

International Standard



7387/1

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Adhesives with solvents for assembly of uPVC pipe elements — Characterization — Part 1 : Basic test methods

Adhésifs à solvant pour assemblages d'éléments de canalisation en uPVC — Identification — Partie 1 : Méthodes d'essai de base

First edition — 1983-09-15

UDC 665.939.5 : 621.643.29 : 620.1

Ref. No. ISO 7387/1-1983 (E)

Descriptors : piping, plastic tubes, unplasticized polyvinyl chloride, adhesive, tests, determination, viscosity, dynamic viscosity, dry matter.

Price based on 10 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 7387/1 was developed by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, and was circulated to the member bodies in May 1981.

It has been approved by the member bodies of the following countries :

Australia	Greece	Romania
Austria	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Italy	Sri Lanka
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Czechoslovakia	Korea, Rep. of	Switzerland
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The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

Adhesives with solvents for assembly of u PVC pipe elements — Characterization —

Part 1 : Basic test methods

0 Introduction

ISO 7387 is a collection of test methods relating to the characterization of adhesives with solvents for the assembly of u PVC pipes and fittings.

These methods have been divided into groups, each of which is issued as a part of ISO 7387.

Part 1 consists of the basic methods which are essential in deciding the uniformity of an adhesive in relation to itself.

Part 2 consists of methods which can, if necessary, be used to supplement the previous ones for the same purpose.

Part 3 gives methods for separating the constituent parts of the adhesives, for the purpose of closer characterization.

Methods of testing suitability for use are planned to supplement the test methods for characterization.

The various test methods are given in the following parts of ISO 7387.

Part 1 : Basic test methods

- Dynamic viscosity according to the shear rate
- Brookfield viscosity
- Conventional dry matter content
- Ash content

Part 2 : Secondary test methods

- Bulk density
- Flash point

Part 3 : Other test methods

- Determination of the total quantity of resin and powder substances
- Extraction and determination of the total quantity of solvents

1 Scope and field of application

This part of ISO 7387 specifies the basic test methods for adhesives with solvents for the assembly of u PVC pipe elements for characterization purposes.

It is divided into three sections :

Section one : viscosity;

Section two : conventional dry matter content;

Section three : ash content. (Applicable only to adhesives which do not contain a filler unstable at the test temperature.)

Section one : Viscosity

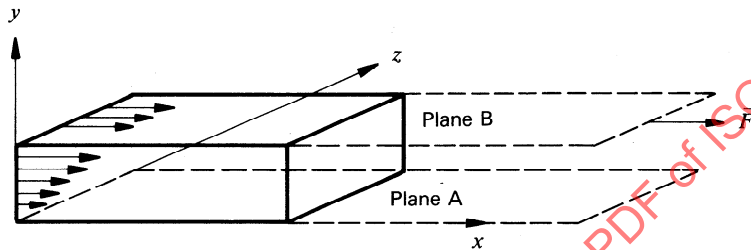
2 General

Two test methods are specified for the determination of this characteristic. The first allows the measurement of dynamic viscosity according to the shear rate; this is the reference method. The second is the Brookfield method which can be reserved, for example, for manufacturing inspection.

3 Dynamic viscosity according to shear rate

3.1 Definitions

For the purpose of this International Standard, the viscosity of a solution is defined as follows :



Consider a volume of liquid lying within two parallel planes, A and B.

Plane A is stationary, plane B is moved at a uniform velocity v by a force F .

Assuming laminar flow, when rate velocity v is low, it can be assumed that the laminar elements of liquid slide in relation to each other at a uniform shear rate defined by the formula:

$$\dot{\gamma} = \frac{dv}{dy}$$

In the case of laminar flow, $\dot{\gamma}$ is in direct proportion to the force applied per surface unit, A :

$$\dot{\gamma} = \frac{1}{\eta} \times \frac{F}{A}$$

where

η is the reciprocal of the coefficient of proportionality, that is the dynamic viscosity of the liquid under consideration; in pascals seconds (Pa.s)*;

$$\frac{F}{A} = \tau \text{ is the shear stress, in pascals (Pa);}$$

$\dot{\gamma}$ is the shear rate, in seconds to the power minus one (s^{-1}).

