been found to be 2 h at 90 °C. When dissolution is complete, cool the solution to 25 °C ± 2 °C.

EXAMPLE

Mass of polyamide in the sample	275 mg
Density of polyamide	1,130 0 kg/dm^3
Density of solvent	1,204 4 kg/dm^3
Volume of the polyamide mass	0,275 g/1,130 0 g/ml = 0,243 4 ml
Volume of solvent to be added	(275/250) × 50 − 0,243 4 = 54,76 ml
Mass of solvent to be added	54,76 ml × 1,204 4 = 65,95 g

NOTE 1 An automated weighing system for preparing the sample solution is often used in production control.

NOTE 2 In the case of polyamides with extremely high relative molecular masses, solutions free from the so-called streaking phenomenon cannot always be obtained, in spite of prolonged periods of shaking or stirring. Such test solutions can only be used for mutual comparison with similar products.

10.3 Measurement of flow times

Determine the mean flow time of the solvent in the same viscometer and in the same manner as that of the solution. The flow times of the solvents shall be determined at least once each day that they are used, or at a different frequency if statistically validated by the laboratory. If the flow time of a solvent differs by more than 0,5 % from the initial value at the time of preparation, the solvent shall be discarded and fresh solvent prepared.

Filter the solution through the sintered-glass filter (6.7) or the metal sieve into tube L of the viscometer (see Figure 1). Alternatively, centrifuge the solution at a rotational frequency of approximately 50 s^{-1} and pour the clear supernatant liquid into the viscometer (6.8). The volume of liquid shall be such that, after draining, the level lies between the filling marks. For hand-filled viscometers, the filling should preferably be done with the viscometer out of the thermostatic bath (6.10) to avoid contamination of the bath in case of accidental spills.

Mount the viscometer in the thermostatic bath maintained at 25,00 °C ± 0,05 °C ensuring that tube N is vertical and that the upper graduation mark E is at least 30 mm below the surface of the liquid in the bath. Allow at least 15 min for the charged viscometer to attain the temperature of the bath.

Close tube M and blow or draw the liquid into the upper bulb of tube N using a rubber bulb or similar equipment. Close tube N. Open tube M so that the liquid drops away from the lower end of the capillary tube. Open tube N and measure the flow time, to the nearest 0,2 s, as the time taken for the bottom of the meniscus to pass from mark E to mark F. With cloudy solutions, view the top of the meniscus. Repeat the measurement of the flow time until two successive values agree within 0,25 %. Take the mean of these two values as the flow time of the solution.

With each polyamide sample, carry out at least two determinations of the viscosity number, using a fresh solution each time, until two successive values meet the repeatability requirement corresponding to the solvent used (see Clause 12). Report the mean of these two values, rounded off to the nearest whole number, as the viscosity number of the sample. If two successive determinations of the solution mean flow time differ by more than 0,5 %, clean the viscometer (see 10.1).

When necessary, filter the solution of the sample through a filter (6.7) before making the measurement. Any glass fibres contained in the sample will sediment completely after 3 h to 4 h. In such cases, the test solution can be decanted for the measurement and thus does not need to be filtered.

For automated solution-viscosity-measurement equipment, the procedure may differ from the described method. However, the requirements concerning the measurement solution, the equipment, bath temperature and the flow times shall be maintained in all respects.

For production quality control purposes, a single viscosity number determination is, in principle, permitted on condition that the precision of the test method is known and permits the process variation to be identified (precision <30 % of the process variation, preferably <10 %), and the process control limits and the process warning limits are determined, based on a known precision interval, and statistically validated. It is strongly recommended that the statistical model is incorporated in the quality manual, including written procedures on how to handle the product and the actions that should be taken if the result of the single determination is outside the warning limits but within the action limits or is outside the action limits. These procedures may comprise activities such as

— repeating the measurement (e.g. twofold),

— in the case of random production control, measurement of the homogenized batch, or

— acceptance of the product based on larger process limits.

A general applicable statistical model is described in ISO 25337.

11 Expression of results

Calculate the concentration of the polymer, in grams per millilitre, according to Formula (2):

$$C = \frac{m_t}{1\,000 \times 50 \times \left(\frac{100}{100 - (w_1 + w_2 + w_3)} \right)} \tag{2}$$

where

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

m_t is the mass of the test portion taken in 10.2.2, 10.2.3 or 10.2.4, in milligrams;

w_1, w_2 and w_3 are as defined in Formula (1).

