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Plastics — Polyamides — Determination of viscosity number

Plastiques — Polyamides — Détermination de l'indice de viscosité

Reference number
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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 fourth edition cancels and replaces the third edition (ISO 307:1994), which has been technically revised.

Plastics — Polyamides — Determination of viscosity number

1 Scope

This International Standard specifies a method for the determination of the viscosity number (also referred to as reduced viscosity) 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 and PA MXD6 as defined in ISO 1874-1, as well as to copolyamides 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 crosslinking agents; such polyamides are normally insoluble in the specified solvents.

In general, polyamide samples have to be completely soluble in the solvents mentioned, and the additives contained in them (e.g. reinforcement fibres, flame-retardants and modifiers) must not interfere with the viscosity measurement. However, the method can be used for production control of compounds containing additives which interfere with the viscosity measurement. In such cases, a particular compound in a particular solvent will have a specific viscosity number which, in principle, cannot be converted from one solvent to another except by means of a relationship specific to the compound concerned. Viscosity numbers converted in this way are only suitable for inter-product comparisons.

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

The determination of the viscosity number of a polyamide provides a measure of the relative molecular mass of the polymer.

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 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 1628-1:1998, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 1874-1:1992, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 1: Designation*

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 3451-4:1998, *Plastics — Determination of ash — Part 4: Polyamides*

ISO 6427:1992, *Plastics — Determination of matter extractable by organic solvents (conventional methods)*

ISO 15512:1999, *Plastics — Determination of water content*

ASTM D 789-98, *Standard Test Methods for Determination of Relative Viscosity and Moisture Content 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
VN
⟨of a polymer⟩ the value given by the equation

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

where

η is the viscosity of a solution of the polymer in a specified solvent;

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

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

NOTE The viscosity number is usually expressed in millilitres per gram.

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

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

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

5.1 Solvents

5.1.1 Sulfuric acid, (96 ± 0,15) % (by mass) solution.

For the determination of the concentration of commercial sulfuric acid [95 % (by mass) to 97 % (by mass)] and adjustment to 96,0 % (by mass), see Annex A.

5.1.2 Formic acid, (90 ± 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 % (by mass) of acetic acid or methyl formate.

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 can be obtained by distillation of chemically pure *m*-cresol, preferably *in vacuo*.

To avoid oxidation, nitrogen should preferably be used for pressure compensation. The purity of the solvent may be checked by gas chromatography. The solvent shall be stored in a brown glass bottle.

5.2 Cleaning liquids

5.2.1 **Chromic acid solution**, prepared by mixing equal volumes of sulfuric acid (ρ = 1,84 g/ml) and a saturated solution of potassium dichromate. If required, the chromic acid solution may be replaced by other, equally effective cleaning liquids.

5.2.2 **Acetone**, freshly distilled.

6 Apparatus

6.1 **Vacuum drying cabinet**, pressure less than 100 kPa.

6.2 **Balance**, accurate to 0,1 mg.

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

6.4 **Shaking apparatus** or **magnetic stirrer**.

6.5 **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.

6.6 **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 equivalent to those of the Ubbelohde viscometer specified above. However, in cases of dispute, an Ubbelohde viscometer shall be used.

6.7 **Thermostatic bath**, capable of being maintained at 25 °C ± 0,05 °C.

6.8 **Stop-watch**, accurate to 0,1 s.

6.9 **Centrifuge**.

7 Preparation of test samples

7.1 General

Polyamide test samples for the determination of the viscosity number shall be completely soluble in the solvents mentioned, and the additives contained in them (reinforcement fibres, flame-retardants and modifiers) shall not interfere with the viscosity measurement, or it shall be possible to separate the additives quantitatively from the solution as with glass and carbon fibres, for instance.

If the content of these additives is greater than 2 % (by mass), it shall be determined quantitatively to allow the exact test portion size to be calculated (see Table 1).

Exception: an extracted sample if it contains extractable additives (see 7.2).

