INTERNATIONAL STANDARD 174

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

Plastics — Determination of viscosity number of PVC resins in dilute solution

Matières plastiques — Détermination de l'indice de viscosité des résines de polychlorure de vinyle en solution diluée

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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 174 was drawn up by Technical Committee ISO/TC 61, *Plastics,* and circulated to the Member Bodies in April 1972.

It has been approved by the Member Bodies of the following countries:

Australia	Ireland	Sweden
Austria	Israel	Switzerland
Belgium	Italy	Thailand
Canada	Japan	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Poland	U.S.S.R.
Germany	Portugal	
Hungary	Romania	
India	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 174-1961.

Printed in Switzerland

Plastics — Determination of viscosity number of PVC resins in dilute solution

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the viscosity number of dilute solutions, in cyclohexanone, of PVC resins or copolymers in which the principal constituent is vinyl chloride.

The viscosity number thus determined gives an approximation of the molecular mass of the PVC resin.

NOTE – For the definition of viscosity number, and for other terms, definitions and formulae, see ISO/R 1628, *Plastics – Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers.*

2 PRINCIPLE

Preparation of a solution of 0,005 g/ml of resin in cyclohexanone according to defined conditions.

Measurement of the time of flow of this solution and of the solvent, using a suspended level capillary viscometer.

Calculation from these measurements of the viscosity number.

In this method, corrections for differences in density and kinetic energy are very small and are not applied.

3 SOLVENT

Cyclohexanone, pure, distilled as indicated below *less than two weeks before use,* with a kinematic viscosity of between 2,06 and 2,14 cSt at 25 °C.

Prepare the solvent by distilling pure cyclohexanone at a pressure of 1 013 mbar and collecting only the fraction boiling between 155,0 and 156,0 °C.

Measure with an Ubbelohde viscometer the kinematic viscosity (or the ratio of viscosity/density) of the purified cyclohexanone : it is normally 2,10 cSt at 25 °C but may vary between 2,06 and 2,14 cSt ($2{,}06 \times 10^{-6}$ and $2{,}14 \times 10^{-6}\ m^2/s$).

Store this solvent in the dark in a dark coloured bottle fitted with a ground glass stopper.

Before use, test the condition of the solvent by remeasuring the kinematic viscosity, which must remain between 2,06 and 2,14 cSt.

4 APPARATUS

4.1 either :

a) for *method 1,* **measuring flask,** capacity 50 ml, with ground stopper, or

b) for *method 2,* **flat-bottomed flask,** capacity 150 ml with ground stopper and automatic pipette, capacity 50 ml.

4.2 Filter funnel of fritted glass with medium porosity (pore sizes 40 to 50 μm).

4.3 Thermostatic water bath, capable of being maintained at 25 ± 0,05 °C.

4.4 Mechanical agitator matched with a heating device to keep the 50 ml one-mark flask and its contents (or the 150 ml flask and its contents) at a temperature of 80 to 85 °C.

As a substitute, a shaking or turning agitator may be installed in an oven maintained at 80 to 85 °C.

4.5 Viscometer, suspended level Ubbelohde type, the essential dimensions of which are given in the figure, or any other viscometer with capillary tube of suspended level known to give identical results.

In the figure, two filling marks are shown on the chamber below tube 1. These marks are not always indicated on commercial viscometers; filling must be in such a way that a space remains under the capillary.

4.6 Balance, accurate to 0,1 mg.

4.7 Chronometer, accurate to 0,1 s.

5 SAMPLE

Select a sample which is representative of the material to be tested.

Treat the sample in accordance with the clearly defined requirements agreed by the interested parties (for example drying, washing, etc.).

6 PROCEDURE

6.1 Preparation of the solution

Prepare a solution of PVC in cyclohexanone with a concentration of 5 g of PVC per litre of solution, using one of the following methods.

6.1.1 *Method 1*

Weigh, to the nearest 0,000 2 g, 0,250 ± 0,005 g of resin and pour[1] it quantitatively into the 50 ml measuring flask.

NOTE – For simplicity of calculation, it is possible to weigh an amount of resin of 0,250 0 ± 0,000 2 g directly.

Add about 40 ml of cyclohexanone, *swirling gently by hand* in order to avoid coagulation or the formation of lumps.

Continue dissolving under mechanical agitation for 1 h, maintaining the temperature at 80 to 85 °C.

Make sure that dissolution is at least visually complete.

If gelatinized particles are visible, start again with a new sample until the required result is obtained.

Cool the solution to 25 ± 1 °C and dilute to the mark with the cyclohexanone at the same temperature; mix the solution thoroughly by snaking.

