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Cellular plastics — Determination of the coefficient of linear thermal expansion of rigid materials at sub-ambient temperatures

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Foreword

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Cellular plastics — Determination of the coefficient of linear thermal expansion of rigid materials at sub-ambient temperatures

1 Scope and field of application

This International Standard specifies two methods for determining the coefficient of linear thermal expansion of rigid cellular plastics at sub-ambient temperatures. Method A is the preferred method since it gives a value on a large sample at a chosen temperature which is more representative because of anisotropy, etc., of the material or product. In practice, difficulties may be encountered in accurately controlling the uniformity of the low temperature inside the test chamber at temperatures below $-70\text{ }^{\circ}\text{C}$. Should this be the case, Method B should be employed to obtain an "average" coefficient for the temperature interval between the cryogenic temperature and the ambient temperature.

The values obtained in these tests are valid measurements only for the specific sample tested, at the time tested. They cannot be applied generally to a product and cannot be used to predict performance of the same material at a future date.

Care should also be taken in applying the value obtained in this test to calculate the overall coefficient of expansion of composite products of which the material under test is part.

Many rigid cellular products are not isotropic. This is usually ascribed to the fact that the cells in the material are elongated in a particular direction. This direction is referred to in these methods as the "direction of anisotropy". Tests carried out in this direction normally give results differing from those obtained from other test directions. The direction of anisotropy may vary within a product and thus the number of test specimens required for product specification will generally exceed those given in this International Standard.

2 References

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

ISO 1923, *Cellular plastics and rubbers — Determination of linear dimensions*.

3 Definition

coefficient of linear thermal expansion : The change in unit length per degree Celsius change in temperature.

4 Method A

4.1 Apparatus

An apparatus found suitable consists of the following:

4.1.1 Test chamber and cooling system

The test chamber shall consist of a well-insulated box of approximate internal dimensions $400\text{ mm} \times 1\,000\text{ mm} \times 150\text{ mm}$, having viewing windows $900 \pm 10\text{ mm}$ apart. These windows shall be insulated with plugs of insulation between readings to ensure good temperature distribution.

The chamber shall be fitted with a cooling system designed to give a sub-ambient internal temperature distribution complying with the requirements of 4.4. A suitable chamber and cooling system is described in the annex and illustrated in figures 1 and 2.

NOTE — Liquid nitrogen has been found in practice to be the most satisfactory coolant.

4.1.2 Reference length material

Inside the box a reference length material of $880 \pm 5\text{ mm}$ shall be positioned so that fiducial marks on the end are at the same level and in the same vertical plane as fiducial lines on the test specimens. The coefficient of expansion of the reference length material should be small and accurately known. A suitable material is silica in the form of a rod with ends ground to knife edges (see figure 3). It shall be adequately supported to prevent bowing.

4.1.3 Measuring system

A travelling microscope or equivalent capable of measuring to $0,01\text{ mm}$ shall be provided. Care shall be taken to ensure that the direction of travel of the instrument is parallel to the edge of the test specimen.

4.1.4 Temperature-measuring device

Any suitable temperature-measuring device may be used; it shall be calibrated over the required range to within $1\text{ }^{\circ}\text{C}$.

Temperatures in the box shall be measured by any suitable means in at least five positions equally spaced along the entire length of the test specimen.

4.2 Test specimen

The test specimen shall be a rectangular board having the following dimensions:

- 900 + $^{20}_0$ mm in length;
- 100 to 300 mm in width;
- 25 to 50 mm in thickness.

The test specimen shall be cut without significant deformation of the original cell structure; the surfaces shall be parallel and unbowed and free of surface skins (hot-wire cutting is not permissible).

To each end of the board shall be fixed fiducial lines. These may consist, for example, of razor blades attached rigidly to the corners by drawing pins so that the knife edges project beyond the edge of the specimen and perpendicular to its length (see figure 3).

4.3 Conditioning

The test specimens shall be conditioned at 23 ± 2 °C and 50 ± 5 % relative humidity for not less than 16 h. The test shall be carried out immediately after conditioning.

4.4 Procedure

4.4.1 Determine the length of both the test specimen and the reference length in accordance with ISO 1923.

4.4.2 Support the test specimen in the apparatus so that it does not sag and is free to move, and so that air may circulate freely round it. Position the ends of the specimen carrying the fiducial lines centrally in the windows and symmetrically with respect to the reference length. Then insert the temperature measuring devices and switch on the fan (if used). After the temperature has been steady within ± 2 °C for 30 min, measure the difference in length between the test specimen and the reference length.

4.4.3 Adjust the thermostat to reduce the temperature by about 20 °C and insert the plugs in the viewing holes. When the temperature is steady to within ± 2 °C for 20 min at the new level, again measure the difference between the length of the test specimen and the reference length. Repeat this measurement procedure at approximately 5-minute intervals until three consecutive measurements agree to within 0,03 mm.

4.4.4 Repeat the above procedure until the lowest temperature required is reached. Also make measurements as the apparatus is brought back to room temperature while observing the same precautions to establish thermal equilibrium. Check the temperature in the box before and after each measurement of length; the average of the total number of readings shall be taken as the temperature to be associated with the length measured. The difference between the average temperature immediately before and after measurement shall not exceed 2 °C and the corresponding difference for any individual temperature measuring device shall not exceed 5 °C.

4.5 Expression of results

Use the results obtained to plot a graph of the length of the test specimen against temperature. Make allowance for the (calculated) change in the length of the reference length. The results should all lie on a smooth curve and there should be no significant hysteresis between descending and ascending temperature points. If there is significant hysteresis, repeat the determination allowing more time for the test specimen to attain thermal equilibrium.