Table 1 — Determination of additives in polyamide samples in order to be able to calculate the correct test portion size

Property determined	Test method	Additives determinable	Remarks	See Subclause
Extractable-matter content	ISO 6427	Low-molecular-mass fractions Plasticizers Stabilizers Slip agents	Extract sample before measurement (thus no correction necessary to size of test portion)	7.2
Residue on hydrolysis in dilute hydrochloric acid (no ash)	—	Polymer modifiers (e.g. ethylene and E/P copolymers) Carbon fibres Organic flame-retardants	Check solubility (if necessary, also determine ash and residue on dissolution)	7.3
Residue in 90 % (by mass) formic acid solution (no ash)	—	Carbonates Metal powders Pigments Organic flame-retardants		7.4
Ash (no oxide conversion)	ISO 3451-4	Glass fibres Silicates Inorganic flame-retardants	If necessary, increase size of test portion	7.5

7.2 Samples with extractable ingredients

The ground polymer remaining after extraction in accordance with ISO 6427 shall be dried by heating for 3 h at 100 °C under vacuum and used as the test sample.

7.3 Samples with auxiliaries (non-hydrolysable and without ash) that are insoluble in hydrochloric acid, e.g. polymer modifiers, carbon fibres and certain flame-retardants

Preliminary tests shall be carried out to determine whether the sample is completely soluble in the solvent to be used, i.e. that it does not form particles of gel.

The fraction of polymer modifier and other additives that are insoluble in hydrochloric acid is determined from the hydrolysis residue and the correct amount of polyamide sample to be weighed out is calculated using Equation (3).

If carbon fibres that can be detected in the hydrolysis residue under an optical microscope are present, they shall be filtered off from the solution of the polyamide sample by means of a filter crucible with a glass frit.

7.4 Samples with auxiliaries that are soluble in dilute hydrochloric acid and/or cannot be determined from the ash owing to oxide formation, e.g. carbonates, metal powders and certain pigments

The residue on dissolution in 90 % (by mass) formic acid is determined as follows:

Shake or stir the polyamide sample for several hours at room temperature in about 10 times its own volume of 90 % (by mass) formic acid using the procedure described in 10.2.

Separate off the solid residue by filtration.

Carefully wash the residue with 90 % (by mass) formic acid and then with acetone. Subsequently dry for 3 h at 100 °C under a vacuum. Weigh.

7.5 Samples with auxiliaries with determinable ash, e.g. glass fibres and silicates

The ash is determined by the method specified in ISO 3451-4, and the amount of polyamide sample to be weighed out is calculated using Equation (3). When necessary, filter the solution of the sample through a filter crucible with a glass frit before making the measurement.

NOTE Any glass fibres of the usual dimensions 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.

8 Calculation of mass of test portion

8.1 Polyamides with extractable-matter content not greater than 2,00 % (by mass)

Calculate the mass m_c, in milligrams, of the test portion as follows:

$$m_c = \frac{250}{1 - \frac{w_1 + w_2 + w_3}{100}} \tag{2}$$

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) of 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.

The corrections for w_1, w_2 and w_3 need only be applied if they exceed 0,5 % (by mass) each.

8.2 Polyamides with extractable-matter content greater than 2,00 % (by mass)

Use the material extracted and dried in accordance with 7.2 as the test sample. Calculate the mass m_c, in milligrams, of the test portion as follows:

$$m_\mathrm{c} = \frac{250}{1 - \dfrac{w_2 + w_3}{100 - w_4}} \qquad (3)$$

where

m_c, w_2 and w_3 have the same meanings as in 8.1;

w_4 is the extractable-matter content of the sample, expressed as a percentage by mass.

It has been assumed in this calculation that w_2 and w_3 have been determined on the unextracted sample and that the extracted and dried material will be kept dry, so that no correction for moisture in the test sample is required.

NOTE When dissolved in 50 ml of solvent, a test portion weighing exactly m_c mg will give a solution containing (almost) exactly 5 mg of polymer per millilitre. For practical reasons, test portion masses of ($m_\mathrm{c} \pm 5$) mg are allowed. The resulting actual polymer concentration is taken into account in the calculation of the viscosity number.

9 Selection of solvent

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

Three different solvents are described in this International Standard: sulfuric acid solution (5.1.1), formic acid solution (5.1.2) and *m*-cresol (5.1.3). The flow times of the solvents shall be determined at least once each day that they are used (see 10.3). If the flow time of a solvent differs by more than 1 % from the initial value at the time of preparation, the solvent shall be discarded and fresh solvent prepared.

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

9.2 For PA 6, PA 46, PA 66, PA 69, PA 610, PA MXD6 and corresponding copolyamides, sulfuric acid or formic acid solution shall be used as solvent, unless these polyamides contain additives that liberate gases in acidic solvents, in which case *m*-cresol shall be used as the solvent.