6.1.2 *Method 2*

Weigh, to the nearest 0,000 2 g, 0,250 ± 0,005 g of resin and pour[1] it quantitatively into the 150 ml flask.

NOTE – To simplify calculation it is possible to weigh an amount of resin of 0,251 0 ± 0,000 2 g. The total volume prepared will be 50,18 ml and this amount of PVC resin corresponds to a solution of 0,250 0 g of PVC in 50 ml of solution.

Add, using the automatic pipette kept at 25 ± 1 °C, 50 ml of cyclohexanone, *swirling gently by hand* in order to avoid coagulation or the formation of lumps.

Continue dissolving under mechanical agitation for 1 h, maintaining the temperature at 80 to 85 °C.

Make sure that dissolution is at least visually complete.

If gelatinized particles are visible, start again with a new sample until the desired result is obtained.

Cool to room temperature.

6.2 Cleaning of viscometer

Serious errors in viscosity measurement can arise from minor traces of foreign matter in the capillary tube of the viscometer and, therefore, scrupulous cleanliness is essential. Variations of greater than 0,2 s between repeat measurements indicate the presence of foreign matter but, if this is attached to the capillary wall, its presence can only be detected *by comparison measurements made with a second similar viscometer.*

Before initial use, or after conflicting readings, and at regular intervals clean the viscometer with a mixture of equal volumes of concentrated sulphuric acid and a saturated solution of potassium dichromate in water, *introduced by means of the filter funnel;* leave the mixture to act for several hours, rinse with water, then acetone, likewise introduced by the funnel, and dry the viscosimeter by passing through it a *dust-free* current of air. Between succeeding successful determinations, rinse the viscometer with acetone and dry it as indicated above.

6.3 Measurement of flow time of solvent

NOTE – The procedure described should be suitably modified if a viscometer other than an Ubbelohde type is used.

Immerse the viscometer in the water bath maintained at 25 ± 0,05 °C. Fix the viscometer in such a way that the level of the bath is at least 20 mm above the top graduation line and the axis of tube 2 is perfectly vertical.

By means of the filter funnel, filter the solvent into tube 1 of the viscometer until, after flowing down, the level establishes itself between the two filling marks.

1) Obviously it is always possible to weigh the resin directly into the flask.

INTERNATIONAL STANDARD ISO 174-1974 (E)/ERRATUM

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Plastics — Determination of viscosity number of PVC resins in dilute solution

ERRATUM

Outside front cover

a) Replace the English title by :

"Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of viscosity number in dilute solution".

b) Replace the French sub-title by :

"Plastiques — Résines d'homopolymères et de copolymères de chlorure de vinyle — Détermination de l'indice de viscosité en solution diluée".

Page 1

Replace the title by :

"Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of viscosity number in dilute solution".

After waiting at least 10 min, transfer the liquid into tube 2 of the viscometer, either by blowing dust-free air into tube 1 or by suction on tube 2 and obstructing the orifice of tube 3. Stop the operation when the liquid reaches the centre of the upper bulb of tube 2.

Then close tube 2 with a finger tip and release the other two tubes. When the end of the capillary tube is free of liquid, release the finger tip from tube 2 and measure the interval of time necessary for the passage of the meniscus from one graduation line to the other.

Blow back or suck the liquid again (as above) into the main tube and repeat the measurement. If the measurements differ by more than 0,2 s, repeat the determinations with fresh solvent; if acceptable agreement is not obtained on the repeat determination, reclean the viscometer.

Empty the viscometer by letting the liquid run out through tube 1.

Then rinse the viscometer in acetone and dry it as indicated in the last paragraph of 6.2.

For verification, the measurement of flow time of the solvent is to be taken both before and after the measurement of flow time of the solution.

6.4 Measurement of flow time of solution

NOTE – The procedure described should be suitably modified if a viscometer other than an Ubbelohde type is used.

Using the same viscometer as for the measurement of flow time of the solvent, take the reading in exactly the same way as when working with the solvent. After each measurement, empty the viscometer by letting the solution flow through tube 1. Rinse it with the cyclohexanone, through the capillary, then drain by tube 1. Then rinse the viscometer with acetone and dry it as indicated in the last paragraph of 6.2.

7 EXPRESSION OF RESULTS

The viscosity number, expressed in millilitres per gram, is calculated from the formula :

$$\frac{t_s - t_o}{t_o C}$$

where

t_o is the arithmetic mean of the two flow times of the solvent;

t_s is the arithmetic mean of the two flow times of the solution;

C is the concentration, in grams of resin per millilitre of solution.

For *method 1* (6.1.1) $C = \frac{m}{50}$

For *method 2* (6.1.2) $C = \frac{m}{50,18}$

Round off the calculated viscosity number to the nearest 0,5.

