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Plastics — Determination of loss of plasticizers — Activated carbon method

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

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 176 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 176:1976), Clause 8 of which is now Clause 9 and a new Clause 8 has been added.

Plastics — Determination of loss of plasticizers — Activated carbon method

1 Scope

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.

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.

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

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.

Two methods are specified:

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

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

2 Normative references

The following referenced documents are indispensable for the application 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*

3 Apparatus and materials

3.1 Analytical balance, accurate to 0,000 1 g.

3.2 Micrometer, accurate to 0,01 mm.

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

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

3.5 Wire 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 a right angles to the periphery of a disk of the gauze, with a similar but slightly larger cylinder acting as a lid.

3.6 Activated carbon, with a grain-size of about 4 mm 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

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

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.

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

NOTE For special purposes, the use of specimens of different shape and thickness might 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 Place one specimen on the bottom of a metal container (3.4) and spread about 120 cm^3 of activated carbon (3.6) over this specimen. Place two additional 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.

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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.

6.1.4 Place the container in the oven or thermostatic bath controlled at a temperature of 70 °C ± 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 differences (1) 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 (2) that the test temperature is 100 °C ± 1 °C.

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

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

7 Expression of results

The change in mass, Δm, expressed as a percentage is given by Equation (1):

$$\Delta m = \frac{m_0 - m_1}{m_0} \times 100 \qquad (1)$$

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 thermostatic bath and reconditioning.

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

8 Precision

Precision data are shown in Table 1.

Table 1 — Precision data for three different plasticizer materials tested according to method A

Plasticizer material	Volatility	Average loss in 24 h (%)	Within-laboratory standard deviation of the average	Between laboratory standard deviation of the average	Repeatability limit within a single laboratory	Reproducibility limit between laboratories
1	high	19,46	0,70	2,43	1,98	6,88
2	medium	3,83	0,35	0,87	0,98	2,48
3	low	0,81	0,12	0,42	0,35	1,20

NOTE This precision statement is based on tests of three materials conducted by five different laboratories. All samples were prepared by one source, but individual specimens were prepared by the laboratory that tested them. Each test result was the average of 23 individual determinations.

For tests conducted in a single laboratory using the same equipment on the same day, two test specimens should be judged as not equivalent if they differ by more than the repeatability limit within a single laboratory.

For tests conducted by different operators using different equipment on different days, two test results should be judged as not equivalent if they differ by more than the reproducibility limit between laboratories.

Any judgement made according to these criteria would have 95 % probability of being correct.

9 Test report

The report shall include the following information:

a) reference to this International Standard;

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

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

d) conditioning procedure used;

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

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

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

h) 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.

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