

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 176

PLASTICS

DETERMINATION OF THE LOSS OF PLASTICIZERS BY THE ACTIVATED CARBON METHOD

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BRIEF HISTORY

The ISO Recommendation R 176, *Determination of the Loss of Plasticizers by the Activated Carbon Method*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American Standards Association, Incorporated (ASA).

Work on this matter which the Technical Committee had begun since 1954, came to an end in 1956, with the adoption of a proposal as a Draft ISO Recommendation.

On 28 November 1958, the Draft ISO Recommendation (No. 191) was distributed to all the ISO Member Bodies and was approved, subject to some editorial amendments, by the following Member Bodies:

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

One Member Body opposed the approval of the Draft: France.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1961, to accept it as an ISO RECOMMENDATION

PLASTICS**DETERMINATION OF THE LOSS OF PLASTICIZERS
BY THE ACTIVATED CARBON METHOD****1. SCOPE**

- 1.1 This method of test covers, in general, a procedure for the quantitative determination of the loss from a plastic material under defined conditions of time and temperature, in the presence of activated carbon.
- 1.2 This method of test is used, in particular, for the quantitative determination of the loss on heating of plasticizers from plasticized plastics materials.

2. SIGNIFICANCE OF TEST

- 2.1 This is an empirical test method, only suitable for a rather rapid comparison of the losses of plasticizers or, in general, of volatile compounds, from different plastics. This comparison is possible only if the test specimens are of the same thickness.
- 2.2 This method may be employed also for the comparison of different types of plasticizers; in this case standard compounds should be prepared, on the basis of well determined resins, in well defined ratios.
- 2.3 In the cases when it can be assumed that, after reconditioning, the moisture content regained by the specimens is equal to the initial one, the effect of moisture may be considered negligible.

3. APPARATUS AND MATERIALS

- 3.1 *Balance* : accurate analytical balance, to weigh to 0.001 g.
- 3.2 *Micrometer* for measuring 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, in 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 in diameter may be suitable.
- 3.5 *Metal cages* constructed from approximately 30 mesh bronze gauze (about 100 mesh per square centimetre), in cylindrical form, having diameter of 60 mm and height of 6 mm, formed by soldering a strip of the gauze at right angles to the periphery of a disk of bronze gauze; one of the bases acts as a lid.
- 3.6 *Activated carbon*, with a grain size of about 4 to 6 mm, free from powder. The carbon should be of a well determined type and grade, in order to obtain concordant results (see Appendix).

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 should be in the form of a disk 50 ± 1 mm in diameter and 1 ± 0.1 mm in thickness.
- 4.2 The test specimens are cut with a special die from a 1 mm thick sheet, obtained by compression moulding of one or more layers of the material to be tested or from the sheets prepared on the mixer, when the material is in granules or in powder form, employing as a mould a convenient frame and bright steel plates.
- 4.3 If the test is carried out for the determination of the characteristics of given plasticizers, a standard compound of a given composition, as agreed between vendor and purchaser, should be prepared.
- 4.4 At least 3 test specimens should be tested for each material.
- 4.5 The use of specimens of different shape and thickness is not precluded. A comparison of the values obtained is possible, however, only for specimens of the same thickness.
- 4.6 In the case of coated fabrics and supported plastic films in general, the specimen should be cut with a die from the material to be tested.

5. CONDITIONING

The test specimens should be conditioned according to the relevant ISO Recommendation.*

6. PROCEDURE

One of two methods of test should be used:

The first, Method A, is in *direct contact* with the carbon and is particularly useful for material which must be tested at relatively low temperature, on account of the fact that they flow at higher temperatures.

The second, Method B, prescribes the use of a *wire cage*, which prevents the direct contact of the material with the carbon.

6.1 Method A : in direct contact with activated carbon

6.1.1 After conditioning, weigh the test specimens individually to the nearest 0.001 g.

6.1.2 On the bottom of a metal container (see Clause 3.4) spread about 120 cm³ of activated carbon (see Clause 3.6). The first specimen is then placed on the top of the carbon and covered with a further 120 cm³ of carbon. A second specimen is placed in the same manner, followed by a third, and covered by 120 cm³ of carbon. A lid is then put on the container, it should not be air-tight or, at least, should have a breather.

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

6.1.4 The container is then placed in an oven or a thermostatic bath at a temperature of 70 ± 1 °C, for 24 hours.

6.1.5 After the test, the container is allowed to cool at room temperature; the specimens are then removed from the container, carefully brushed free from any trace of carbon particles which may have adhered to them, and reconditioned as indicated under clause 5.

6.1.6 The specimens are reweighed to the nearest 0.001 g.

* The ISO Recommendation relating to standard atmospheres for conditioning and testing plastics materials is being prepared.

6.2 Method B : with wire cage

- 6.2.1 The procedure is the same as for Method A, with the difference that every single specimen is introduced into a small metal wire-mesh cage, as indicated under clause 3.5, which avoids direct contact between the plastic and the carbon.
- 6.2.2 Maintain the temperature at 100 ± 1 °C (the duration of the test, unless otherwise agreed, is 24 hours).
- 6.2.3 After the test, the specimens are removed from the container, reconditioned and reweighed (as described above under Method A, clause 6.1.6.).

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

7. CALCULATION AND EXPRESSION OF RESULTS

The percentage mass change is calculated as follows for each specimen:

$$\Delta M_1 = \frac{M_1 - M'_1}{M_1} 100$$

where M_1 = mass, in grammes, of the test specimen after conditioning.

M'_1 = mass, in grammes, of the test specimen after treatment in the oven or thermostatical bath and reconditioning.

ΔM_1 = change (loss) of mass.

8. REPORT

The report should include the following:

- 8.1 Complete identification of the sample and procedure of preparing the specimens.
- 8.2 The thicknesses of the single specimens, to the nearest 0.01 mm.
- 8.3 The absolute values of the mass of test specimens, in grammes, before the test and the absolute value of gain or loss in mass, in milligrammes, after testing.
- 8.4 The mass change of the individual specimens, expressed as a percentage.
- 8.5 The mean of the values obtained on three specimens.
- 8.6 Any observation as to change in appearance of the specimens.

APPENDIX

BRANDS OF ACTIVATED CARBON

The use of the following brands of activated carbon may be suggested:

1. **in the U.S.A.:** Columbia Activated Carbon, Grade AC 6/14 mesh or AAC 6/14 mesh, as manufactured by Carbide and Carbon Chemical Company, U.S.A.;
2. **in Europe:** Activated Carbon Ref. 207 C-5 14 mesh, manufactured by Sutcliffe Speakman & Co., Ltd., Leigh, Lancashire, United Kingdom, or

Aktivkohle TD 2 and TD 4, manufactured by Bayer Farbenfabriken, Leverkusen, Germany.