8 TEST REPORT

The test report shall include the following information :

a) the reference of the method used; (for example : Method 1 of ISO 174)

b) complete identification of the sample;

c) an exact description of any treatment undergone by the sample before it was dissolved;

d) any difference between the type of viscometer used and that described in this International Standard;

e) any incidents which may have influenced the result;

f) date of test.

Dimensions in millimetres

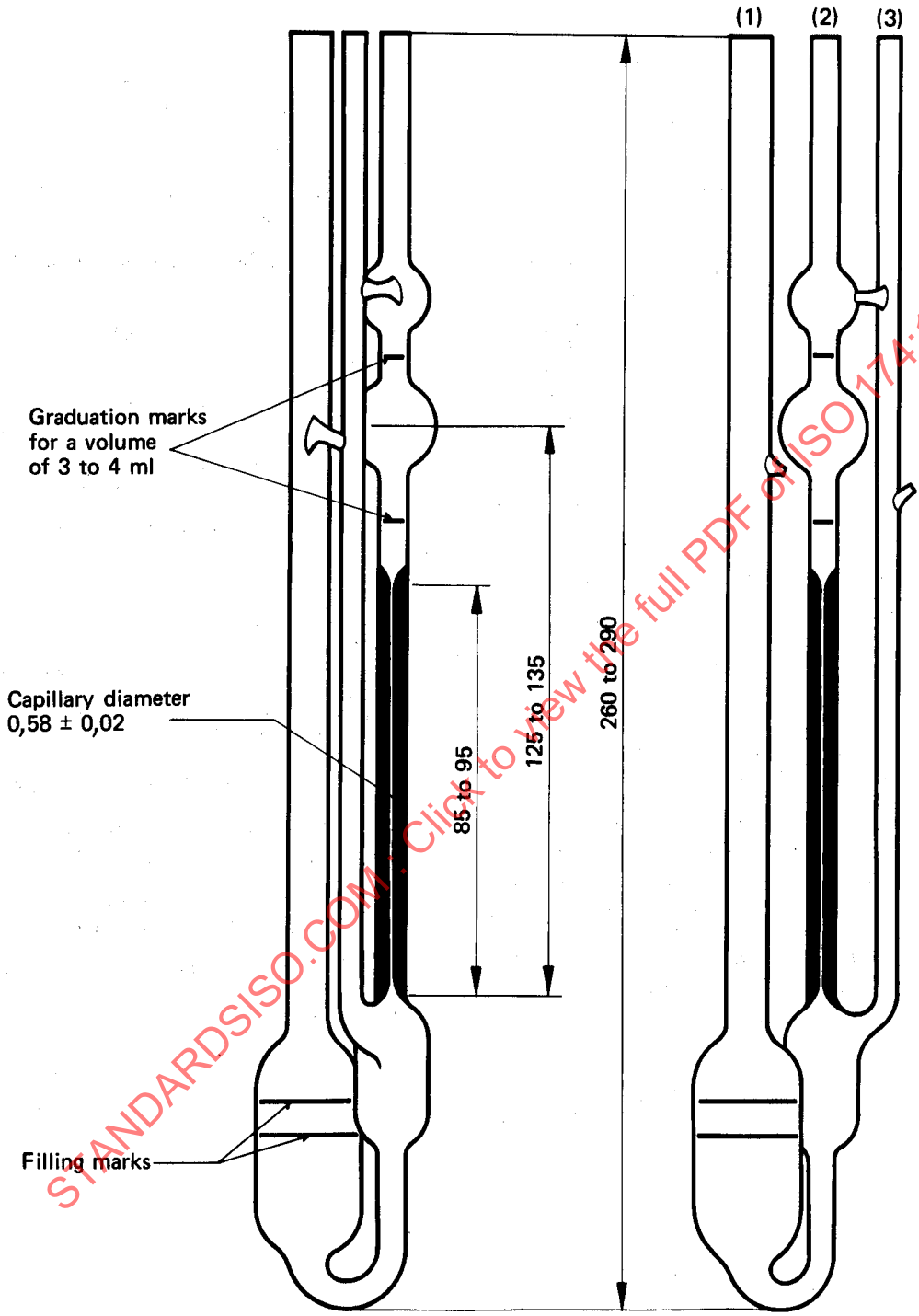

FIGURE — **Ubbelohde viscometer**

NOTE — The viscosimeter shown in the diagram conforms to model C of ISO/R 1628 and the dimensions of the capillary agree with the description of ISO/R 174. It gives a flow time of the order 3 to 6 min. The time may be reduced without significantly altering the precision of the determination by using a viscometer of the same dimensions except that the volume of the reservoir is smaller (1,5 to 2 ml).