Liquids for which, at a given temperature, τ is independent of $\dot{\gamma}$ are called Newtonian. In other cases, the liquid is said to be non-Newtonian.

A liquid can therefore only be characterized by its viscosity coefficient if it is Newtonian. If this is not so, the value of $\dot{\gamma}$ or τ for which η is indicated shall be stated.

3.2 Principle

Measuring, at a determined temperature, the value of torque required to overcome the resistance of adhesive to the movement of an immersed rotor turning at a determined and constant rate.

Plotting of the shear stress curve as a function of the shear rate ($\tau = \eta \dot{\gamma}$).

3.3 Apparatus

Viscometer, with a cylindrical moving body, turning in a vessel which is also cylindrical. The vessel and the cylinder are strictly coaxial.

This viscometer shall be equipped with the following :

- manual switch allowing selection of known shear rates;
- measuring equipment allowing a value p proportional to the viscosity and to the shear stress τ to be indicated.
- thermostatically controlled environment, enabling the revolving moving body and the vessel to be kept at a constant temperature.

3.4 Preparation of the test sample

Mix the adhesive to make it homogeneous and take a sample of sufficient volume to enable two measurements to be carried out.

* 1 Pa.s = 1 N.s/m² = 10³ cP (centipoise).

3.5 Procedure

Condition the following for 16 h at a temperature of $23 \pm 1^\circ\text{C}$:

- the sample (in a closed container to avoid evaporation of the solvents);
- the moving body and vessel chosen (perfectly clean and dry).

Carry out the measurements after selecting the cylinder and the required range for the shear rate and setting the thermostatically controlled environment at the temperature of $23 \pm 1^\circ\text{C}$.

NOTE — It may be necessary to coat the outside of the vessel with glycerine (for example) to ensure suitable thermal contact between it and the thermostatically controlled environment.

Determine the curve $\tau = \eta\dot{\gamma}$ by varying the shear rate $\dot{\gamma}$ first by increasing values followed by decreasing values.

3.6 Expression of results

The measuring appliance gives an indication of a value p such that :

$$p = \eta \frac{B}{AU}$$

where

η is the dynamic viscosity, in pascals seconds (Pa.s);

A and B are constants relating to the equipment used;

$U = \frac{B}{\dot{\gamma}}$ is the instrument reading which is in inverse proportion to the shear rate $\dot{\gamma}$.

From the above formula and the ratio $\tau = \eta\dot{\gamma}$, calculate the values of shear stress τ , in pascals (Pa), obtained for each value of $\dot{\gamma}$, at the temperature of the determination.

Record for each value of $\dot{\gamma}$ the arithmetic mean of the two values of τ measured.

The test shall be considered invalid if the two results differ by more than 5 %.

From the paired values of $\dot{\gamma}$ and τ , obtained by increasing and then decreasing the value of $\dot{\gamma}$, plot the curves $\tau = f(\dot{\gamma})$.

3.7 Test report

The test report shall include the following information :

- a) reference to this International Standard;
- b) the full reference of the adhesive examined;

c) the temperature of the determination;

d) the values of τ obtained for each determination, and the mean value;

e) the $\tau = f(\dot{\gamma})$ curves, determined as shown in 3.5.

4 Brookfield viscosity

4.1 General

This method is largely based on ISO 2555, *Resins in the liquid state or as emulsions or dispersions — Determination of the Brookfield RV viscosity*.

It applies, in particular, to solvent-containing adhesives for the assembly of unplasticized polyvinyl chloride (u PVC) pipe elements, and specifies :

- sampling;
- preparation and conditioning of the test portion;
- a further possibility regarding the choice of speed and of the moving body;
- the temperature at which the test shall be carried out.

4.2 Principle

A moving body of cylindrical or related shape (disc), driven by a synchronous motor, rotates at constant speed around its axis in the adhesive being examined.

The resistance exerted by the adhesive against the moving body, which depends on the viscosity of the product, causes tension in a spring coil which is indicated by the movement of a needle on a dial.

