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Plastics — Poly(vinyl chloride) pastes — Determination of apparent viscosity using the Severs rheometer

Plastiques — Pâtes de poly(chlorure de vinyle) — Détermination de la viscosité apparente au rhéomètre Severs

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Contents

Page

Foreword

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ISO 4575 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 4575:1985), which has been technically revised.

Plastics — Poly(vinyl chloride) pastes — Determination of apparent viscosity using the Severs rheometer

1 Scope

This International Standard specifies a method for determining the apparent viscosity, at high shear, of poly(vinyl chloride) (PVC) pastes prepared from PVC paste polymers and plasticizers, using the Severs rheometer.

It applies in particular to “standard pastes” prepared in accordance with ISO 4612 or ISO 11468.

2 Principle

A sample of the paste is placed in a Severs rheometer and the jacket set to a defined temperature. The flow rate of the paste through a calibrated die is measured at different pressures. The shear rate and apparent viscosity are calculated for each flow rate corresponding to each of the pressures applied. Optionally, a graph of apparent viscosity as a function of the shear rate can be prepared.

3 Apparatus

3.1 Severs rheometer, of capacity 500 ml to 1 000 ml, of one of the two basic designs shown in Figure 1, and consisting essentially of the following components:

3.1.1 Measuring vessel, for example a stainless-steel or bronze cylinder with polished internal surfaces to which can be fixed both a base having an orifice in which can be placed a die, and a cover which can be connected to the source of pressure or to the atmosphere. The whole shall be airtight.

3.1.2 Jacket, connected to a system for temperature control which allows the contents of the body of the measuring vessel to be maintained at a temperature of (23 ± 0,5) °C.

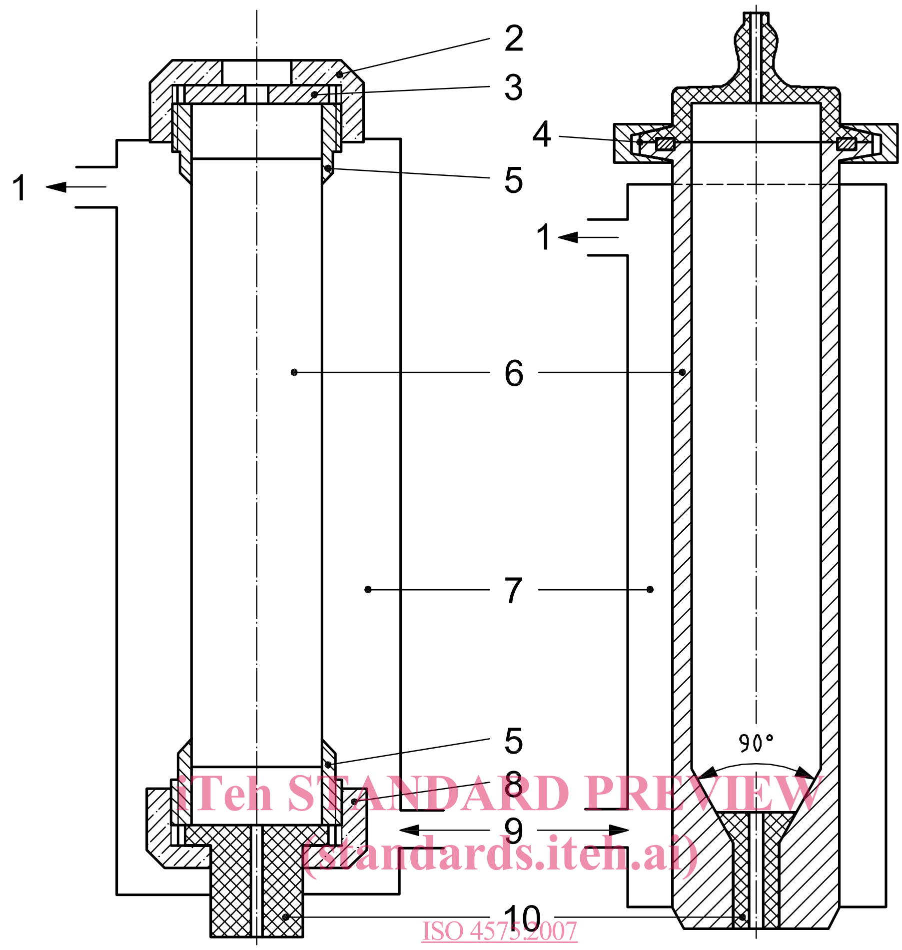
3.1.3 Non-deformable die, for example of stainless steel or PTFE, consisting of a cylindrical tube which can be fitted in airtight fashion to the bottom of the measuring vessel. The outer and internal surfaces of the tube shall be polished. The die is defined by the diameter and the height of the tube. Two dies, A and B, are specified in the application of this International Standard. The dimensions of the dies are given in Table 1.

