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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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 177 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 177 : 1976), of which it constitutes a technical revision.

Plastics — Determination of migration of plasticizers

1 Scope

This International Standard 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 may proceed may 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;
- 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 should be 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 may 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291 : 1977, *Plastics — Standard atmospheres for conditioning and testing*.

ISO 293 : 1986, *Plastics — Compression moulding test specimens of thermoplastic materials*.

ISO 294 : 1975, *Plastics — Injection moulding test specimens of thermoplastic materials*.

ISO 4649 : 1985, *Rubber — Determination of abrasion resistance using a rotating cylindrical drum device*.

3 Definition

For the purposes of this International Standard, the following definition applies.

migration of plasticizers: The 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 $\pm 2^\circ\text{C}$, in the range 50 to 100 $^\circ\text{C}$.

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.

Materials recommended for the test are :

- a standard rubber (see ISO 4649 : 1985, annex B, clause B.2);
- 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 (see ISO 293) or injection-moulded (see ISO 294) 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 (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 °C $\pm$ 2 °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.

NOTE — 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, 2, 5, 10, 15 and 30 days. 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.

NOTE — 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) reference to this International Standard;
- b) 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.

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