
Plastics — Determination of migration of plasticizers

Plastiques — Détermination de la migration des plastifiants

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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 on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 61 *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This third edition cancels and replaces the second edition (ISO 177:1988), of which it constitutes a minor revision with the following change:

- the normative references have been updated.

Plastics — Determination of migration of plasticizers

1 Scope

This document specifies a method for the determination of the tendency of plasticizers to migrate from plastics in which they are contained into other materials or other plastics when they are brought into close contact.

NOTE 1 The surfaces into which the migration can proceed can also consist of organic surface coatings, such as lacquers.

This test is suitable

- a) for evaluating the tendency displayed by plastics, particularly in the form of films and sheets, to lose certain of their liquid constituents when they are brought into contact with materials that have an affinity for plasticizers, and
- b) for studying the tendency to migrate of plasticizers contained in a resin or a series of resins, in one or more concentrations.

In case b), standard compounds are prepared on the basis of a well-characterized resin with well-defined ratios of plasticizer to resin.

NOTE 2 When the absorbent sheets themselves contain a substance capable of migrating, simultaneous migrations can occur from the test specimens into the absorbent sheets and vice versa.

The results may also be affected by the migration of other constituents of the plastic material (for example, oligomers) or by the loss of any volatile constituents other than plasticizers from the plastic material or the absorbent layer.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles and moulding of multipurpose and bar test specimens*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1 migration of plasticizers

loss of mass of a sheet of plasticized plastic when placed in close contact between two absorbent sheets of another material, under specified conditions

4 Principle

A test specimen cut from a sheet or plate of the material or from the finished product to be tested is placed in close contact with two sheets, capable of absorbing plasticizers. It is then subjected to heating under defined conditions. The loss in mass of the test specimen, theoretically equal to the increase in mass of the sheets, is a measure of the migration of the plasticizer.

5 Apparatus

5.1 **Analytical balance**, accurate to 0,001 g.

5.2 **Micrometer**, accurate to 0,01 mm.

5.3 **Air circulating oven**, capable of maintaining the temperature to within ± 2 °C, in the range 50 °C to 100 °C.

5.4 **Glass plates**, with plane surfaces, of sufficient size to cover the absorbent backing discs (5.6).

5.5 **Weights**, of 5 kg.

5.6 **Absorbent backing discs**, with an affinity for plasticizers, 60 mm $\pm$ 1 mm in diameter and at least 0,5 mm in thickness.

The materials recommended for the test are:

- a standard rubber (according to ISO 4649), or
- polyethylene without additive, or
- polyvinyl acetate without plasticizer.

In the case of a particular application, an absorbent backing disc of the material with which the plastic under test will be in contact when in service shall be used. The surface of the absorbent backing disc shall be sufficiently smooth to ensure continuous contact with the test specimen.

6 Test specimens

6.1 The test specimens shall be in the form of discs 50 mm $\pm$ 1 mm in diameter and at least 0,5 mm in thickness, cut from a compression-moulded (in accordance with ISO 293) or injection-moulded (in accordance with ISO 294-1 and ISO 294-3) sheet of suitable thickness.

The surface of the test specimens shall be sufficiently smooth to ensure continuous contact with the absorbent backing discs (5.6).

6.2 In the case of films, the test specimen of not less than 0,5 mm thickness shall be produced by pressing an adequate number of films at a suitable temperature for about 1 min.

6.3 If the test is intended to determine the migration from a finished product, the latter shall be tested at a uniform thickness.

6.4 If the product to be tested consists of a support (fabric, paper or other suitable material), coated on one face only by the spreading or calendering of a plasticized resin (such as fabrics coated with vinyl resins or with similar products), the test specimens shall consist of two discs cut from the material itself and superposed in such a way that the uncoated surfaces of the support are touching and the plastic is situated on the other faces of the sandwich so formed.

6.5 Three test specimens shall be tested for each material.

7 Conditioning

Unless otherwise specified, the test specimens and absorbent backing discs (5.6) shall be conditioned in one of the atmospheres specified in ISO 291.

8 Procedure

8.1 After conditioning, weigh the test specimens (see Clause 6) and the discs (5.6) to the nearest 0,001 g and determine their mean thickness to the nearest 0,01 mm.

8.2 Place a test specimen of the material under test between two absorbent backing discs, in such a way that their axes coincide and the assembly forms a sandwich. Place the latter between two glass plates (5.4).

8.3 If the test is intended to determine the characteristics of particular plasticizers, standard compounds of a specified composition shall be used, as agreed between the interested parties.

8.4 To equalize the distribution of pressure between the absorbent backing disc and the test specimen, a sheet of rubber shall be inserted between the absorbent backing disc and the glass plate. The absorbent backing disc shall be separated from the sheet of rubber by aluminium foil.

8.5 Place one of the 5 kg weights (5.5) on the assembly of discs and plates to be tested and place the assembly in the oven (5.3), maintained at a temperature of $70\text{ °C} \pm 2\text{ °C}$.

It is permissible to superimpose several assemblies, but five at most, separated by glass plates, thus forming a column loaded with a single 5 kg weight. In this event, the temperature shall be measured directly at the interior of the stack, for example, between an absorbent backing disc and the sheet of rubber inserted to equalize the pressure.

When it is required to take account of the service temperature of the material, the test should be conducted at the temperature recommended in the specification for the application of the material. In the absence of any particular specification, if it is desired to perform a test at a temperature either lower or higher than 70 °C , 50 °C or 85 °C , respectively, should be used.

8.6 Fresh absorbent backing discs shall be used for each test.

8.7 After 24 h, remove the assemblies from the oven. Separate the test specimens from the absorbent backing discs and recondition all of them under the same conditions as those applied prior to the initial weighing. Reweigh the test specimens and the absorbent backing discs.

8.8 In order to determine the progress of migration as a function of time, it is suggested that measurements be effected after test periods of 1 d, 2 d, 5 d, 10 d, 15 d and 30 d. In such cases, three test specimens shall be used for each test duration.

9 Expression of results

Calculate the arithmetic means of the changes in mass in grams:

- a) of the three test specimens;
- b) of the three sets of two discs.

If the maximum spread of the results relating to a series of three test specimens or to a series of three pairs of discs differs by $\pm 10\%$ relative to the value of the mean of the three respective results, repeat the test on a new series of three test specimens.

The migration of the plasticizer in the product under test is expressed by the above two numbers taken in sequence, for example:

ISO 177 plasticizer migration: mean for test specimens 0,450 g; mean for pairs of absorbent discs 0,380 g.

The loss in mass of the test specimen should be, theoretically, equal to the gain in mass of the two absorbent backing discs. This is, however, very rarely the case; the observed difference is probably due to volatile loss.

10 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 177;
- b) a complete identification of the sample and the method of preparing the test specimens;
- c) the chemical composition and designation of the absorbent backing discs;
- d) the conditioning procedure;
- e) the dimensions of the test specimens and of the absorbent backing discs (the thickness shall be reported to the nearest 0,01 mm);
- f) the mass, in grams, of the test specimens and of the absorbent backing discs before the test;
- g) the loss or gain of mass, in milligrams, of each test specimen and of the corresponding pair of absorbent backing discs, and the test duration;
- h) the arithmetic mean of the values obtained for three test specimens and for the corresponding three pairs of absorbent backing discs;
- i) observations concerning any changes occurring in the appearance of the test specimens.