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Plastics — Polyvinyl chloride pastes — Determination of apparent viscosity using the Severs rheometer

Plastiques — Pâtes de polychlorure de vinyle — Détermination de la viscosité apparente au rhéomètre Severs

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ISO 4575-1978 (E)

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 4575 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in November 1976.

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

Austria	Iran	Romania
Belgium	Italy	Spain
Brazil	Japan	Sweden
Bulgaria	Korea, Rep. of	Switzerland
Czechoslovakia	Mexico	Turkey
Finland	Netherlands	United Kingdom
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India	Portugal	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Canada
Israel

Plastics – Polyvinyl chloride pastes – Determination of apparent viscosity using the Severs rheometer

1 SCOPE AND FIELD OF APPLICATION

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

It applies in particular to "standard pastes" prepared according to ISO 4612, *Plastics – Polyvinyl chloride paste resins – Preparation of a paste.*[1)]

2 PRINCIPLE

Placing of a sample of the paste in a Severs rheometer and setting of the jacket to a defined temperature.

Measurement of the flow rate of the paste through a calibrated die at different pressures.

Calculation of the shear rate and apparent viscosity for each flow rate corresponding to each of the pressures applied.

Optionally, preparation of a graph of apparent viscosity as a function of the corresponding shear rate.

3 APPARATUS

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

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 must 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 the 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 airtight to the bottom of the measuring vessel. The outer and internal surfaces of the tube are 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 :

Dimension	Die A	Die B
Radius of tube, mm	1,5 ± 0,05	1,5 ± 0,05
Height, mm	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. (Furthermore, its use simplifies the cleaning of the instrument.) A sketch of the piston is given in figure 2.

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.

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, to measure a temperature of 23 ± 0,5 °C.

4 CONDITIONING

The paste may be tested immediately after its preparation or after conditioning at a temperature of 23 °C. The conditioning time shall be indicated in the test report.

1) At present at the stage of draft.

5 PROCEDURE

5.1 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, 160, 250, 400, 630, 1 000, 1 600, 2 500 kPa*

Conduct a test with die A and pressure 2 500 kPa; from the measured flow *D* (6.1), calculate according to 6.2 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}, the tests shall be carried out with die B.

5.2 Introduction of paste, and temperature control

Position the base together with the appropriate die (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).

5.3 Regulation of the test pressure

Start the tests with 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 for regulating the other test pressures, but without opening valve D for pressures above its maximum gauge reading.

5.4 Determination

5.4.1 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.)

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.

5.4.2 Close the valve to atmosphere F and regulate the pressure to its new value. Repeat the test under the conditions specified in 5.4.1.

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

6 CALCULATIONS

6.1 Calculation of flows

For each one of the test pressures, calculate the flows D_1, D_2 and D_3, in grams per second, corresponding to the measurements by the formulae

$$D_1 = \frac{m_1}{t_1} \qquad D_2 = \frac{m_2}{t_2} \qquad D_3 = \frac{m_3}{t_3}$$

where m_1, m_2 and m_3, and t_1, t_2 and t_3 have the significance given in 5.4.1.

Calculate the arithmetic mean of the three values, designating *D* as the flow, in grams per second corresponding to the test pressure.

* 1 kPa = 1 kN/m^2

6.2 Calculation of shear rates and apparent viscosities

For each of the values of flow D (calculated according to 6.1), calculate the shear rate and apparent viscosity using the formulae shown in the following table (see notes 1 and 2) :

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

where

r is the radius of the cylindrical tube of the die, in centimetres;

h is the height of the cylindrical tube of the die, in centimetres;

p is the test pressure in kilopascals;

D is the paste flow at pressure p, in grams per second (calculated according to 6.1);

ϱ is the density of the paste at 23 °C, in grams per cubic centimetre.

NOTES

1 Strictly these formulae are applicable to Newtonian fluids. Nevertheless they have been applied to PVC pastes because they are easy to use and the values determined are sufficiently accurate for the purpose intended. The exact formulae are given in the annex for information.

2 The shear force, σ, is equal to $\eta\epsilon$ and can be calculated for each test pressure by the formulae $\sigma = p\,r/2\,h$.

7 EXPRESSION OF RESULTS

Indicate the apparent viscosities for the corresponding shear rates in the form of a table (see figure 4).

Plot a graph showing the apparent viscosity (ordinate) versus the shear rate (abscissa). An example of the curve is given in figure 4.

8 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the PVC paste polymer and plasticizer;

c) the formula of the paste;

d) the density of the paste;

e) the die used;

f) the conditioning time at 23 °C before measurement;

g) a table of apparent viscosities at the various shear rates used;

h) optionally, a graph of apparent viscosity versus shear rate.

ANNEX

FORMULAE FOR NON-NEWTONIAN FLUIDS

The exact formulae for non-Newtonian fluids include the term m, the reciprocal of the non-Newtonian symbol n, and are as follows :

Quantity	Any die
Shear rate, ϵ s^{-1}	$\frac{(m+3)\,D}{\pi\,r^3\,\varrho}$
Apparent viscosity, η_{app} Pa·s	$10^3\,\frac{\pi\,r^4\,p\,\varrho}{2\,h\,(m+3)\,D}$

in which the symbols (other than m) have the same meaning and are expressed in the same units as given in 6.2.