NOTE Graphs for interconversion of the viscosity numbers determined in sulfuric acid and in formic acid as solvent are presented in Figure 2. The reliability of the conversion is discussed in 13.1.

9.3 For PA 612 and corresponding copolyamides, sulfuric acid solution or *m*-cresol shall be used as solvent.

NOTE Graphs for interconversion of the viscosity numbers determined in these two solvents are presented in Figure 3. The reliability of the conversion is discussed in 13.2.

9.4 For PA 11, PA 12, PA 11/12 copolymers and PA 6T/66, *m*-cresol shall be used as solvent.

9.5 For other polyamides, any of the three solvents may be used.

10 Procedure

10.1 Cleaning of viscometer

Clean the viscometer (6.6) 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 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.

10.2 Preparation of test solution

Weigh, to the nearest 0,2 mg, a test portion of ($m_c \pm 5$) mg, where m_c is the mass calculated in accordance with Clause 8, working rapidly to minimize moisture pick-up by the polymer. If the weighing takes more than 2 min, discard 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 selected in accordance with Clause 9. Close the flask and shake, or stir with the magnetic stirrer (6.4), the contents 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 in 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 is used as the solvent, the temperature may be raised to 95 °C to 100 °C. For PA 6T/66, 2 h at 90 °C have been found to be suitable conditions. If, when heating at such higher temperatures, dissolution takes more than 2 h, this shall be reported.

When dissolution is complete, cool the solution to approximately 25 °C, dilute to the mark with the solvent and mix well. The temperature of the solution during dilution shall lie between 23 °C and 27 °C. If the magnetic stirrer (6.4) is used, remove it from the solution before dilution and rinse it with the solvent, adding the rinsings to the flask before further dilution.

NOTE 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 may only be used for mutual comparison with similar products.

10.3 Measurement of flow times

Filter the solution through the sintered-glass filter or metal sieve (6.5) 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 (6.6). The volume of liquid shall be such that, after draining, the level lies between the filling marks. Preferably, the filling should be done with the viscometer out of the thermostatic bath (6.7) to avoid contamination of the bath in the event of accidental spills.

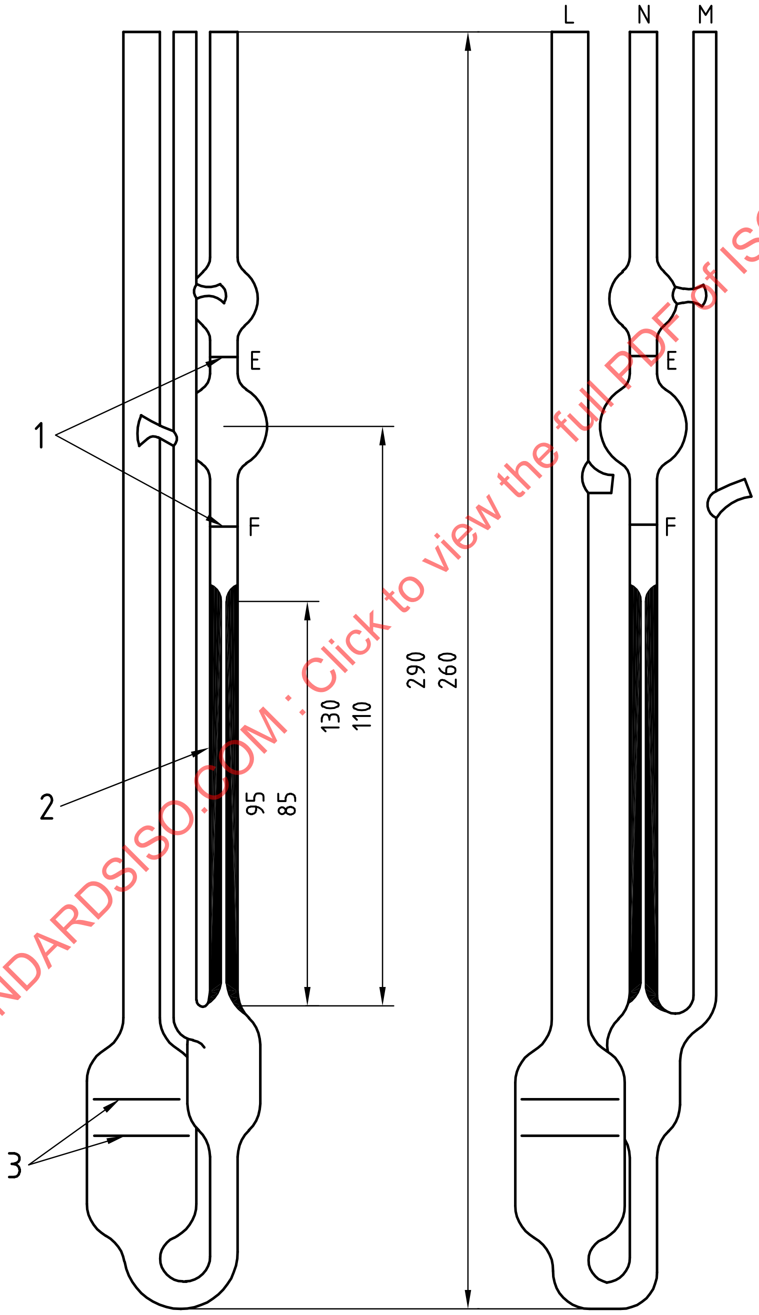
Mount the viscometer in the thermostatic bath maintained at 25 °C ± 0,05 °C, ensuring that tube N is vertical and that the upper graduation mark, E, is at least 30 mm below the surface of the liquid in the bath. Allow at least 15 min for the charged viscometer to attain the temperature of the bath.