Table 1 — Dimensions of the dies

Dimensions in millimetres

Dimension	Die A	Die B
Radius of tube	1,5 ± 0,05	1,5 ± 0,05
Height	45 ± 0,5	22,5 ± 0,5

3.1.4 Piston (if necessary), of rigid plastic material with a diameter slightly less than that of the measuring vessel to prevent the fluid, under pressure, from passing directly through the die in the case of pastes which cavitate. (Its use also simplifies the cleaning of the instrument.) A sketch of the piston is given in Figure 2.



Key

1 temperature-regulating fluid out
2 lid
3 PTFE washer
4 clamped joint
5 threaded joint
6 cylinder
7 jacket
8 die clamp
9 temperature-regulating fluid in
10 die

Figure 1 — Diagram showing the basic principles of two types of Severs rheometer

Dimensions in millimetres

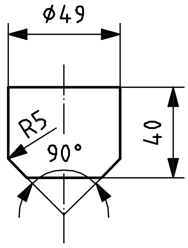
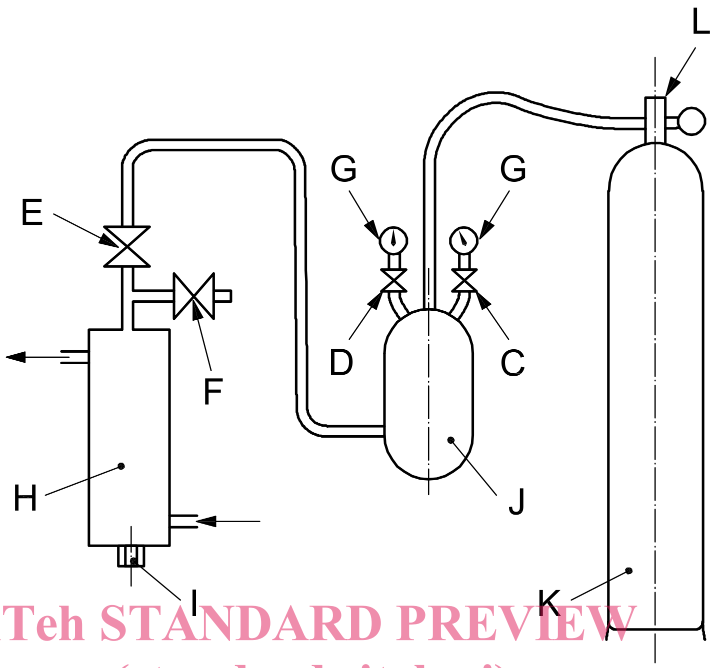


Figure 2 — Piston

3.1.5 Pressurizing device, consisting of a cylinder of pressurized nitrogen, an expansion chamber with pressure gauges, and a number of valves. An example is shown in Figure 3.



Key

C valve
D valve
E pressurized-nitrogen inlet valve
F valve to atmosphere
G pressure gauges (with different scales)
H Severs rheometer
I die
J expansion chamber
K nitrogen cylinder
L pressure reduction valve

Figure 3 — Example of pressurizing device

3.2 Beakers, of capacity about 50 ml.

3.3 Timer, accurate to 0,1 s.

3.4 Balance, accurate to ± 0,5 g.

3.5 Thermometer, capable of measuring a temperature of (23 ± 0,5) °C.

4 Procedure

4.1 Choice of test pressures and die

Depending on the test paste and its intended use, choose at least four pressures in the following list of preferred numbers:

100 kPa, 160 kPa, 250 kPa, 400 kPa, 630 kPa, 1 000 kPa, 1 600 kPa, 2 500 kPa [1)].