Calculate the viscosity number VN, in millilitres per gram, according to Formula (3).

In calculating the viscosity number of the polymer, it is assumed that the ratio of the viscosities of the solution and the solvent may be replaced by the ratio of the corresponding times of flow. Furthermore, the concentration of the polymer may be expressed as grams per millilitre of solvent instead of grams per millilitre of solution without introducing appreciable error.

With kinetic energy correction:

$$\mathrm{VN} = \left(\frac{t - t_c}{t_0 - t_{0c}} - 1 \right) \frac{1}{C} \tag{3}$$

where

t is the flow time of the solution, in seconds;

t_c is the Hagenbach correction of the solution, in seconds, if applicable;

t_0 is the flow time of the solvent, in seconds;

t_{0c} is the Hagenbach correction of the solvent, in seconds, if applicable;

C is the concentration of the polymer, in grams per millilitre of solution.

NOTE In the case of (automated) sample preparation in relation with the exact polymer content, the relative viscosity is also suitable for production control purposes.

12 Repeatability and reproducibility

The precision of this test method is not known because interlaboratory data are currently not available.

Information on reproducibility and repeatability is given in Table EX1.

13 Relationship between the viscosity number determined in 96 % (by mass) sulfuric acid solution and the viscosity determined in various solvents

The following interconversions of viscosity numbers determined in different solvents and presented in Annex E are only meant as guidance for pure polyamides (PAs). The interconversions are intended to be excluded at the next revision of this document.

— Interconversion of viscosity number in 96 % (by mass) sulfuric acid and 90 % (by mass) formic acid for PA 6, PA 66, PA 69 and PA 610.

— Interconversion of viscosity number in 96 % (by mass) sulfuric acid and *m*-cresol for PA 612.

— Interconversion of relative viscosity determined in accordance with ASTM D789 and viscosity number determined in 96 % (by mass) sulfuric acid (according to this document) for PA 6 and PA 66.

— Interconversion of relative viscosity determined in 98 % (by mass) sulfuric acid solution according to Annex JA of JIS K 6920-2:2009 and the viscosity number in 96 % (by mass) sulfuric acid (according to this document) for PA 6 and PA 66.

— Interconversion of relative viscosity in 95,7 % (by mass) sulfuric acid at a concentration of 0,01 g/ml and the viscosity number in 96 % (by mass) sulfuric acid (according to this document) for PA 6 and PA 66.

14 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 307:2019;

b) complete identification of the material tested, including any preparation steps;

c) the solvent used;

d) if longer than 2 h, the time required to dissolve a sample in *m*-cresol at 95 °C to 100 °C;

e) the viscosity number (individual values and arithmetic mean of the two determinations);

f) if present, the method for determination of additives;

g) any deviations from the method specified in this document.

Annex A
(informative)

Determination of the concentration of commercial sulfuric acid(95 % to 98 %) and adjustment to 96 % by titration

A.1 General

The concentration of commercially available sulfuric acid is between 95 % and 98 %. The concentration of the sulfuric acid is adjusted to 96 % ± 0,20 %. The sulfuric acid concentration is determined by titration.

A.2 Apparatus and reagents

A.2.1 Calibrated burette, 50 ml or, preferably, 100 ml, measurement accuracy 0,1 % or better.

A.2.2 Sodium hydroxide, standard volumetric aqueous solution, c(NaOH) = 1 mol/l, preferably commercial ready-to-use solutions or solutions made up from ampoules.

A.2.3 Hydrochloric acid, standard volumetric aqueous solution, c(HCl) = 1 mol/l, preferably commercial ready-to-use solutions or solutions made up from ampoules.

A.2.4 Tris(hydroxymethyl)amino methane, ≥99,5 % (by mass), certified standard titrimetric reference substance.

A.2.5 Anhydrous sodium carbonate, ≥99,5 % (by mass), certified standard titrimetric reference substance, alternative for tris(hydroxymethyl)amino methane, ≥99,5 % (by mass).

A.2.6 Phenolphthalein, 0,1 % (by mass) solution in methanol.

A.3 Procedure

A.3.1 Check on the titre of hydrochloric acid

The titre of hydrochloric acid should be checked by titration of, preferably, tris(hydroxy)aminomethane or anhydrous sodium carbonate. The volume of hydrochloric acid used shall be approximately 20 ml to 25 ml.