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 for the solution.

Determine the mean flow time for the solvent in the same viscometer and in the same manner as that of the solution. Measure the solvent mean flow time only once for each series of determinations; however, measure the time at least once each day the solvent is used. If two successive determinations of the solvent mean flow time differ more than 0,4 s, clean the viscometer (see 10.1).

With each polyamide sample, carry out at least two determinations of the viscosity number, each time using a fresh solution, 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.

Dimensions in millimetres



Key

1 graduation marks for a volume of 4 ml ± 0,2 ml

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

3 filling marks

Figure 1 — Ubbelohde viscometer

11 Expression of results

Calculate the viscosity number VN, in millilitres per gram, from the equation:

$$\mathrm{VN} = \left(\frac{t}{t_0} - 1 \right) \times \frac{1}{c}$$

where

t is the flow time for the solution;

t_0 is the flow time for the solvent;

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

NOTE Density difference and kinetic energy corrections can be neglected in this method; therefore, in calculating the viscosity number of the polymer, the ratio of the viscosities of the solution and the solvent (see Clause 3) can be replaced by the ratio of the corresponding times of flow. Furthermore, the concentration of the polymer may be expressed in grams per millilitre of solvent instead of grams per millilitre of solution, without introducing appreciable error.

12 Repeatability and reproducibility

The repeatability and reproducibility of the determination of the viscosity number depend on the solvent used. The values are given in Table 2.