The "Brookfield viscosity" is measured by multiplying the value of this movement by a coefficient depending on the speed of rotation and the characteristics of the moving body.

The products to which this method applies are generally non-Newtonian, and the viscosity measured depends on the shear rate to which the product is subjected during measurement.

However, in this type of viscometer, the shear rate is not the same at all points on the moving body. Therefore, for non-Newtonian liquids, the result of measurement is not that of a "viscosity" at known shear rate, which is why it is conventionally called "Brookfield RV viscosity".

4.3 Apparatus

4.3.1 RV model synchro-lectric Brookfield viscometer, (RVF, RVF-100 or RVT), to be chosen according to the product being studied and the accuracy of measurement required.

NOTE — RV model Brookfield viscometers allow viscosity measurements between 0,02 Pa·s (20 cP) and 8 000 Pa·s (8×10^6 cP)*.

Details of the principle of operation of this equipment, its description and the characteristics of these three types are given in the annex.

Each viscometer is made up of the following elements :

- the viscometer body;
- seven removable, interchangeable moving bodies, numbered from 1 to 7 (No. 1 being the largest); these moving bodies have a guide mark on their shafts, indicating the level of immersion into the liquid; they are common to the three types of viscometer;
- removable guard.

The rotational speeds which can be used in the three types of RV Brookfield viscometer are as follows :

Type	Speeds in r/min						
RVF		2	4	10	20		
RVF-100				10	20	50	100
RVT	0,5	1	2,5	5	10	20	50

The shapes and dimensions of the moving bodies are such that the "Brookfield RV viscosities" corresponding to the maximum deviation of the needle on the dial, in terms of the speed, are as indicated in table 1.

Table 1 — Brookfield viscosity

Type of viscometer	Speed r/min	Viscosity in Pa·s corresponding to the whole scale for moving body No.						
		1 (large)	2	3	4	5	6	7 (small)
RVT, RVF-100	100	0,1	0,4	1	2	4	10	40
	50	0,2	0,8	2	4	8	20	80
RVF, RVT, RVF-100	20	0,5	2	5	10	20	50	200
	10	1	4	10	20	40	100	400
RVT	5	2	8	20	40	80	200	800
RVF	4	2,5	10	25	50	100	250	1 000
RVT	2,5	4	16	40	80	160	400	1 600
RVF	2	5	20	50	100	200	500	2 000
RVT	1	10	40	100	200	400	1 000	4 000
	0,5	20	80	200	400	800	2 000	8 000

Adjustment and calibration of these viscometers are normally carried out by the manufacturer.

Checking of this adjustment and calibration, which it is recommended should be carried out from time to time, can be undertaken using Newtonian liquids of known viscosity, either by the user laboratories or by public laboratories.

* 1 Pa·s = 10^3 cP

4.3.2 Thermostatically controlled water baths, equipped with thermostats to enable the product undergoing examination to be maintained at the test temperature within a tolerance of $\pm 0,2$ °C.

4.3.3 Support, enabling the equipment to be held in position and the viscometer to be moved in a vertical plane.

4.3.4 Beaker, 90 to 92 mm in diameter and 115 to 160 mm in height.

4.3.5 Thermometer, graduated in 0,1 °C, for measuring the temperature of the test product.

4.4 Preparation of the test sample

Mix the adhesive to make it homogeneous and take a test sample of sufficient volume to enable the requisite test portions to be taken.

The test sample shall be free from air bubbles, skin and foreign bodies in suspension.

Pour the test sample into a container; seal it hermetically.

Place the vessel containing the test sample in the thermostatically controlled bath, set at the temperature chosen for the determination, with the tolerance prescribed for it. Leave it in the bath long enough for the temperature of the test sample to be uniform at all points.

4.5 Choice of speed and moving body

Select the speed/moving body pairing according to the value of the viscosity to be measured, the required accuracy and the shear rate envisaged.

This choice shall be made in such a way that no measurement corresponds to less than 20 % or more than 95 % of the total scale. However, in order to obtain greater accuracy, it is advisable to limit the difference to 45 to 94 %.

NOTES

1 If comparison of the viscosities of non-Newtonian products is intended, it is essential to use the same speed/moving body pairing for these measurements, even if the accuracy of one of the measurements is greatly reduced.