A.3.2 Preparation of sodium hydroxide solution, 1 mol/l

Decarbonate distilled water by bubbling nitrogen into boiling water in a flask provided with a reflux condenser for at least 2 h. The sodium hydroxide solution prepared from a 1 mol/l ampoule shall be diluted with the decarbonated distilled water and stored in a bottle protected from carbon dioxide.

When preparing the sodium hydroxide solution from solid sodium hydroxide, decarbonate the sodium hydroxide solution by, for example, removing carbonate by precipitation with a slight excess of barium chloride followed by filtration. A portion of the clear solution may be diluted to the desired volume.

Sodium hydroxide may, for example, be freed from carbonate by preparing and filtering a 1:1 (by mass) water solution in which sodium carbonate is almost insoluble. The sodium hydroxide 1 mol/l solutions

can be prepared by diluting the 1:1 (by mass) solution with decarbonated distilled water. For other methods, the user is referred to books on quantitative analysis such as References [1] and [2].

A.3.3 Determination of the titre of sodium hydroxide solution

The titre of the sodium hydroxide solution shall be determined with hydrochloric acid. The titre of sodium hydroxide of three successive determinations shall not differ by more than 0,02 % from each other. Determine the titre of the sodium hydroxide solution by calculating the mean value of three determinations.

The use of hydrogen potassium phthalate, $C_6H_4COOH(COOK)$, as standard for the determination of the titre of sodium hydroxide is not allowed.

A.3.4 Determination of the titre of initial sulfuric acid solution

Weigh, to the nearest 0,1 mg, 2 g of the acid into a dry weighing bottle with a ground-glass lid. Pour the acid carefully and very slowly into approximately 50 ml of distilled water in a 250 ml conical flask. Rinse the weighing bottle with distilled water, adding the rinsings to the solution in the conical flask, to give a total volume of about 100 ml. Then titrate with the sodium hydroxide solution (A.2.2), using phenolphthalein (A.2.6) as indicator, until the first recognizable, permanent pink coloration is detected (the volume of sodium hydroxide will be approximately 40 ml).

Carry out at least three determinations. Calculate the arithmetic mean of the results. The individual test results shall not differ by more than 0,15 % of their mean.

Add a few drops of phenolphthalein to the water as a check on rinsing the weighing bottle and transferring all the sulfuric acid to the conical flask.

A potentiometric titration may also be used. The titration should preferably be carried out under N_2 to avoid the influence of CO_2.

A.4 Expression of results

Calculate the titre, as a percentage by mass, of the sulfuric acid, using Formula (A.1):

$$n = \frac{m_s \times V}{m} \tag{A.1}$$

where

V is the volume, in millilitres, of sodium hydroxide solution used;

m is the mass, in grams, of the test portion of sulfuric acid;

m_s is the mass, in milligrams, of sulfuric acid, 4,903 9 mg, corresponding to 0,10 ml of 1 mol/l sodium hydroxide solution;

n is the titre, as a percentage by mass, of the sulfuric acid solution.

A.5 Adjustment of sulfuric acid concentration

A.5.1 Concentration of sulfuric acid solution lower than 96 %

Mix the initial sulfuric acid with a higher concentrated sulfuric acid of previously determined concentration, in a ratio calculated from the value of the concentration of the two solutions, to the required 96,00 % ± 0,20 %. Determine the concentration of the mixed sulfuric acid.

A.5.2 Concentration of sulfuric acid solution higher than 96 %

Mix the initial sulfuric acid with a less concentrated sulfuric acid, of previously determined concentration, in a ratio calculated from the value of the concentration of the two solutions, to the required 96,00 % ± 0,20 %. Determine the concentration of the mixed sulfuric acid.

CAUTION — The diluting of concentrated sulfuric acid with water is discouraged for the following reasons. The quantity of water needed is very small and will often lead to dilution errors. For safety reasons, concentrated sulfuric acid should never be diluted by adding water but always by adding sulfuric acid to water. The latter is not practical for diluting concentrated sulfuric acid to somewhat less concentrated sulfuric acid.

Annex B
(informative)

Determination of the concentration of sulfuric acid (95 % to 98 %) and adjustment to 96 % by flow time measurement in a small capillary viscometer

B.1 General

The concentration of commercially available sulfuric acid is between 95 % and 98 %. The concentration of the sulfuric acid is adjusted to 96 % ± 0,20 %. The sulfuric acid concentration is determined by flow time measurement in a small capillary viscometer.