Conduct a test with die A and a pressure of 2 500 kPa. From the measured flow rate q_m (see 5.1), calculate as indicated in 5.2 the corresponding shear rate:

— if the shear rate is more than 1 000 s^{-1}, tests shall be carried out with die A;

— if the shear rate is less than or equal to 1 000 s^{-1}, tests shall be carried out with die B.

4.2 Introduction of the paste and control of the temperature

Position the base together with the appropriate die (see 3.1.3) and introduce the paste to be tested into the measuring vessel (3.1.1). If necessary, place the piston (3.1.4) on the paste.

Bring the temperature of the paste to (23 ± 0,5) °C by circulating water maintained at that temperature in the jacket (3.1.2) (generally 5 min is sufficient).

Screw down the lid and connect it to the pressurizing device (3.1.5).

4.3 Regulation of the test pressure

Start the tests at the lowest pressure. Regulate the pressure to ± 10 kPa according to the device used. In the case of the example given in Figure 3, proceed in the following manner:

Close valves E and F, open valves C and D, and operate the pressure reduction valve L progressively until the pressure reading on the gauge having the higher scale range (C) is practically equal to the desired value. Then read the pressure on the gauge having the lower scale range (D) and carefully regulate the pressure to the desired value to the nearest ± 10 kPa.

Operate in the same way when regulating the other test pressures, but without opening valve D for pressures above its maximum gauge reading.

4.4 Determination

4.4.1 Weigh three 50 ml beakers (3.2) to the nearest 0,5 g.

Clean the exit orifice of the die and open valve E to admit nitrogen under pressure to the rheometer. Under pressure, the paste will pass through the die. Make sure that the pressure does not vary; regulate, if necessary, by operating the pressure reduction valve L.

Place a weighed beaker under the emerging paste stream and at the same time start the timer (3.3).

When the mass of paste obtained is at least 10 g, remove the beaker and stop the timer simultaneously; note the time of flow t_1 in seconds to the nearest ± 0,1 s. (If the amount is insufficient at the end of 2 min, abandon the test at this pressure and carry it out under greater pressure.)

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1) 1 kPa = 1 kN/m^2

Repeat the measurements with the other two weighed beakers in turn, designating the corresponding flow times t_2 and t_3.

Close the pressurized-nitrogen inlet valve E and open the valve to atmosphere F in order to arrest the flow of paste.

Weigh the containers, determining the masses of paste m_1, m_2 and m_3, in grams, to the nearest 0,5 g.

4.4.2 Close the valve to atmosphere F and regulate the pressure to its new value. Repeat the test under the conditions specified in 4.4.1.

4.4.3 After testing, clean the rheometer, avoiding the use of fluffy materials.

5 Calculations

5.1 Calculation of flow rates

For each one of the test pressures, calculate the flow rates q_{m1}, q_{m2}, and q_{m3}, in grams per second, corresponding to the measurements, using the equations

$$q_{m_1} = \frac{m_1}{t_1} \qquad q_{m_2} = \frac{m_2}{t_2} \qquad q_{m_3} = \frac{m_3}{t_3}$$

where m_1, m_2 and m_3 and t_1, t_2 and t_3 have the same meanings as in 4.4.1.

Calculate the arithmetic mean of the three values, i.e. the flow rate q_m, in grams per second, corresponding to the test pressure.

5.2 Calculation of shear rates and apparent viscosities

For each of the values of flow rate q_m (calculated as indicated in 5.1), calculate the shear rate and apparent viscosity using the formulae shown in Table 2 (see Notes 1 and 2):

Table 2 — Formulæ for shear rate and apparent viscosity

Quantity	Any die	Die A	Die B
Shear rate, ε (s^{-1})	$\frac{4q_m}{\pi r^3 \rho}$	$377 \frac{q_m}{\rho}$	
Apparent viscosity, η_{app} (Pa·s)	$10^3 \frac{\pi r^4 p \rho}{8 h q_m}$	$44 \times 10^{-3} \frac{p \rho}{q_m}$	$88 \times 10^{-3} \frac{p \rho}{q_m}$