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Plastics — Determination of thermal stability of poly(vinyl chloride), related chlorine-containing homopolymers and copolymers and their compounds — Discoloration method

*Plastiques — Détermination de la stabilité thermique du poly(chlorure
de vinyle), des homopolymères et copolymères chlorés apparentés et de
leurs compositions — Méthode du changement de couleur*



Reference number
ISO 305:1990(E)

Foreword

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International Standard ISO 305 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 305:1976), of which it constitutes a technical revision.

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Plastics — Determination of thermal stability of poly(vinyl chloride), related chlorine-containing homopolymers and copolymers and their compounds — Discoloration method

1 Scope

1.1 This International Standard specifies two methods for the determination of the thermal stability of products and compounds based on vinyl chloride homopolymers and copolymers (referred to simply as PVC in the following text) by the extent of the discoloration that occurs when they are exposed, in the form of sheet, to elevated temperatures.

- Method A: Oil-bath method
- Method B: Oven method

1.2 These methods are particularly applicable to the determination of the resistance of PVC to degradation by heat, as assessed by the change in colour after different times of heating under standardized conditions. The results are comparative only, and may be unsatisfactory when coloured PVC materials are tested.

1.3 Method A is a simple method, which requires little expenditure on apparatus and permits materials to be tested almost in the absence of air.

Method B requires a forced-air oven, in which the air flow is adjusted to provide a sufficiently uniform temperature throughout the entire test area. This method is not applicable to materials that will cross-contaminate during oven exposure.

The stability times given by the two methods may not be similar and cannot be used for direct-comparison purposes.

2 Principle

2.1 Method A: Oil-bath method

A series of test specimens is heated at an elevated temperature for different lengths of time in a temperature-controlled oil bath. The test specimens are placed between an aluminium block and an aluminium cylinder to promote heat transfer and restrict air access.

2.2 Method B: Oven method

A series of test specimens is heated at an elevated temperature for different lengths of time in a forced-air-circulation oven. The test specimens are supported by new, clean aluminium foil laid on removable racks.

3 Preparation and number of test specimens

3.1 The test specimens shall consist of

- discs of diameter 14 mm and thickness approximately 1 mm, for method A;
- squares of side 15 mm and thickness approximately 1 mm, for method B.

They shall be punched out from the sheets to be tested.

3.2 The number of test specimens required is the expected time of the test in minutes divided by 5. If the stability of the compound is very high, remove the test specimens every 10 to 15 minutes instead of every 5 minutes during the first stage of heating, before the appearance of discoloration. Thus, the number of test specimens used can be less than that specified above.

3.3 If the material to be tested is an extrusion or moulding material in granule, powder or pellet form, this shall be sheeted on a roll-mill under the conditions specified in the material specification, or as agreed upon between the interested parties (ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials*, may be helpful in this respect).

3.4 If the material to be tested is in the form of a paste (plastisol), it shall be gelled to give a well fused sheet; the test specimens shall be punched out from the sheet thus obtained.

NOTES

1 If the surface finish of specimens (particularly those made from unplasticized materials) prepared by milling does not ensure sufficient contact with the aluminium block and cylinder, press-polishing of the specimens is recommended as an optional additional step.

2 Warming of the milled unplasticized sheets will permit test specimens to be cut without shattering.

4 Test temperature

The test temperature shall be that stated in the material specification or as agreed upon between the interested parties; in the latter case, the temperature shall be chosen so that the test duration is in the range 60 min to 120 min. If there is no specification or agreement, a temperature of 180 °C shall be used.

5 Method A: Oil-bath method

5.1 Apparatus

5.1.1 Thermostatically controlled oil bath, capable of maintaining the temperature within $\pm 0,5$ °C in the range 120 °C to 200 °C, fitted with a suitable stirrer and a suitable device for holding a convenient number of test tubes immersed to a depth of 60 mm to 70 mm.

5.1.2 Glass test tubes, of the following dimensions:

- external diameter: 18 mm $\pm$ 0,4 mm;
- wall thickness: 1,2 mm $\pm$ 0,2 mm;
- length: 150 mm minimum.

5.1.3 Aluminium blocks, as shown in figure 1.

Dimensions in millimetres

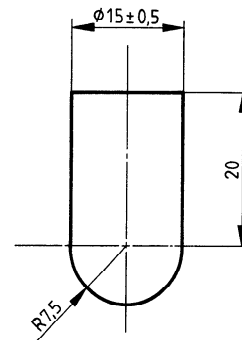


Figure 1 — Aluminium block

5.1.4 Aluminium cylinders, with a diameter of 15 mm $\pm$ 0,5 mm and a height of 30 mm.

5.1.5 Timing device, for example a stopwatch, with an accuracy of 0,5 min or better.

5.2 Procedure

5.2.1 Prepare a sufficient number of test tubes (5.1.2) and place an aluminium block (5.1.3) in each tube; then insert a test specimen and cover it with an aluminium cylinder (5.1.4).