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

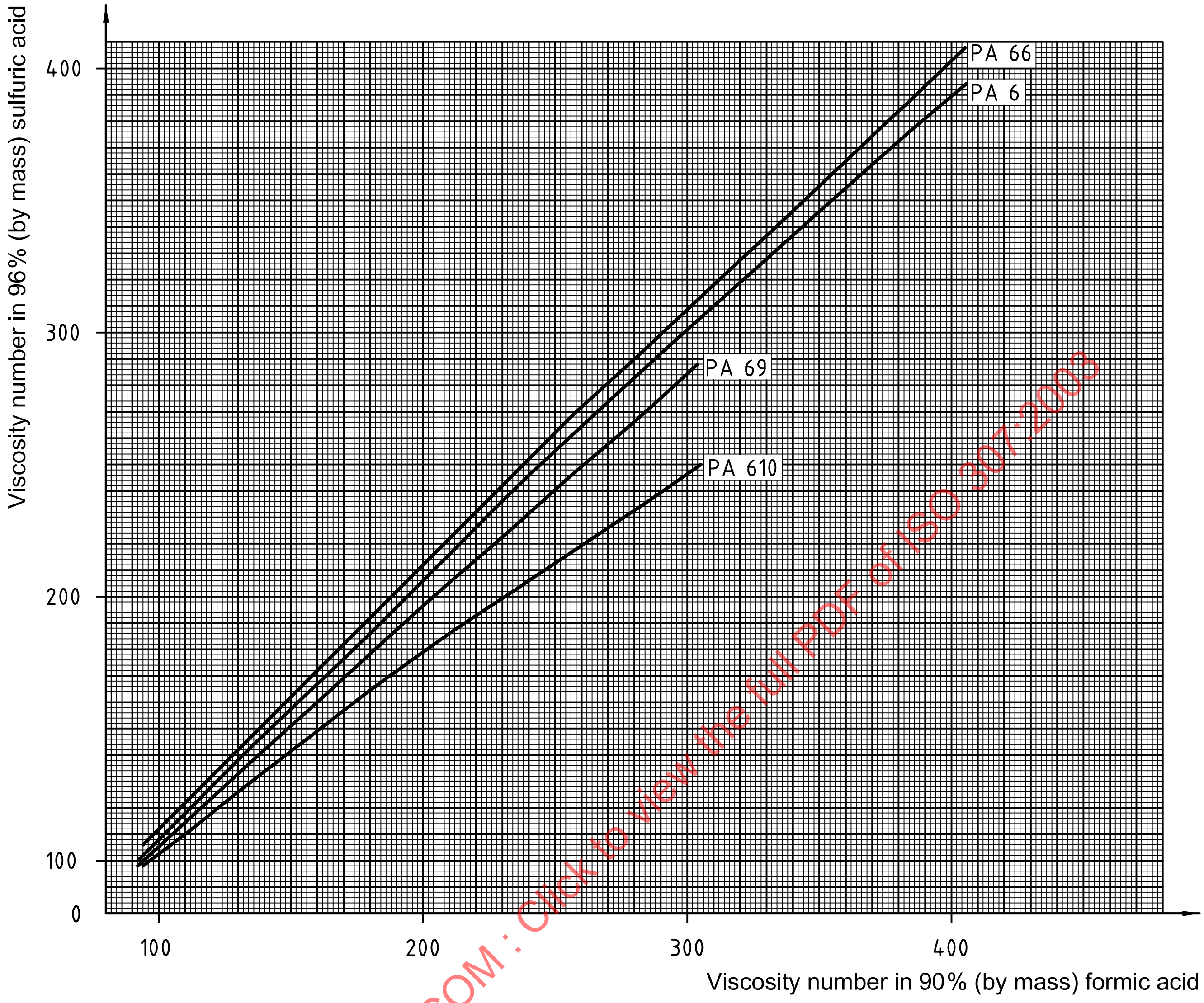
13.1 Relation between the viscosity number determined in 96 % (by mass) sulfuric acid, 90 % (by mass) formic acid and *m*-cresol

13.1.1 Viscosity number in 96 % (by mass) sulfuric acid and 90 % (by mass) formic acid, respectively

Graphs of the relationship between the viscosity number 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 2.

The value measured by a particular 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, expressed as percentages of the converted values, are:

for PA 6	± 9 %
for PA 66	± 9 %
for PA 69	± 10 %
for PA 610	± 14 %



Equations

For PA 6

$$\ln y = 0{,}416\,1 + 0{,}927\,6 \ln x$$

For PA 66

$$\ln y = 0{,}454\,1 + 0{,}926\,1 \ln x$$

For PA 69

$$\ln y = 0{,}463\,4 + 0{,}909\,5 \ln x$$

For PA 610

$$\ln y = 0{,}982\,3 + 0{,}793\,2 \ln x$$

where

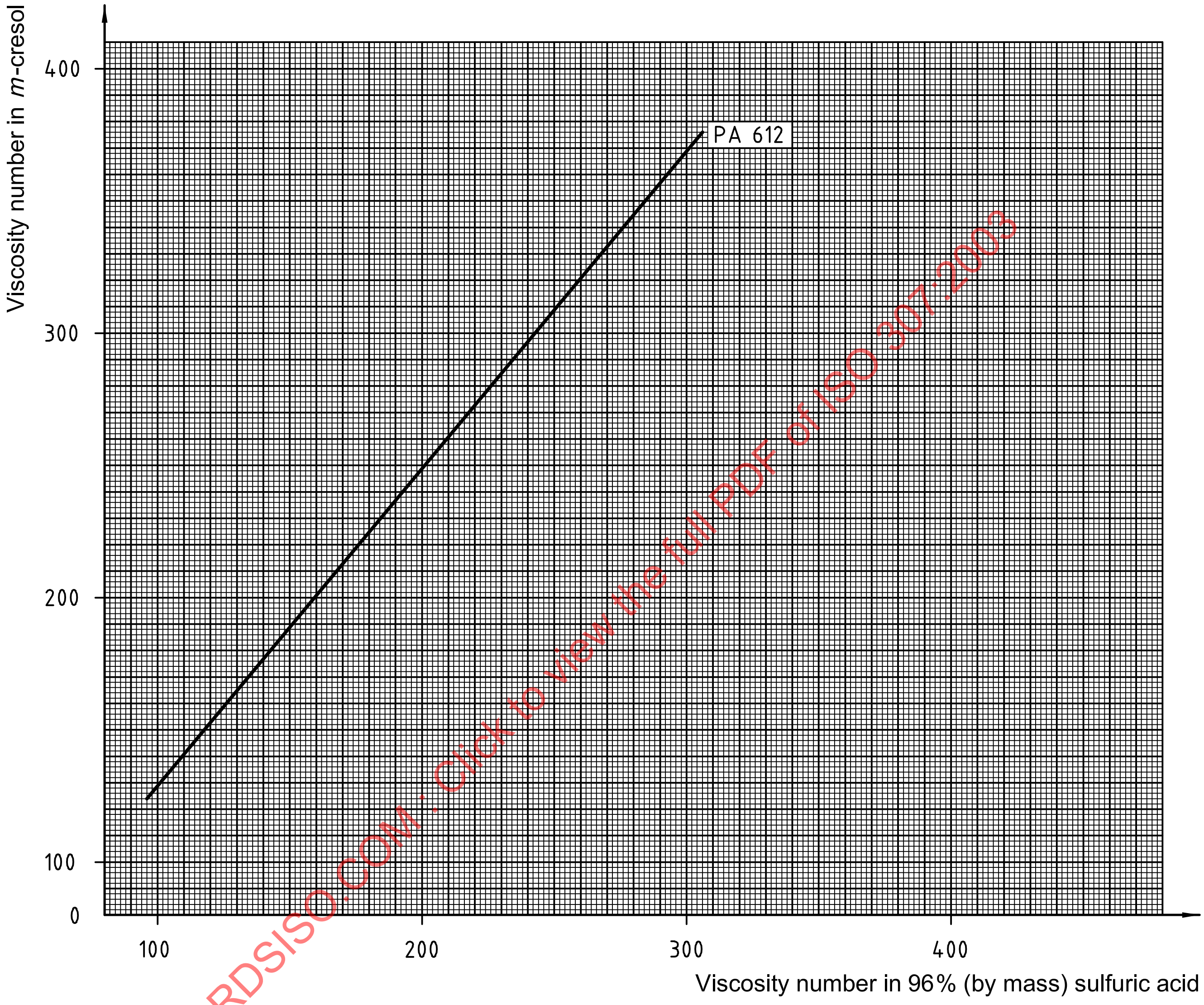
x is the viscosity number in 90 % (by mass) formic acid;

y is the viscosity number in 96 % (by mass) sulfuric acid.

Figure 2 — Calibration lines for the interconversion of the viscosity number of PA 6, PA 66, PA 69 and PA 610 in 90 % (by mass) formic acid solution and in 96 % (by mass) sulfuric acid solution

13.1.2 Viscosity number in 96 % (by mass) sulfuric acid and *m*-cresol, respectively

A graph of the relation between the viscosity number of PA 612 in sulfuric acid solution (5.1.1) and in *m*-cresol (5.1.3) is presented in Figure 3.



Equation

For PA 612

$$\ln y = 0{,}285\,7 + 0{,}985\,9 \ln x$$

where

x is the viscosity number in 96 % (by mass) sulfuric acid;

y is the viscosity number in *m*-cresol.

Figure 3 — Calibration lines for the interconversion of the viscosity number of PA 612 in 96 % (by mass) sulfuric acid solution and in *m*-cresol

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, expressed 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 %.

13.1.3 Precision

The relationship between the viscosity number determined in different solvents was determined in the interlaboratory investigation referred to in the note to Table 2.

