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R 1137

PLASTICS

METHOD OF TEST FOR THE DETERMINATION OF THE BEHAVIOUR OF PLASTICS IN A VENTILATED TUBULAR OVEN

1st EDITION

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1137, *Plastics – Method of test for the determination of the behaviour of plastics in a ventilated tubular oven,* was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In July 1967, this Draft ISO Recommendation (No. 1300) was circulated to all the Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Iran	Sweden
Bulgaria	Israel	Switzerland
Canada	Japan	Turkey
Chile	Netherlands	U.A.R.
Colombia	New Zealand	United Kingdom
Czechoslovakia	Poland	U.S.A.
France	Portugal	U.S.S.R.
Greece	Romania	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1137 | November 1969

PLASTICS

METHOD OF TEST FOR THE DETERMINATION OF THE BEHAVIOUR OF PLASTICS IN A VENTILATED TUBULAR OVEN

1. SCOPE

This ISO Recommendation describes a procedure for determining the behaviour of plastics during exposure in a ventilated tubular oven.

2. PRINCIPLE

Each plastics test specimen is exposed in a tube in an air current having carefully controlled temperature and velocity. After an interval of time, previously determined, the changes in one or more properties are measured, such as loss in mass, change of mechanical, thermal or other properties, change in colour, etc.

Loss in mass (usually of plasticizer) is proportional to the surface area of the test specimens, but is also influenced by their thickness. Consequently, in comparing different products, it is absolutely necessary to examine test specimens of equal thickness.

3. APPARATUS

The apparatus (see Fig. 1) consists of

- a device supplying heated air (see clause 3.1);
- a thermostated enclosure containing tubes through which the air circulates at constant temperature and velocity (see clause 3.2);
- auxiliary control equipment (see clauses 3.3 and 3.4);
- specimen holders (see clause 3.5).

3.1 **Device supplying heated air at a constant temperature** (between 70 and 160 °C)

This contains a fan which draws air from outside of the apparatus. This air is heated and homogenized, for example by passing it over electric heating coils controlled by a thermoregulator at the entrance of one of the tubes of the enclosure described in clause 3.2. After mixing, the air passes through the tubes.

3.2 **Thermostated enclosure**

This enclosure is at the same temperature as the heated air and this temperature is confirmed by a thermometer. In the enclosure are fixed demountable glass tubes of internal diameter between 35 and 50 mm and of length greater than 500 mm. In any case, the internal diameter must be greater by at least 10 mm than the width of the specimens to be inserted into them.

The tubes may be mounted horizontally or vertically. Valves are placed at the entrance and, if desired, at the exit of the tubes to allow regulation of the air speed to 100 ± 10 m/min*.

A simple means of obtaining uniform flow, such as glass wool plugs, is placed at the entrance and exit of the tubes.

The air is finally discharged to the atmosphere.

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* Upon agreement between supplier and purchaser, other velocities, for instance 250 ± 25 m/min, may be used.

3.3 **Device for measuring the rate of air flow through the tubes to ± 2 %**

The measurement may be made in one of the three following ways :

(*a*) either before and after the test, directly at the tubes, or by replacing one tube by a dummy calibrating tube;

(*b*) during the test, by measuring at the exit of the tube; in this case the measuring apparatus must not alter the rate of flow;

(*c*) by a permanently installed measuring device.
A suitable device is a rotameter.

The air flow measurement enables the air velocity through the tubes to be calculated.

3.4 **Temperature-measuring device** (thermometer or thermocouple) to measure the temperature at the centre of each tube to ± 0.5 °C

If this method cannot be applied, a single device should be fixed at the common entrance of the hot air, as close as possible to the flow regulation valves.

3.5 **Specimen holders**

These holders should maintain each test specimen on a plane passing through the axis of the tube. The forms and dimensions of the specimen holders must be such that

(*a*) the area of contact of the test specimen is as small as possible;

(*b*) they give minimum obstruction to the passage of air;

(*c*) one or several of them may be placed in each tube, each holder containing one or several test specimens of the dimensions given in section 4.

For horizontal tubes, Figure 2 represents, schematically, a specimen holder that has given satisfaction.

For vertical tubes, a hole may be made in the test specimens by which they may be hung from glass hooks. The hooks should be attached with a thread to the upper end of the tube.

4. TEST SPECIMENS

4.1 If *loss in mass* is to be determined, the test specimen should be a rectangle of minimum dimensions 100 mm × 25 mm, cut out of the sheet of material.