B.2 Apparatus

B.2.1 Ubbelohde viscometer, constant approximately 0,005 mm^2/s^2, d = 0,46 mm.

B.2.2 Ultra-thermostatic bath, capable of being maintained at 25 °C ± 0,03 °C.

B.2.3 Thermometer, accurate to 0,01 °C.

B.2.4 Calibrated burette, 50 ml or, preferably, 100 ml, with measurement accuracy 0,1 % or better.

B.3 Preparation of calibration curve

Prepare at least five sulfuric acid solutions with approximately the following nominal concentrations: 95,50 %; 95,75 %; 96,00 %; 96,25 %; 96,50 %. Determine their exact concentrations by titration according to the method prescribed in Annex A. The titration should be carried out in, at least, three-fold for each concentration.

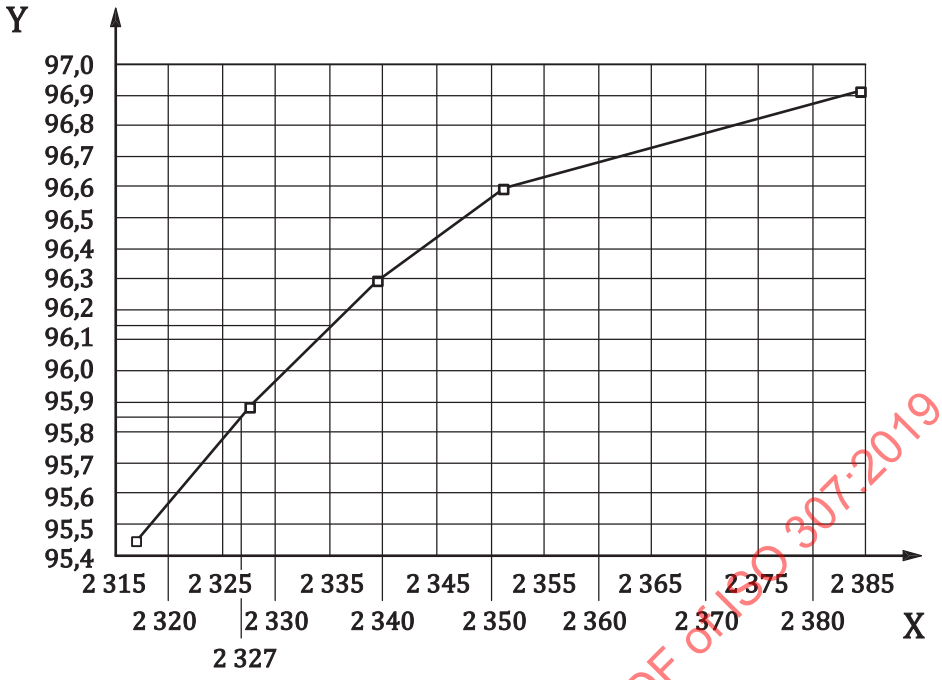
Determine the flow time for each concentration in the viscometer in, at least, three-fold. Calculate the arithmetic mean of the results. The individual test results shall not differ by more than 2 s of their mean.

An example of a calibration curve is given in Table B.1 and Figure B.1.

Table B.1 — Example of a calibration curve of concentration of sulfuric acid as a function of flow time

Nominal concentration % (by mass)	Flow time s	Average s	Sulfuric acid conc. % (by mass)	Average %
95,75	2 316; 2 317; 2 318	2 317,0	95,38; 95,52; 95,46	95,453
96,00	2 329; 2 327; 2 328	2 328,0	95,93; 95,91; 95,81	95,883
96,25	2 339; 2 339; 2 340	2 339,3	96,24; 96,35; 96,29	96,293
96,50	2 350; 2 353; 2 351	2 351,3	96,46; 96,60; 96,73	96,596
96,75	2 385; 2 384; 2 384	2 384,3	96,88; 96,89; 96,97	96,913

NOTE The standard deviation of the determination of the sulfuric acid content by flow time measurement is expected to be 0,061 % (absolute) sulfuric acid.