Table 2 — Repeatability and reproducibility

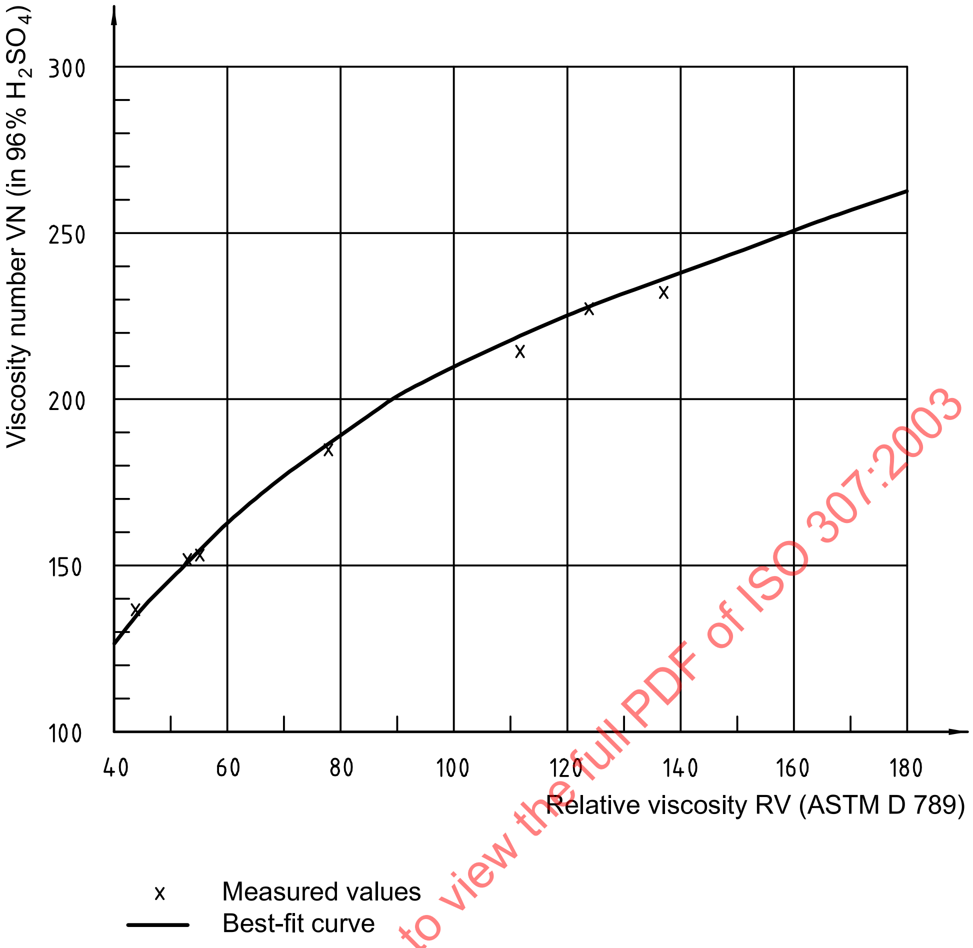
Solvent	Repeatability %	Reproducibility %
Sulfuric acid solution (5.1.1)	2	5
Formic acid solution (5.1.2)	2	10
m-Cresol (5.1.3)	3	10

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, nine of PA 66, three of PA 69, four of PA 610, five of PA 612 and two of PA 6T/66. The viscosity numbers of the samples were determined in duplicate in both solvents in accordance with this International Standard.

13.2 Interconversion of relative viscosity determined in accordance with ASTM D 789 and viscosity number determined in 96 % (by mass) sulfuric acid

A conversion table and a graph of the relationship between the relative viscosity of PA 6 and PA 66 and the viscosity number of PA 6 and PA 66 in sulfuric acid solution (5.1.1) are presented in Table 3 and Figure 4, respectively.

The precision of the relationship between the relative viscosity of PA 6 and PA 66 and the viscosity number 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 Figure 4 and Table 3 is only given as an indication of what relation to expect.



Equation

For PA 6 and PA 66

$$\mathrm{VN} = A + B \ln(\mathrm{RV})$$

where

$A = -206{,}521\ 24$;

$B = 90{,}233\ 55$.

Figure 4 — Interconversion of relative viscosity (ASTM D 789) and viscosity number for PA 6 and PA 66

Table 3 — Relative viscosity RV (ASTM D 789) and viscosity number VN for PA 6 and PA 66
(values taken from curve in Figure 4)

RV (ASTM D 789)	VN (ISO 307) [in 96 % (by mass) H_2SO_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
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
187	265,50
189	266,46
191	267,41
193	268,35
195	269,28
197	270,20
199	271,11
201	272,01
203	272,91
205	273,79
207	274,67
209	275,54
211	276,40
213	277,25
215	278,09
217	278,93
219	279,75
221	280,57
223	281,39
225	282,19

13.3 Interconversion of relative viscosity (JIS K 6920-2:2000, Annex B) and viscosity number for PA 6 and PA 66

Round-robin testing was carried out by eight laboratories in 1999 to check the interconvertibility between the relative viscosity of PA 6 and PA 66, measured in 98 % (by mass) sulfuric acid solution in accordance with JIS K 6920-2:2000, Annex B, and the viscosity number, measured in 96 % (by mass) sulfuric acid in accordance with ISO 307.