If the test material is not a sheet, it is necessary to prepare a sheet of 1 ± 0.1 mm thickness, unless a contrary stipulation is made in a specification or in an agreement between supplier and purchaser.

4.2 If the measurements cover *variation of mechanical or thermal properties,* etc., the test specimens should be as prescribed in the corresponding ISO Recommendations.

5. PROCEDURE

5.1 Determination of properties before exposure

5.1.1 If *loss in mass* is being determined, weigh the test specimen to 0.2 mg and determine the thickness to 0.1 mm and the two other dimensions to 1 mm.

At least three test specimens are required.

NOTE. – If the sample contains moisture, it may be decided by agreement between purchaser and supplier to weigh specimens just as they are, to dry them by appropriate means, then to weigh them again before putting them into a ventilated oven.

Loss of mass on drying can be regarded as being mainly loss of moisture. Loss of mass of dried test pieces can be regarded as being mainly loss of other volatiles.

No established correlation exists between loss of mass determined by this method on dried test pieces and that determined by the method given in ISO Recommendation R 176, *Plastics – Determination of the loss of plasticizers by the activated carbon method.*

5.1.2 If *changes of mechanical, thermal, electrical or other properties* are being determined, condition the specimens and measure the properties by the corresponding standardized methods, using the specimens themselves for non-destructive tests or, in the case of destructive tests, using additional specimens cut from the same sample. In the latter case, double the standard number of specimens will be required.

5.2 Adjustment of apparatus

The apparatus should be adjusted so that

(*a*) the air speed in each tube, measured by the means described in clause 3.3, is 100 m/min;

(*b*) the temperature measured in the thermostated enclosure (3.2) and that determined at the centre of several tubes by the means described in clause 3.4 are equal to one of the specified temperatures with a tolerance of ± 2 °C.

Unless there is a contrary stipulation, one of the following temperatures should be selected :

70 °C, 85 °C, 100 °C, 125 °C, 155 °C

5.3 Exposure to current of heated air

Mount the test specimens in the specimen holders.

Place the loaded specimen holders in the tubes.

These two operations must be carried out in such a way that test specimens do not touch each other and the distance from any point of a specimen to the tube wall is at least 5 mm. Only specimens of the same composition should be put into the same tube.

Operate at the selected temperature (see paragraph (*b*) of clause 5.2) for a period determined in advance and selected from the following list :

1, 2, 4, 8, 16 hours; 1, 2, 4 days; 1, 2, 4, 8, 16 weeks, etc.

Check the enclosure temperature and the temperature of the tubes periodically during the test to ensure that they remain within the specified limits of ± 2 °C.

If checking the air velocity in each tube during the test is possible, carry out the check periodically to ensure that the velocity remains within the limits of 100 ± 10 m/min, adjusting the vanes if necessary.

If checking the air velocity in each tube during the test is not possible, carry out the check at the end of the test for every tube and disregard the results for those tubes for which the final speed is not within the limits of 100 ± 10 m/min.

At the end of the exposure period, take out the loaded specimen holders and allow them to cool. If loss of mass is being determined, use a desiccator.

5.4 Determination of properties after exposure

Proceed exactly as for determination of properties before exposure (see clause 5.1).

6. CALCULATION AND EXPRESSION OF RESULTS

6.1 If *loss in mass* is being determined, calculate this loss per unit surface area as follows :

$$\text{Loss in mass} = \frac{M_1 - M_2}{S} \text{ g/m}^2$$

where

M_1 is the initial mass, in grammes, of the test specimen;

M_2 is the mass, in grammes, of the test specimen after exposure;

S is the surface area, in square metres, of the test specimen.

Calculate the mean for identical test specimens.

6.2 If *change in some physical property* (mechanical, thermal, etc.) is being determined, calculate separately the means of the values before and after exposure.

For the measurable physical properties, also calculate the final percentage of this characteristic relative to the initial value, as follows :

$$\frac{V_2}{V_1} \times 100$$

where

V_1 is the mean of the values before exposure;

V_2 is the mean of the values after exposure.

This percentage may be less than or greater than 100.

7. TEST REPORT

The test report should include the following information :

(*a*) the form, dimensions and, if required, the surface area of the test specimen;

(*b*) the conditions of exposure (temperature and duration);

(*c*) reference to the test method used;

(*d*) the results obtained in each case, expressed in accordance with section 6, as follows :

- the mean values of physical characteristics of test specimens before and after exposure;
- the percentage values of these characteristics, related to initial values;
- the mean value of loss in mass of the test specimens;
- if requested, the values obtained for each test specimen.