Determine the mean coefficient of linear expansion over the selected temperature range as follows:

From the graph, read off the lengths of the specimen at the upper and lower temperatures of the range over which it is required to know the coefficient. Calculate the mean coefficient over the selected temperature range from the following equation:

$$\bar{\alpha} = \frac{\Delta L}{L_0} \times \frac{1}{T_1 - T_2}$$

where

$\bar{\alpha}$ is the mean coefficient of linear expansion, in reciprocal kelvins;

T_1 is the higher temperature selected, in kelvins;

T_2 is the lower temperature selected, in kelvins;

ΔL is the change in length, in millimetres, of the test specimen between temperatures T_1 and T_2 ;

L_0 is the original length, in millimetres, of the test specimen at 23 ± 2 °C.

4.6 Test report

The test report shall include the following:

- a) reference to this International Standard, followed by "Method A";
- b) the description and identity of the sample;
- c) the age of the sample and the date of the test;
- d) the direction of any known anisotropy in relation to the measured length;
- e) the mean coefficient of linear expansion to two significant figures and the temperature range to which the result relates;
- f) copy of the experimental graph;
- g) details of conditioning, if other than those specified in 4.3.

5 Method B

5.1 Apparatus

The apparatus illustrated in figures 4 and 5 consists of the following:

5.1.1 Silica tube (1), which holds the test specimen, fixed in the vertical position on a laboratory support (2) and having an internal diameter of 32 mm and a height of 250 mm. The bottom end of this silica tube is closed by a horizontal wall (3) of the same material, which is welded in a position in which it is absolutely perpendicular to the axis of the tube; this wall serves as a support face for the test specimen (4). An opening (5) about 55 mm high and 24 mm wide in the lower part of the tube makes it possible to insert and position the test specimen.

5.1.2 Silica piston (6), consisting of a rod (diameter 10 mm, height 225 mm) and a cylindrical base (diameter 25 mm, height 5 mm), serving as a displacement pick-up. It is centred inside the tube which carries the test specimen with the aid of a washer (7) which is made of light alloy and is positioned at the top of the tube.

The silica tube/piston assembly is fixed to the support (2) with the aid of a metal clamping part (8) and a securing rod.

5.1.3 Micrometer (9), fixed to the same support (2) and allowing a measurement to be read off to the nearest 0,001 mm. The point of the sensing probe is centred on the rod of the piston. The reaction of the micrometer and the weight of the piston exert a force of the order of 1,4 N on the test specimen.

Auxiliary equipment:

5.1.4 Dewar flask, having a capacity of about 2 l and fixed in a flat-bottomed insulating case.

5.1.5 Lifting support, intended for the continuous vertical displacement of the Dewar flask.

5.1.6 Container of cryogenic liquid.

5.1.7 Fine saw, provided with a device for cutting out the test specimens.

5.1.8 Reference test specimens, having a known coefficient of expansion (Cu, Al, glass, etc.) which may be used for checking the apparatus and the reproducibility of the measurements. Dimensions of reference specimens shall be: diameter 20 mm, height 50 mm.

5.2 Test specimens

5.2.1 Dimensions

The test specimens shall be parallelepipeds having a square cross-section and the following dimensions: length 50 ± 1 mm; cross-section 20 ± 1 mm \times 20 ± 1 mm.

5.2.2 Preparation

The test specimens shall be cut from the middle of the product with the aid of a saw which is suitable for ensuring a satisfactory surface state, parallelism of the faces and perpendicularity of the rectangular lateral faces to one another.

Since certain rigid cellular plastic materials are anisotropic, it is necessary to note the direction of sampling of the test specimens in relation to the direction of expansion, or simply in relation to the faces of the panel from which the sample was removed.

5.2.3 Number

Carry out the test on at least five test specimens.

5.3 Conditioning

See 4.3.

5.4 Procedure

5.4.1 Work in an air-conditioned room in which the temperature, which is selected at between 20 and 25 °C (T_0), is kept constant to within ± 1 °C, or failing this, place the entire apparatus in an enclosure which is air-conditioned to produce these conditions.

5.4.2 Determine, in accordance with ISO 1923, the length L_0 of the test specimen (corresponding to the temperature T_0) with an accuracy of $\pm 0,1$ mm.

5.4.3 Place the test specimen in the silica tube (5.1.1) and arrange the piston (5.1.2), which detects displacement, in such a way that it is properly centred and in contact with the upper face of the test specimen. Position the micrometer (5.1.3) so that it is in contact with the piston and adjust its dial so that it indicates the graduation "0" at the temperature T_0 . The reaction of the micrometer and the weight of the piston ensure good contact between the test specimen and the piston which detects displacement.

5.4.4 With the aid of the lifting support (5.1.5), raise the Dewar flask (5.1.4) filled with cryogenic liquid (5.1.6) in a progressive manner, avoiding excessively violent bubbling, in such a way that, in the end, the test specimen is completely immersed in the liquid and the surface of the latter is at least 30 mm above the test specimen. After a stabilization period of about 10 min, record the micrometer graduation (ΔL) at which the needle of the comparator has stopped.

5.4.5 Lower the Dewar flask and allow the temperature of the test specimen and of the apparatus to rise to room temperature T_0 again. It is possible to speed up the rise in the temperature of the apparatus by using air at a low rate of flow.

NOTE — If it is necessary to avoid direct contact between the cryogenic liquid and the test specimen, the silica cylinder may be protected by a metal jacket having a slightly larger diameter. In this event, the final temperature T of the test specimen shall be measured with the aid of a thermocouple.