The linear relationship obtained, shown in Figure 5, is described by the following equation:

$$\mathrm{VN} = 69{,}771\,\mathrm{RV} - 49{,}372$$

where

RV is the relative viscosity measured in accordance with JIS K 6920-2:2000, Annex B;

VN is the viscosity number measured in accordance with ISO 307 (ml/g).

The regression coefficient = 0,982 3.

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

- solvent: (98,0 ± 0,2) % (by mass) sulfuric acid
- polymer concentration: (0,250 ± 0,001) g/25 ml of solvent (0,01 g/ml)
- temperature: 25,0 °C ± 0,1 °C
- viscometer: Ostwald type

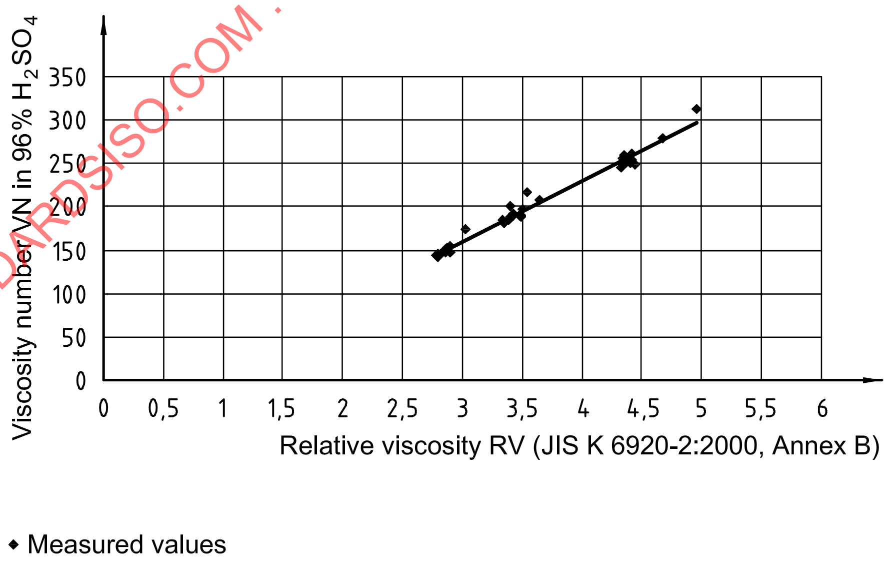


Figure 5 — Interconversion of relative viscosity in 98 % (by mass) sulfuric acid solution (JIS K 6920-2:2000, Annex B) and viscosity number for PA 6 and PA 66

13.4 Interconversion of relative viscosity in 95,7% (by mass) sulfuric acid and viscosity number in 96 % (by mass) sulfuric acid (both measured using ISO 307) for PA 6 and PA 66

Round-robin testing was carried out by five laboratories in 1999 to check the interconvertibility between the relative viscosity of PA 6 and PA 66, measured in 95,7 % (by mass) sulfuric acid at a polymer concentration of 0,01 g/ml using the procedure in ISO 307, and the viscosity number, measured in 96 % (by mass) sulfuric acid in strict accordance with ISO 307.

The linear relationship obtained for PA 6, shown in Figure 6, is described by the following equation:

$$\mathrm{VN} = 77{,}450\,2\ \mathrm{RV} - 59{,}194\,7$$

where

RV is the relative viscosity η/η_0, measured in 95,7 % (by mass) sulfuric acid using the procedure in ISO 307;

VN is the viscosity number, measured in 96 % (by mass) sulfuric acid in strict accordance with ISO 307 (ml/g).

The regression coefficient = 0,998 9.

The linear relationship obtained for PA 66, shown in Figure 7, is described by the following equation:

$$\mathrm{VN} = 77{,}573\,9\ \mathrm{RV} - 59{,}897\,0$$

where

RV is the relative viscosity η/η_0, measured in 95,7 % (by mass) sulfuric acid using the procedure in ISO 307;

VN is the viscosity number, measured in 96 % (by mass) sulfuric acid in strict accordance with ISO 307 (ml/g).