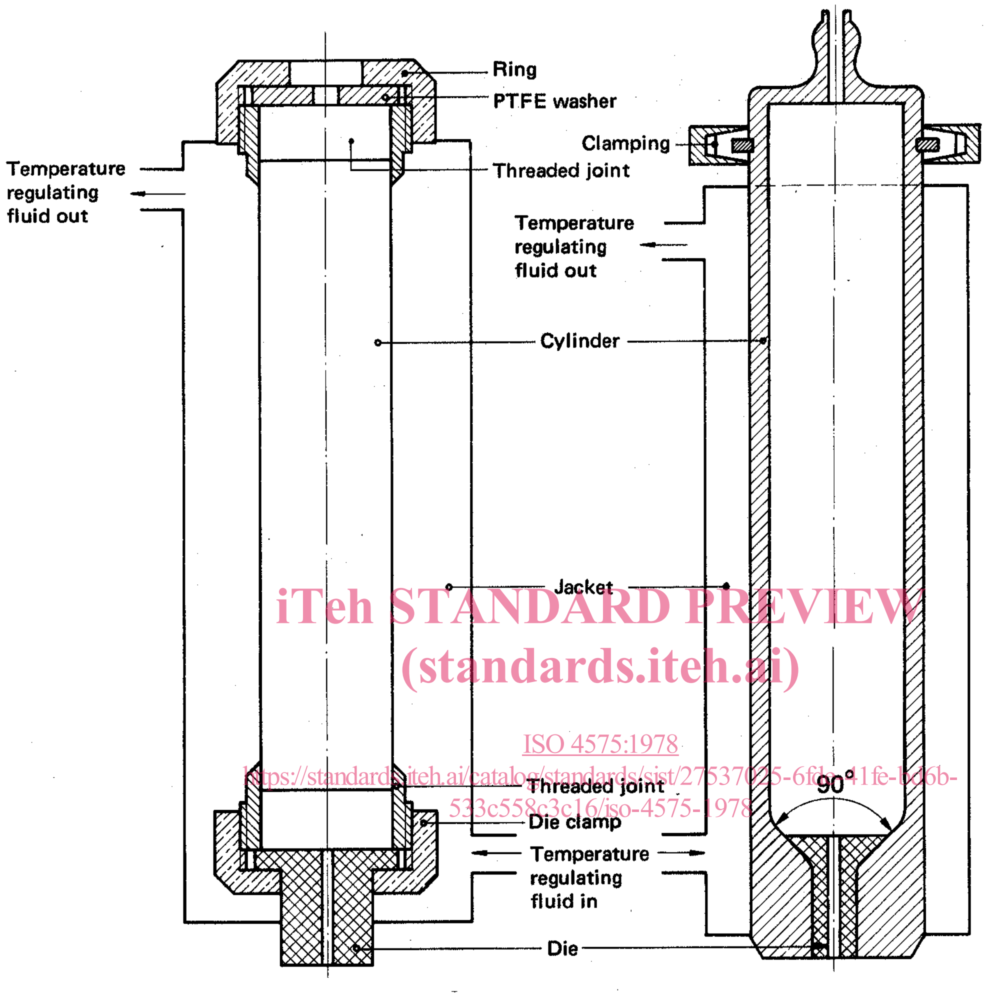


FIGURE 1 — **Diagram showing basic principles of two types of Severs rheometer**

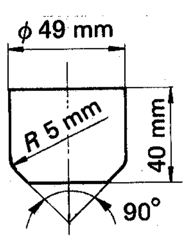
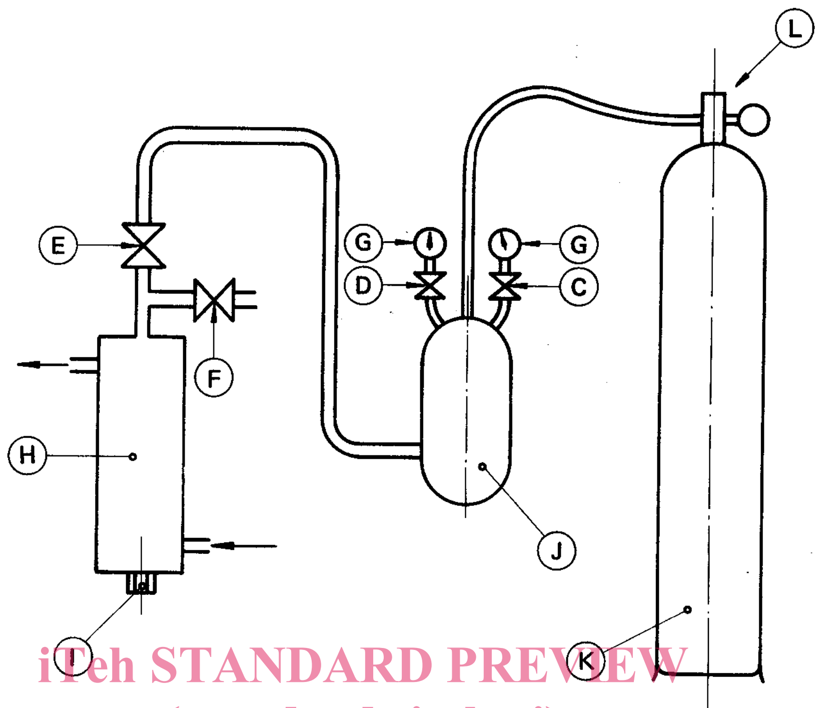


