INTERNATIONAL STANDARD

ISO 14446

First edition
1999-05-01

Binders for paints and varnishes — Determination of the viscosity of industrial cellulose nitrate solutions and classification of such solutions

Liants pour peintures et vernis — Détermination de la viscosité des solutions de nitrate de cellulose industrielles et classification de ces solutions

Reference number
ISO 14446:1999(E)

Foreword

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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.

International Standard ISO 14446 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

Annex A forms a normative part of this International Standard.

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Printed in Switzerland

Binders for paints and varnishes — Determination of the viscosity of industrial cellulose nitrate solutions and classification of such solutions

1 Scope

This International Standard specifies a method of determining the viscosity of industrial cellulose nitrate, usually referred to as nitrocellulose, the nitrogen content of which can vary between 10,7 % by mass and 12,6 % by mass, depending on the type.

It also gives a classification system for industrial cellulose nitrate solutions (see annex A) which is based on viscosity measurements made using the method. The use of a standard procedure results in "standard" types and avoids classification differences which could be caused by the fact that there are many ways of determining viscosity and a wide variety of solvents available.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 760:1978, *Determination of water — Karl Fischer method (General method).*

ISO 2811-1:1997, *Paints and varnishes — Determination of density — Part 1: Pyknometer method.*

ISO 2811-2:1997, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method.*

ISO 2811-3:1997, *Paints and varnishes — Determination of density — Part 3: Oscillation method.*

ISO 12058-1:1997, *Plastics — Determination of viscosity using a falling-ball viscometer — Part 1: Inclined-tube method.*

ISO 15528:—[1)], *Paints and varnishes — Sampling*.

3 Principle

The cellulose nitrate is dissolved in an acetone/water mixture at a concentration which will depend on the type of cellulose nitrate concerned. The density of the solution is determined using a pyknometer and the viscosity using a Höppler falling-ball viscometer. The dynamic viscosity of the solution is calculated from the results obtained.

[1)] To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

4 Apparatus and reagents

Ordinary laboratory apparatus and glassware, together with the following:

4.1 Falling-ball viscometer, as specified in ISO 12058-1, using ball No. 4.

4.2 Thermostat, capable of maintaining the water jacket round the falling-ball viscometer at (20 ± 0,1) °C.

4.3 Stopwatch, accurate to ± 0,1 s.

4.4 Drying oven, capable of being maintained at (65 ± 2) °C, with a back-up safety system to ensure that this temperature is not exceeded.

4.5 Analytical balance, capable of weighing to 1 mg.

4.6 Bottle-shaking or rolling device.

4.7 Pyknometer, capacity 25 ml, as specified in ISO 2811-1, or **plummet**, as specified in ISO 2811-2, or **oscillator**, as specified in ISO 2811-3.

4.8 Weighing bottle, capacity 250 ml, with a wide mouth and stopper.

4.9 Watch glass or **Petri dish.**

4.10 Acetone/water mixture, containing (5 ± 0,2) % by mass of water. The water content of the mixture shall be checked using the method described in ISO 760.

5 Sampling and preparation of samples for testing

WARNING — For safety reasons, cellulose nitrate should always be moistened with water or a suitable organic liquid, normally ethanol, isopropanol or butanol.

5.1 Take a representative sample of the cellulose nitrate to be tested, as described in ISO 15528.

5.2 Spread the moist sample in a thin layer on a watch glass or Petri dish and heat it in an air oven at (65 ± 2) °C to constant mass. This is reached when the results of two weighings made at an interval of 30 min do not differ by more than 1 mg.

For safety reasons, the door of the oven shall be left slightly open.

5.3 Allow the dried cellulose nitrate to cool in a desiccator and keep it in the desiccator until needed for weighing out the test portion.

NOTE Cellulose nitrate is hygroscopic.

Do not store dried cellulose nitrate. Any dried cellulose nitrate not used shall be moistened again.

6 Preparation of apparatus for testing

6.1 Clean the viscometer, the pyknometer or plummet or oscillator and the weighing bottle with acetone before use and dry in a stream of clean, dry air.

Repeat this cleaning procedure before every determination.

6.2 Determine the viscometer constant K (see ISO 12058-1) at a temperature of (20 ± 0,1) °C with a certified reference liquid. Repeat this determination from time to time.

7 Procedure

7.1 Weigh into the weighing bottle, to the nearest 1 mg, an amount of the dried cellulose nitrate sufficient for the determination of the dynamic viscosity and the density. If the type of cellulose nitrate in the sample is known, add the quantity of acetone necessary to give the concentration required for that particular type of cellulose nitrate (see Table A.1) and stopper the bottle. If the type of cellulose nitrate is not known, make an educated guess as to the amount of acetone necessary.

7.2 Shake or roll the bottle for 6 h at a temperature of (20 ± 5) °C to dissolve the cellulose nitrate. If the cellulose nitrate is not completely dissolved after this time, discard the solution.

