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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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 305:1990), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— editorial changes have been applied to align the document with the ISO structure;

— Clauses 2 and 3 have been added and subsequent clauses have been renumbered.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document 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. The two methods are:

— Method A: Oil-bath method;

— Method B: Oven method.

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 can be unsatisfactory when coloured PVC materials are tested.

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

4.1 Method A: Oil-bath method

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

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.

4.2 Method B: Oven method

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.

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.

5 Preparation and number of test specimens

5.1 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.

5.2 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 min to 15 min instead of every 5 min 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.

5.3 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 may be helpful in this respect).

5.4 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.

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.

Warming of the milled unplasticized sheets permits test specimens to be cut without shattering.

6 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 of 60 min to 120 min. If there is no specification or agreement, a temperature of 180 °C shall be used.

7 Method A: Oil-bath method

7.1 Apparatus

7.1.1 Thermostatically controlled oil bath, capable of maintaining the temperature within ±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.

7.1.2 Glass test tubes, of the following dimensions:

— external diameter: 18 mm ± 0,4 mm;

— wall thickness: 1,2 mm ± 0,2 mm;

— length: 150 mm minimum.

7.1.3 Aluminium blocks, as shown in Figure 1.

Dimensions in millimetres

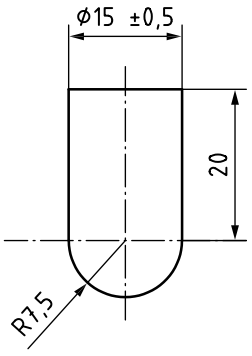

Figure 1 — Aluminium block

7.1.4 Aluminium cylinders, with a diameter of 15 mm ± 0,5 mm and a height of 30 mm.

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

7.2 Procedure

7.2.1 Prepare a sufficient number of test tubes (7.1.2) and place an aluminium block (7.1.3) in each tube; then insert a test specimen and cover it with an aluminium cylinder (7.1.4).

7.2.2 Place the test tubes vertically in the oil bath (7.1.1) that has been brought to within 0,5 °C of the specified or agreed temperature and start the timing device (7.1.5).

7.2.3 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.

7.2.4 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.

8 Method B: Oven method

8.1 Apparatus

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

8.1.1.1 The temperature of the oven shall be controlled by a thermostat capable of maintaining it to within ±0,5 °C of the temperature selected.

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

8.1.1.3 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 8.1.2, second paragraph). This will usually require an air flow of 0,3 m^3/min.

8.1.1.4 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.

8.1.1.5 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.

8.1.2 **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.

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

8.2 Procedure

8.2.1 Prepare, for each exposure time envisaged, a specimen support (8.1.2) of sufficient size to hold one specimen of each of the different compositions under test.

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

8.2.3 Place all the supports on the rack.

8.2.4 Place the rack in the oven (8.1.1) 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.

8.2.5 Start the timing device (see 8.1.3).

8.2.6 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.

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

9 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.

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.

10 Precision

The precision of this test method is not known because interlaboratory 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.

11 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 305:2019;

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).