FIGURE 2 — **Piston**



C, D	Valves
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**

Tests with the Severs rheometer

PVC paste "100 and 50"

ϱ_{23} = 1,22 g/cm³

Die A

Immediate measurement

p	kPa	100	400	630	1 000
D	g/s	0,32	1,40	2,38	4,44
ϵ	s^{-1}	98	430	730	1 360
η_{app}	Pa·s	16,9	15,4	14,3	12,2

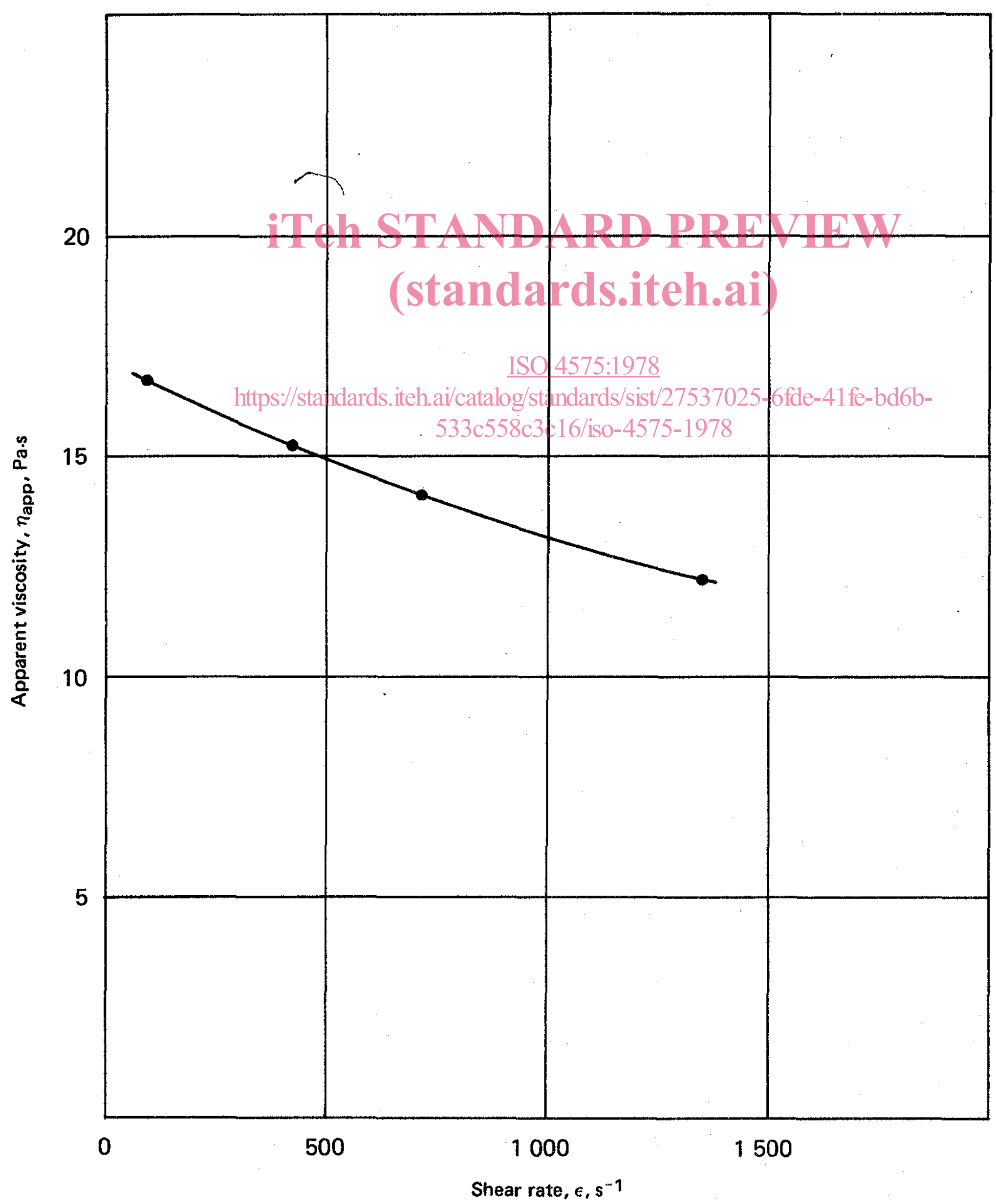


FIGURE 4 — **Examples of presentation in tabular form and graphically of results obtained using the Severs rheometer**