Key

X flow time, in seconds

Y concentration, in % (by mass)

Figure B.1 — Example of flow time/concentration relationship

B.4 Adjustment of sulfuric acid concentration

B.4.1 Concentration of sulfuric acid solution lower than 96 %

Mix the initial sulfuric acid with a more concentrated sulfuric acid of previously determined concentration, in a ratio calculated from the value of the concentration of the two solutions, to the required 96,00 % ± 0,20 %. Determine the concentration of the mixed sulfuric acid.

B.4.2 Concentration of sulfuric acid solution higher than 96 %

Mix the initial sulfuric acid with a less concentrated sulfuric acid, of previously determined concentration, in a ratio calculated from the value of the concentration of the two solutions, to the required 96,00 % ± 0,20 %. Determine the concentration of the mixed sulfuric acid.

CAUTION — The diluting of concentrated sulfuric acid with water is discouraged for the following reasons. The quantity of water needed is very small and will often lead to dilution errors. For safety reasons, concentrated sulfuric acid should never be diluted by adding water but always by adding sulfuric acid to water. The latter is not practical for diluting concentrated sulfuric acid to somewhat less concentrated sulfuric acid.

Annex C
(informative)

Determination of the concentration of commercial formic acid and adjustment to 90 % by titration

C.1 General

The concentration of commercially available formic acid is approximately between 98 % and 100 %. The concentration is adjusted to 90 % ± 0,15 %.

The formic acid concentration is determined by potentiometric titration.

C.2 Apparatus and reagents

C.2.1 Potentiometric titration equipment, with combined glass electrode.

C.2.2 Sodium hydroxide, standard volumetric aqueous solution, c(NaOH) = 1 mol/l (e.g. made up from ampoules).

C.2.3 Decarbonated water, max. conductivity of 0,056 µS/cm at 298 K.

C.2.4 Potassium hydrogen phthalate.

C.2.5 Calibrated burette, 50 ml or, preferably, 100 ml, with measurement accuracy 0,1 % or better.

C.3 Procedure

C.3.1 Determination of the titre of the sodium hydroxide solution

Weigh, to the nearest 0,000 1 g, approximately 1,5 g of potassium hydrogen phthalate and dissolve in 80 ml of water. Titrate the solution at 20 °C with the sodium hydroxide solution. Determine the titre of the sodium hydroxide solution by calculating the mean value of five determinations according to Formula (C.1).

$$n = \frac{m_t}{204{,}23 \times v_1 \times 0{,}001} \qquad \text{(C.1)}$$

where

m_t is the mass, in grams, of the test portion of potassium hydrogen phthalate used;

204,23 is the molar mass, in g/mol, of potassium hydrogen phthalate;

v_1 is the volume, in millilitres, of sodium hydroxide solution used;

0,001 is a factor to convert millilitres to litres;

n is the titre of the sodium hydroxide solution, expressed in mol/l.

The titration should preferably be carried out under N_2 to avoid the influence of CO_2.

C.3.2 Determination of the formic acid concentration

Weigh, to the nearest to 0,000 1 g, approximately 0,6 g of formic acid and dissolve it in approximately 80 ml of water. Titrate the solution at 20 °C with the sodium hydroxide solution. Determine the formic acid concentration by calculating the mean value of three determinations according to Formula (C.2):

$$c = \frac{v_2 \times 0{,}001 \times n \times 46{,}03}{m_F} \times 100\,\% \qquad \text{(C.2)}$$

where

v_2 is the volume, in millilitres, of sodium hydroxide solution used;

0,001 is a factor to convert millilitres to litres;

n is the titre of the sodium hydroxide solution, expressed in mol/l;

46,03 is the molecular mass, in g/mol, of formic acid;

m_F is the mass, in grams, of formic acid used;

c is the formic acid concentration, in % (by mass).

NOTE The standard deviation of the determination of the formic acid content by titration is expected to be <0,05 % absolute. For a determination in three-fold, the standard deviation is expected to be <0,03 % (absolute).

C.4 Adjustment of formic acid concentration

If the formic acid concentration is too high, carefully mix the acid with distilled water to give the required concentration. The acid should always be run very slowly into the distilled water. If the concentration is too low, mix the acid with acid of a higher concentration.

Annex D
(informative)

Determination of the concentration of commercial formic acid and adjustment to 90 % by density measurement

D.1 General

The concentration of commercially available formic acid is approximately between 98 % and 100 %. The concentration is adjusted to 90 % ± 0,15 %. The formic acid concentration is determined by density measurement.