2 The choice of the speed automatically leads to the choice of one or more types of equipment. It is therefore recommended that speeds of 10 or 20 r/min be used if possible, as only these speeds are common to the three types.

3 It should be noted that type RVF has a lower limit of about 0,1 Pa·s (100 cP), whereas types RVF-100 and RVT have a lower limit of about 0,02 Pa·s (20 cP).

4 It may be of interest to carry out two determinations using speeds in the ratio of 1 to 10. Thus the product may be characterized by the following ratio :

$$\frac{\text{Brookfield viscosity at speed } v_1}{\text{Brookfield viscosity at speed } v_2} = \begin{cases} \text{thixotropy index or} \\ \text{thixotropy ratio} \end{cases}$$

where v_1 is the lower speed, at least 2 r/min.

The determination at the lower speed shall be carried out first.

4.6 Procedure

For the normal test temperature, take a temperature of 23 ± 1 °C. Mount the viscometer (4.3.1), fitted with its guard, on its support.

Pour into the beaker (4.3.4) a sufficient, homogeneous amount of test sample, taking care not to admit any air bubbles, by for example trickling the adhesive either along a glass rod or down the inside of the beaker. Place it in the thermostatically controlled water bath (4.3.2), having a tolerance of 0,2 °C, for sufficient time to allow it to come fairly close to the required temperature.

Mount the selected moving body on the shaft of the equipment, keeping this shaft stationary and adjusting the assembly coupling.

Keeping the beaker in the water bath, lower the equipment on its support so that the moving body is immersed in the liquid up to the bottom of the guide mark on its shaft. Check that this shaft is vertical using a spirit level and place the thermometer (4.3.5) in the test sample.

Wait until the temperature of the test sample comes within the specified limits.

Operate the motor, reaching the required speed while respecting the manufacturer's instructions.

Release the needle and allow the assembly to rotate until the needle has achieved a stable position in relation to the dial (usually within 5 to 10 s).

NOTE — If the needle does not remain truly stable, but moves slowly over the dial, this often indicates the presence of a thixotropic or theopexic product. In this case, the position of the needle should be noted a given time after pseudo-stabilization of the needle. It is also possible to build up the viscosity curve as a function of duration of rotation.

Lock the needle and stop the motor so that the reading can be taken.

Read the value obtained on the dial, assessing the quarter units (0,25). (The dial is graduated in half units.)

Operate the motor again and make another measurement.

Continue measuring until two consecutive values which do not vary by more than 3 % are obtained.

Take the mean of these two values.

4.7 Expression of results

Calculate the Brookfield RV viscosity, in pascals seconds, of the product under examination, by means of the following formula :

$$\frac{k \times l}{1\,000}$$

where

k is a coefficient depending on the speed/moving body pairing used, as shown in table 2;

l is the mean of the two values read off the dial.

Table 2 — Coefficient k (scale 0 to 100) for each speed/moving body pairing

Speed r/min	Coefficient k for moving body No.						
	1	2	3	4	5	6	7
100	1	4	10	20	40	100	400
50	2	8	20	40	80	200	800
20	5	20	50	100	200	500	2 000
10	10	50	100	200	400	1 000	4 000
5	20	80	200	400	800	2 000	8 000
4	25	100	250	500	1 000	2 500	10 000
2,5	40	160	400	800	1 600	4 000	16 000
2	50	200	500	1 000	2 000	5 000	20 000
1	100	400	1 000	2 000	4 000	10 000	40 000
0,5	200	800	2 000	4 000	8 000	20 000	80 000

Express the results to three significant figures, indicating not only the Brookfield RV viscosity but also the speed/moving body pairing in accordance with the following example :

Brookfield RV viscosity (20,3) = 4,25 Pa.s

4.8 Test report

The test report shall include the following information :

- reference to this International Standard;
- the full reference of the adhesive;
- the test temperature;
- the viscometer model used;
- the value or values of the Brookfield RV viscosity calculated as shown in clause 4.7.