5.2.2 Place the test tubes vertically in the oil bath (5.1.1) that has been brought to within 0,5 °C of the specified or agreed temperature, and start the timing device (5.1.5).

5.2.3 Every 5 min take a test tube from the bath. Remove the test specimen from the tube and allow it to cool, using slight pressure, if necessary, to prevent deformation. Number the test specimens consecutively.

5.2.4 Fasten the test specimens on a card with a note of the exposure time in minutes of each specimen and the test temperature.

WARNING — In order to make handling of the test tubes, which are covered with hot oil, safer after they have been removed from the oil bath, an optional drainage and handling time can be agreed upon between the interested parties. Use tongs or another suitable device to hold the tubes when tipping out the aluminium blocks.

6 Method B: Oven method

6.1 Apparatus

6.1.1 Forced-air-circulation oven, having the following additional features:

6.1.1.1 The temperature of the oven shall be controlled by a thermostat capable of maintaining it to within $\pm 0,5$ °C of the temperature selected.

6.1.1.2 The oven shall be equipped with a calibrated thermometer and the proper system correction shall be applied to the temperature measurement.

6.1.1.3 The air flow in the oven shall be adjusted to provide a temperature which is sufficiently uniform throughout the test area of the oven that specimens of uniform colour are produced (see 6.1.2, second paragraph) This will usually require an air flow of $0,3 \text{ m}^3/\text{min}$.

6.1.1.4 Prior to the test, the uniformity of the temperature within the oven shall be verified. Temperature uniformity is usually verified by placing thermocouples near each corner and at the centre; checks are made at 5 min intervals. It may also be done by inserting, at the test temperature, a rack with 8 to 10 specimens from the same sample distributed over the test area and exposing them until an early stage of discoloration is reached. The formulation shall be such as to cause a sharp change in colour within 45 min to 60 min under the test conditions. A non-uniform temperature distribution, as indicated by differences in the colours of the exposed specimens, shall be corrected before proceeding with the test.

6.1.1.5 The inside of the oven shall be free of contamination and surface deposits. Use stainless-steel oven liners to reduce corrosion due to extended exposure to decomposition gases.

6.1.2 Specimen supports, made of new, clean aluminium foil laid on a removable oven rack (grille).

If an oven equipped with a rotating specimen-holder is employed, the specimens shall be supported so that no appreciable elongation or necking down of the test specimens occurs during the test, since this would alter the specimen dimensions, and especially the thickness. In this case, the uniformity of the oven temperature shall be checked with the specimen holders mounted on the rotating device.

6.1.3 Timing device, for example a stopwatch, with an accuracy of 0,5 min or better.

6.2 Procedure

6.2.1 Prepare, for each exposure time envisaged, a specimen support (6.1.2) of sufficient size to hold one specimen of each of the different compositions under test.

6.2.2 Place one specimen of each composition on each of the specimen supports.

6.2.3 Place all the supports on the rack.

6.2.4 Place the rack in the oven (6.1.1) at the test temperature, keeping the oven door open for the minimum time necessary. The oven air-circulation fan shall be off while the door is open.

6.2.5 Start the timing device (6.1.3).

6.2.6 Remove one support together with its specimens, at selected intervals over the period of exposure, preferably continuing exposure until blackening occurs. Number the specimens consecutively.

6.2.7 Fasten the test specimens on a card with a note of the exposure time in minutes of each specimen and the test temperature.

7 Expression of results

Note the times, in minutes, from the start of the test to

- the first observed change in colour and
- the end of the test.

NOTE 3 In cases of dispute, when greater precision is required, it is recommended that test specimens be compared by means of an agreed colour scale or by means of a photometer.

8 Precision

The precision of this test method is not known because inter-laboratory data are not available. This method may not be suitable for use in specifications or in case of disputed results until these data are available.

9 Test report

The test report shall include the following information:

- a) a reference to this International Standard;

- b) the method used (A or B);
- c) all details necessary for the complete identification of the sample, including the composition of the compound and the method of preparation of the test specimens (for example thermal treatment);
- d) the test temperature;
- e) the times, in minutes, from the start of the test to

- the first observed change in colour and
- the end of the test;

- f) the date of the test.

The test report shall be accompanied by a piece of an untreated test specimen and the complete series of treated test specimens fastened on a card (it is essential that this be stored in the dark).

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