D.2 Apparatus

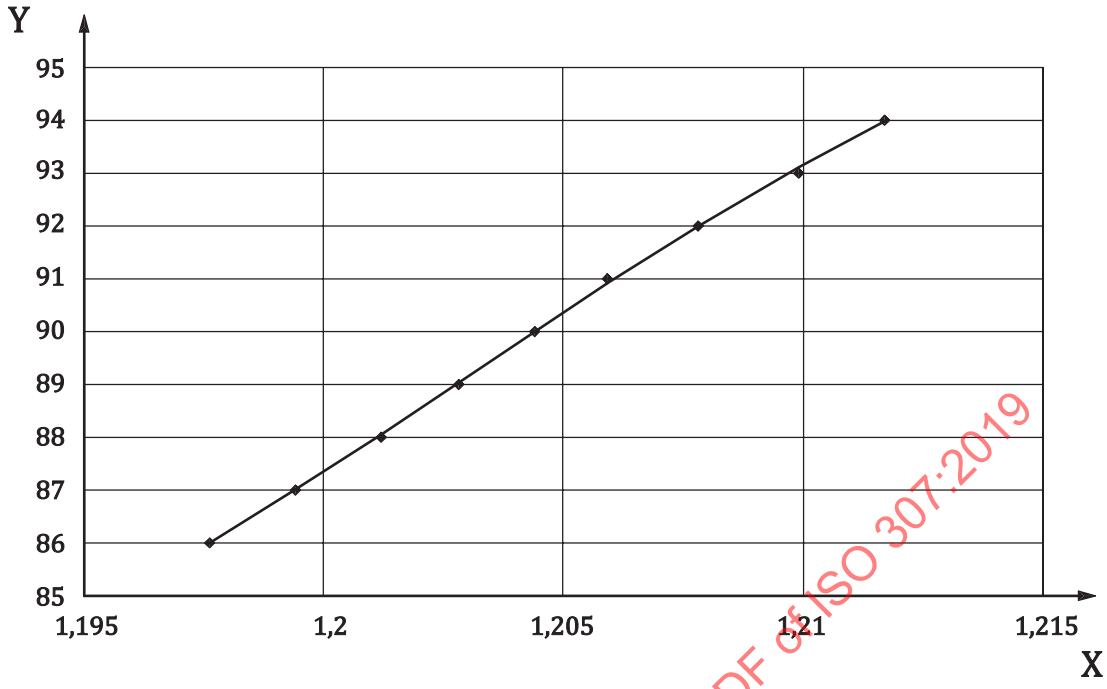
D.2.1 Density measurement equipment, accurate to 0,000 01 kg/m^3, e.g. measurement equipment based on the so-called "oscillating U-tube principle".

D.3 Procedure

Determine the density of the formic acid at 20 °C to at least five decimal places. Determine the concentration according to Table D.1.

If the formic acid concentration is too high, carefully mix the acid with distilled water to give the required concentration. The acid should always be run very slowly into the distilled water. If the concentration is too low, mix the acid with acid of a higher concentration.

NOTE The standard deviation of the determination of the formic acid content by density is expected to be <0,01 % absolute.



Key

X density at 20 °C

Y formic acid concentration, % (by mass) (from Reference [3])

Figure D.1 — Concentration of formic acid as a function of density

Table D.1 — Density of formic acid (kg/dm³) at 20 °C as a function of the formic acid concentration (curve-fitting data)

Density kg/dm³	Formic acid concentration[a]	Formic acid concentration Curve-fitting data % (by mass)	Density kg/dm³	Formic acid concentration[a]	Formic acid concentration Curve-fitting data % (by mass)
1,202 80	89	89,017 53	1,204 46		90,008 22
1,203 60		89,494 86	1,204 48		90,020 15
1,203 78		89,602 37	1,204 50		90,032 07
1,203 96		89,709 86	1,204 52		90,043 99
1,204 12		89,805 39	1,204 54		90,055 90
1,204 14		89,817 33	1,204 56		90,067 82
1,204 16		89,829 27	1,204 58		90,079 74
1,204 18		89,841 20	1,204 60		90,091 65
1,204 20		89,853 14	1,204 62	90,1	90,103 56
1,204 22		89,865 07	1,204 64		90,115 48
1,204 24		89,877 01	1,204 66		90,127 39
1,204 26		89,888 94	1,204 68		90,139 30
1,204 28	89,9	89,900 87	1,204 70		90,151 20
1,204 30		89,912 81	1,204 72		90,163 11
1,204 32		89,924 74	1,204 74		90,175 01
1,204 34		89,936 67	1,204 76		90,186 91
1,204 36		89,948 59	1,204 78		90,198 81
1,204 38		89,960 52	1,204 96		90,305 84
1,204 40	90	89,972 45	1,205 12		90,400 84
1,204 42		89,984 37	1,205 28		90,495 70
1,204 44		89,996 30	1,205 90	91	90,861 65

[a] From Reference [3].