7.3 Determine immediately, to the nearest 0,1 s, the time of fall using the falling-ball viscometer method described in ISO 12058-1 four times at a temperature of (20 ± 0,1) °C using ball No. 4.

The times of fall shall not differ by more than 0,5 s.

7.4 Determine from another portion of the test solution the density with a pyknometer, using the method described in ISO 2811-1, or a plummet, using the method described in ISO 2811-2, or an oscillator, using the method described in ISO 2811-3.

7.5 If the type of cellulose nitrate in the sample is not known, carry out two further determinations using test solutions of concentrations which will give viscosities successively closer to (400 ± 25) mPa·s.

8 Expression of results

8.1 The dynamic viscosity η, in mPa·s, is given by the following equation:

$$\eta = K(\rho_1 - \rho_2) \times t$$

where

K is the viscometer constant, in mPa·s·cm^3/(g·s) (see ISO 12058-1:1997, Table 1);

ρ_1 is the density, in g/cm^3, of the ball;

ρ_2 is the density, in g/cm^3, of the cellulose nitrate solution;

t is the time of fall, in s, of the ball.

8.2 To determine the type of cellulose nitrate in a sample in which the type is not known, plot a curve of concentration versus viscosity for the three determinations carried out (see 7.5) and read off the concentration of the solution having a viscosity of (400 ± 25) mPa·s. Knowledge of the N_2 content will enable the designation to be read from Table A.1.

9 Precision

The following precision data have been taken from ISO 12058-1.

9.1 Repeatability (*r*)

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability is 0,5 %.

9.2 Reproducibility (R)

The value below which the absolute difference between two test results, each the mean of duplicates, obtained on identical material by operators in different laboratories using the standardized test method, may be expected to lie with a 95 % probability is 2 %.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 14446);

c) the concentration of cellulose nitrate in the test solution, in % by mass;

d) the result of the test, as indicated in clause 8;

e) details of any incident which may have affected the result;

f) any deviation from the test method specified;

g) the date of the test.

Annex A
(normative)

Classification system for industrial cellulose nitrate

The classification system for industrial cellulose nitrate comprises two elements (see Table A.1):

— a number indicating the concentration of the solution that is required to give a viscosity of (400 ± 25) mPa·s, followed by

— a letter identifying the solvent in which it is soluble (corresponding to the N_2 content), i.e.

E in the case of an ester-soluble grade (N_2 content about 11,8 % to 12,3 %),

M in the case of a medium-soluble grade (N_2 content about 11,3 % to 11,8 %),

A in the case of an alcohol-soluble grade (N_2 content about 10,7 % to 11,3 %).

Since solutions of cellulose nitrate are non-Newtonian in behaviour, the results of viscosity measurements on solutions of different types of cellulose nitrate are only comparable if they have been made within the same restricted viscosity range, as well as at the same shear rate.

The viscosity range (400 ± 25) mPa·s has been chosen since solutions having a viscosity within this range are convenient to prepare, they require only relatively small quantities of cellulose nitrate and their concentrations correspond to those used in many practical applications.

The Höppler falling-ball viscometer is used as a convenient way of determining the viscosity.

All measurements are made in solutions prepared using as solvent a mixture of 95 % by mass of acetone plus 5 % by mass of water.

EXAMPLE An ester-soluble cellulose nitrate which, when dissolved in the above-mentioned solvent mixture at a concentration of 24 % by mass, gives a viscosity of (400 ± 25) mPa·s is designated type 24E.

A dynamic viscosity other than (400 ± 25) mPa·s may be agreed between the interested parties, but shall be reported in the test report.

Table A.1 — Classification of cellulose nitrate solutions by the concentration of the test solution that gives a viscosity of (400 ± 25) mPa·s

Concentration of test solution % by mass[a]	Classification by type[b]		
	Soluble in esters	Medium-soluble[c]	Soluble in alcohols
3			
4	Type 4E		
5			
6			
7	Type 7E		
8	Type 8E		
9	Type 9E		
10			
11			
12	Type 12E	Type 12M	
13			
14		Type 14M	
15	Type 15E		Type 15A
16		Type 16M	
17		Type 17M	
18		Type 18M	
19		Type 19M	
20			
21	Type 21E	Type 21M	
22	Type 22E		
23			Type 23A
24	Type 24E	Type 24M	
25			
26		Type 26M	
27	Type 27E	Type 27M	Type 27A
28	Type 28E		
29		Type 29M	
30			Type 30A
31			
32	Type 32E	Type 32M	
33			
34	Type 34E	Type 34M	
35			
36		Type 36M	
37			
38	Type 38E		

[a] Of dry cellulose nitrate.

[b] The designations listed are examples of typical types and do not exclude additional types.

[c] Medium-soluble cellulose nitrate is normally used in printing inks and for coating of cellophane.

ICS 83.080.20; 87.060.20

Price based on 6 pages