Section two : Conventional dry matter content

5 Definition

conventional dry matter : The relation to its original mass, expressed as a percentage, of the mass of the residue obtained after heating a quantity of a product at a specified temperature for a specified time, well defined experimental conditions being observed.

6 Principle

Depositing of the test portion on a suitable support which has been dried beforehand, natural evaporation of the solvents, then final drying at 110 °C for 60 min and calculation of the conventional dry matter content.

7 Apparatus

The apparatus shall comprise the following elements :

7.1 Drying oven, with natural ventilation, of high thermal inertia, equipped with a grid within the upper third, thermostated to 110 ± 2 °C.

7.2 Balance, with an accuracy of 0,1 mg.

7.3 Desiccator.

7.4 Syringe, of sufficient capacity to hold at least three test portions of the sample.

7.5 Six square plates having sides of approximately 75 mm, or **circular ones** of about 75 mm in diameter, made from glass or other material not likely to be attacked by the adhesive.

8 Preparation of the test sample

Homogenize the adhesive and take a sample having a sufficient volume to enable three test portions to be taken under the conditions defined in clause 9.

9 Procedure

9.1 Clean the six plates (7.5) and dry them in the oven for 30 min at 110 °C.

9.2 Take the plates from the oven and place them in the desiccator for about 15 min.

During subsequent operations :

- keep the oven at 110 °C,
- group the plates in pairs and number them, for example (1, 2), (3, 4), (5, 6).

9.3 Weigh separately each of the plates in the three pairs to within 0,1 mg; (m_1 and m_2), (m_3 and m_4) and (m_5 and m_6) are the respective masses of each of the pairs of plates.

9.4 Suck into the syringe (7.4) a sufficient quantity of adhesive to make three test portions.

Weigh the syringe to within 0,1 mg.

Transfer the test amount, about 0,5 to 1 g, into the centre of one of the plates of one of the three pairs.

Immediately weigh the syringe to within 0,1 mg.

Calculate the mass m_0 of the test amount by subtraction.

9.5 Immediately place the second plate on the test portion and spread the product as evenly as possible by means of gentle rotation, without letting it overflow.

9.6 Separate the two plates and leave them for 10 to 15 min at the laboratory temperature, so that most of the solvents evaporate.

Repeat the operation with the two other pairs of plates.

9.7 Put the plates in the oven for 60 min at 110 °C.

Take the plates out of the oven and put them in the desiccator in such a way that they do not overlap.

Leave the plates in the desiccator to cool.

9.8 Weigh separately each of the elements of the three pairs of plates without putting them on top of one another, so as to avoid loss of dry matter; the respective masses of the pairs of plates are known as (m'_1 and m'_2), (m'_3 and m'_4) and (m'_5 and m'_6).

10 Expression of results

For the pair of plates (1, 2), calculate the percentage of conventional dry matter by means of the formula :

$$m_d = \frac{(m'_1 + m'_2) - (m_1 + m_2)}{m_0} \times 100$$

where

m_d is the conventional dry matter;

m_0 is the mass, in grams, of the test portion (see clause 9);

m_1 and m_2 are the masses, in grams, of the cleaned and dried plates before the test;

m'_1 and m'_2 are the masses, in grams, of the same plates after the test.

Follow the same procedure for pairs of plates (3, 4) and (5, 6).

Take, as the conventional dry matter content, the arithmetic mean of the percentages obtained for each of the determinations. Express the result to one decimal place.

The test shall be considered invalid if the results of each of the three determinations differ from the arithmetic mean by 2 % or more.

In this case begin the test again.

NOTES

1 If more than three measurements are carried out on the same batch of products in the same laboratory, the test result is also given by the arithmetic mean of all the measurements; the requirement relating to the precision indicated above shall also be respected if the test is to be considered valid.

2 If tests are carried out by several laboratories on the same batch of products the absolute values of the deviations between each of the

measurements and the result given by the arithmetic mean shall be less than 5 % of this mean if the test is to be considered valid.

11 Test report

The test report shall include the following information:

- a) reference to this International Standard;
- b) the full reference of the adhesive;
- c) the results of each of the three determinations;
- d) the value of the conventional dry matter;
- e) the date of the test.

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