Annex E
(informative)

Relationship between the viscosity number determined in 96 % (by mass) sulfuric acid solution and the viscosity determined in various solvents

E.1 Relationship between the viscosity numbers determined in 96 % (by mass) sulfuric acid, 90 % (by mass) formic acid and *m*-cresol

E.1.1 Viscosity numbers in 96 % (by mass) sulfuric acid and 90 % (by mass) formic acid, respectively

Graphs of the relationship between the viscosity numbers of PA 6, PA 66, PA 69 and PA 610 determined in sulfuric acid solution (5.1.1) and in formic acid solution (5.1.2) are presented in Figure E.1.

The value measured by one laboratory in one of the solvents will, in general, differ from the value obtained by conversion from a measurement by another laboratory in the other solvent. The 95 % confidence intervals for this difference, as percentages of the converted values, are

— for PA 6 ±9 %

— for PA 66 ±9 %

— for PA 69 ±10 %

— for PA 610 ±14 %

E.1.2 Viscosity numbers in 96 % (by mass) sulfuric acid and *m*-cresol, respectively

A graph of the relationship between the viscosity numbers of PA 612 in sulfuric acid solution (5.1.1) and in *m*-cresol (5.1.3) is presented in Figure E.2.

For PA 612, the 95 % confidence interval for the difference between an actually measured value and a converted value depends on the direction of the conversion. The intervals, as percentages of the converted values, are

— value in *m*-cresol converted to value in 96 % (by mass) sulfuric acid ±17 %

— value in 96 % (by mass) sulfuric acid converted to value in *m*-cresol ±9 %

E.1.3 Precision

The relationship between the viscosity numbers determined in different solvents was determined in the interlaboratory investigation referred to in the Note to Table E.1.

Table E.1 — Repeatability and reproducibility

Solvent	Repeatability %	Reproducibility %
Sulfuric acid solution (5.1.1)	2	5
Formic acid solution (5.1.2)	2	10
m-Cresol (5.1.3)	3	10
NOTE The repeatabilities and reproducibilities were determined in an interlaboratory investigation carried out in 1982. Seven laboratories participated in this investigation. The programme included 11 samples of PA 6, 9 of PA 66, 3 of PA 69, 4 of PA 610, 5 of PA 612 and 2 of PA 6T/66. The viscosity numbers of the samples were determined in duplicate in both solvents in accordance with this document.		

E.2 Relative viscosities determined in accordance with ASTM D789 and viscosity numbers determined in 96 % (by mass) sulfuric acid

A conversion table and a graph of the relationship between the relative viscosities of PA 6 and PA 66 and the viscosity numbers of PA 6 and PA 66 in sulfuric acid solution (5.1.1) are presented in Table E.2 and Figure E.3, respectively.

The precision of the relationship between the relative viscosities of PA 6 and PA 66 and the viscosity numbers of PA 6 and PA 66 in 96 % (by mass) sulfuric acid solution is not known because interlaboratory data are not available. The relationship given in Table E.2 and Figure E.3, respectively, is only given as an indication of what relation to expect.

E.3 Interconversion of relative viscosity (JIS K 6920-2:2009, Annex JA) and viscosity number for PA 6 and PA 66 (this document)

A graph of the relationship between the relative viscosity of PA 6 and PA 66 in 98 % (by mass) sulfuric acid solution and the viscosity number in 96 % (by mass) sulfuric acid solution (5.1.1.) is presented in Figure E.4.

Interlaboratory testing was carried out by 8 laboratories in 1999 to check the interconvertibility of viscosity of PA 6 and PA 66 between the relative viscosity in 98 % (by mass) sulfuric acid solution and the viscosity number in 96 % (by mass) sulfuric acid.

