INTERNATIONAL STANDARD 176

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

Plastics — Determination of loss of plasticizers — Activated carbon method

Matières plastiques — Détermination des pertes en plastifiants — Méthode au charbon actif

First edition — 1976-07-15

UDC 678.5/.8 : 543.813

Ref. No. ISO 176-1976 (E)

Descriptors : plastics, tests, measurement, losses, plasticizers.

Price based on 2 pages

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommandations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 176 and found it technically suitable for transformation. International Standard ISO 176 therefore replaces ISO Recommendation R 176-1961 to which it is technically identical.

ISO Recommendation R 176 was approved by the Member Bodies of the following countries :

Australia	Israel	Sweden
Austria	Italy	Switzerland
Belgium	Japan	Turkey
Bulgaria	Netherlands	United Kingdom
Czechoslovakia	Poland	U.S.A.
Germany	Portugal	U.S.S.R.
Hungary	Romania	
India	Spain	

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

France

The Member Bodies of the following countries disapproved the transformation of ISO/R 176 into an International Standard :

France
Netherlands

Printed in Switzerland

Plastics — Determination of loss of plasticizers — Activated carbon method

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies two empirical methods for the quantitative determination of the loss of mass from a plastic material under defined conditions of time and temperature, in the presence of activated carbon.

1.2 These methods are used, in particular, for the quantitative determination of the loss on heating of plasticizers from plasticized plastic materials in which case it is generally assumed that no significant amounts of other volatile materials are present.

1.3 These are empirical test methods, only suitable for a rather rapid comparison of the losses of plasticizers or, in general, of volatile compounds, from different plastics.

1.4 They may also be employed for the comparison of different types of plasticizers; in this case standard compounds should be prepared, on the basis of a well characterized resin, with known ratios of resin to plasticizer.

NOTE — These comparisons are possible only if the test specimens are of the same thickness. If it can be assumed that, after reconditioning, the moisture content of the exposed specimens is equal to that obtaining after the original conditioning, the effect of moisture may be ignored.

1.5 Two methods are specified :

Method A : the test specimens are in direct contact with the carbon; this method is particularly useful for materials which have to be tested at relatively low temperatures because they flow at higher temperatures.

Method B : the test specimens are placed in wire cages which prevent direct contact between the test specimens and the carbon.

2 REFERENCES

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

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

3 APPARATUS AND MATERIALS

3.1 Analytical balance accurate to 0,001 g.

3.2 Micrometer accurate to 0,01 mm.

3.3 Thermostatical bath or **oven** capable of maintaining the temperature to within ± 1 °C of the test temperature, in the range of 50 to 150 °C.

3.4 Containers : Metal cans, of cylindrical form, about 100 mm in diameter and 120 mm in height provided with non-air-tight cover; a lid with a small vent hole of 3 mm diameter may be suitable.

3.5 Cylindrical metal cages, constructed from bronze gauze having apertures of approximately 500 μm, with a diameter of 60 mm and a height of 6 mm, formed by soldering a strip of the gauze at right angles to the periphery of a disk of the gauze; a similar but slightly larger cylinder acts as a lid.

3.6 Activated carbon with a grain size of about 4 to 6 mm, free from powder. The carbon shall be of a well determined type and grade, in order to obtain concordant results.[1)]

Before use, the carbon should be sieved and dried to constant mass at 70 °C, preferably under vacuum, and then stored in an air-tight container. Use fresh material for each test.

4 TEST SPECIMENS

4.1 The test specimens shall be in the form of disks 50 ± 1 mm in diameter and 1 ± 0,1 mm in thickness cut from compression moulded sheet of the appropriate thickness. Attention is drawn to the provisions of ISO 293.

4.2 If the test is carried out for the determination of the characteristics of specific plasticizers, standard compounds of a given composition, as agreed between vendor and purchaser, shall be used.

1) Suitable brands of activated carbon are available commercially. Detailed information may be obtained from the Secretariat of ISO/TC 61 or from the ISO Central Secretariat.

4.3 At least three test specimens shall be tested for each material.

NOTE — For special purposes the use of specimens of different shape and thickness may be necessary. However, a comparison of the values obtained is possible only for specimens of the same thickness.

Coated fabrics and other supported plastics films may be tested by this method using specimens cut directly from the sample as received.

5 CONDITIONING

Unless otherwise specified, test specimens shall be conditioned in one of the atmospheres specified in ISO 291.

6 PROCEDURE

6.1 Method A : Test specimen in direct contact with activated carbon

6.1.1 After conditioning, weigh each test specimen to the nearest 0,001 g and determine its mean thickness to the nearest 0,01 mm.

6.1.2 On the bottom of a metal container (3.4) spread about 120 cm^3 of activated carbon (3.6). Place a test specimen on top of the carbon and cover it with a further 120 cm^3 of carbon. Place two further specimens in the container, each covered by 120 cm^3 of carbon. Finally, put the lid on the container.

6.1.3 Only test specimens of the same composition shall be placed in one container, in order to avoid the possibility of plasticizers or other volatile components migrating from one specimen to another.

6.1.4 Place the container in the oven or thermostatic bath controlled at a temperature of 70 ± 1 °C.

6.1.5 After 24 h remove the container from the oven or bath and allow it to cool at room temperature. Remove the specimens from the container, carefully brush them free from any trace of carbon particles and recondition under the same conditions as those to which they were subjected before the original weighing.

6.1.6 Reweigh each specimen to the nearest 0,001 g.

6.2 Method B : Test specimens in wire cages

The procedure is similar to that used in method A, with the difference that each test specimen is put into a small metal wire-mesh cage (3.5) thus avoiding direct contact between the plastic and the carbon, and that the test temperature is 100 ± 1 °C.

After 24 h, remove the specimens from the container, recondition and reweigh (as specified in 6.1.5 and 6.1.6).

NOTE — For different materials, different temperatures and durations of test may be agreed between the interested parties, maintaining the same test procedure.

7 EXPRESSION OF RESULTS

The change in mass, Δm, expressed as a percentage, is given by the formula :

$$\Delta m = \frac{m_0 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test specimen after conditioning;

m_1 is the mass, in grams, of the test specimen after treatment in the oven or thermostatical bath and reconditioning.

Note the arithmetic mean of the values obtained from the three test specimens as the loss of plasticizers from the material under test.

8 TEST REPORT

The report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the sample and the procedure used for preparing the specimens;

c) thicknesses of each test specimen, to the nearest 0,01 mm;

d) the conditioning procedure used;

e) the test temperature and the duration of the test, and the method employed (i.e. method A or method B);

f) the mass of each test specimen, in grams, before the test and the gain or loss in mass, in milligrams, during the test;

g) the mass change of each test specimen, expressed as a percentage of the original mass (see clause 7);

h) the arithmetic mean of the values obtained from three test specimens;

i) observations on any change in appearance of the test specimens;

j) date of test.