The linear relationship as shown in Figure E.4 is described by Formula (E.1):

$$VN = 69{,}771 \times RV - 49{,}372 \quad \text{(E.1)}$$

where

RV is the relative viscosity measured according to JIS K 6920-2:2009, Annex JA;

VN is the viscosity number, in ml/g, measured according to this document.

The linear regression coefficient R^2 = 0,982 3.

The test conditions for measurement of relative viscosity in 98 % (by mass) sulfuric acid solution are as follows.

— solution 98,0 % ± 0,2 % (by mass) sulfuric acid

— polymer concentration (0,250 ± 0,001) g per 25 ml of solvent (0,01 g/ml)

— temperature 25,0 °C ± 0,1 °C

— viscometer Ostwald type

E.4 Interconversion of relative viscosity in 95,7 % (by mass) sulfuric acid at a concentration of 0,01 g/ml and viscosity number for PA 6 and PA 66 (this document)

Two graphs of the relationship between the relative viscosity of PA 6 and PA 66 in 95,7 % (by mass) sulfuric acid at a concentration of 0,01 g/ml and the viscosity number in 96 % (by mass) sulfuric acid solution (5.1.1.) are presented in Figures E.5 and E.6, respectively.

The reproducibility of the relative viscosity determination in 95,7 % (by mass) sulfuric acid was estimated by analysis of the same PA 6 sample once a day for 21 weeks, 5 days a week, with 2 different viscometers and different operators involved. The estimation of the standard deviation s_R was found to be 0,013 3.

Interlaboratory testing was carried out in 1999, with 5 laboratories involved, to determine the interconvertibility of the solution viscosity of PA 6 and PA 66 between the relative viscosity (RV), measured in 95,7 % (by mass) sulfuric acid at a sample concentration of 0,01 g/ml, and the viscosity number (VN) in 96 % (by mass) sulfuric acid.

The viscosity measurements were carried out using the methodology of this document with the following exceptions for measurement of the relative viscosity.

— solution 95,7 % ± 0,2 % (by mass) sulfuric acid

— polymer concentration (0,500 0 ± 0,000 5) per 50 ml of solvent (0,01 g/ml)

— viscometer Ubbelohde type, diameter 1,36 mm

The linear relationship as shown in Figure E.5 is described by Formula (E.2):

$$VN = 77,450\,2 \times RV - 59,194\,7 \quad \text{(E.2)}$$

where

RV is the relative viscosity measured in 95,7 % (by mass) sulfuric acid at a concentration of 0,01 g/ml;

VN is the viscosity number, in ml/g, measured according to this document.

The linear regression coefficient R^2 = 0,998 9.

The linear relationship as shown in Figure E.6 is described by Formula (E.3):

$$VN = 77,573\,9 \times RV - 59,897\,0 \quad \text{(E.3)}$$

where the symbols are as in Formula (E.2).

The linear regression coefficient R^2 = 0,993 6.

Table E.2 — Relative viscosity (RV) and viscosity number (VN) for PA 6 and PA 66 (values taken from curve in Figure E.3)

RV (ASTM D789)	VN (ISO 307) in 96 % (by mass) H_2S0_4
25	83,93
27	90,87
29	97,32
31	103,34
33	108,98
35	114,29
37	119,30
39	124,05
41	128,57
43	132,87
45	136,97
47	140,89
49	144,65
51	148,26
53	151,73
55	155,07
57	158,30
59	161,41
61	164,42
63	167,33
65	170,15
67	172,88
69	175,54
71	178,12
73	180,62
75	183,06
77	185,44
79	187,75
81	190,01
83	192,21
85	194,35
87	196,45
89	198,50
91	200,51
93	202,47
95	204,39
97	206,27
99	208,11
101	209,92

Table E.2 *(continued)*

RV (ASTM D789)	VN (ISO 307) in 96 % (by mass) H_2S0_4
103	211,69
105	213,42
107	215,12
109	216,80
111	218,44
113	220,05
115	221,63
117	223,19
119	224,72
121	226,22
123	227,70
125	229,15
127	230,59
129	232,00
131	233,39
133	234,75
135	236,10
137	237,43
139	238,73
141	240,02
143	241,29
145	242,55
147	243,78
149	245,00
151	246,21
153	247,39
155	248,56
157	249,72
159	250,86
161	251,99
163	253,11
165	254,21
167	255,29
169	256,37
171	257,43
173	258,48
175	259,52
177	260,54
179	261,56
181	262,56
183	263,55
185	264,53