International Standard

ISO 4897

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

Plastiques alvéolaires — Détermination du coefficient de dilatation linéique thermique des plastiques alvéolaires rigides aux températures inférieures à l'ambiante

First edition — 1985-04-01

ISO 4897-1985 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4897 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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 °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 mm × 1 000 mm × 150 mm, having viewing windows 900 ± 10 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 ± 5 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 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 °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 $^{+\ 20}_{\quad 0}$ 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:

$$\bar{\alpha} = \frac{\triangle L}{L_o} \times \frac{1}{T_1 - T_2}$$

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;

$\triangle L$ is the change in length, in millimetres, of the test specimen between temperatures T_1 and T_2;

L_o 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.

2

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_o), is kept constant to within $\pm$ 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_o of the test specimen (corresponding to the temperature T_o) 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_o. 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 ($\triangle L$) 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_o 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.

5.5 Expression of results

The mean coefficient of linear expansion $\bar{\alpha}$ between the temperatures T_o and T, is given, in reciprocal kelvins, by the formula

$$\bar{\alpha} = \frac{1}{L_o} \times \frac{\triangle L}{T_o - T}$$

where

L_o is the initial length, in millimetres, of the test specimen;

T_o is the temperature, in kelvins, of the room;

T is the evaporation temperature, in kelvins, of the cryogenic liquid used;

$\triangle L$ is the micrometer reading, in millimetres, indicating the contraction of the test specimen.

5.6 Test report

The test report shall include the following:

a) reference to this International Standard, followed by "Method B";

b) the description and identity of the sample;

c) the age of the sample and the date of the test;

d) the conditioning to which the test specimens have been subjected;

e) the temperature of the room at the time of the test;

f) the evaporation temperature and the nature of the cryogenic fluid;

g) the lengthwise direction of the test specimens relative to the panel;

h) the mean coefficient of linear expansion to two significant figures;

j) any deformation or deterioration in the cellular material;

k) operational details not specified in the standard, and also any incidents which may have affected the results.

5.7 Notes to Method B

1 The choice of silica as the material for most of the test device emerged after comparative tests with metallic supports of various constructions. The uncertain results obtained with the latter led to the production of the model described herein. The mean coefficient of contraction of silica between ambient temperature and cryogenic temperatures is of the order of $0,02 \times 10^{-5} \, K^{-1}$; that of cellular plastics varies from 2 to $10 \times 10^{-5} \, K^{-1}$.

2 The method B relates to measurement of the **mean** coefficient of contraction between two given extreme temperatures, and could not serve as a substitute, in all cases, for determination of the true coefficient of expansion as a function of temperature. Nevertheless, it offers great advantages: its simplicity and the economy of resources which it permits, without sacrificing accuracy.

3 The use of cryogenic baths in direct contact with samples, which is a very simple technique, has been tried on a wide range of cellular plastics without, it seems, giving rise to disturbances (e.g. thermal shock) in their physical state, and therefore in the measurements. After their return to ambient temperature, the samples practically regain their initial length (permanent deformations, if any are found, are of the order of magnitude of the reproducibility of the measurement). Moreover, successive measurements on the same sample, involving immersion in liquid nitrogen, give fairly reproducible results.

4 It may occur in certain cases (this has been verified, for example, on samples of expanded polystyrene) that the mean coefficient of contraction measured by this method (between the temperature of a bath of liquid nitrogen and ambient temperature)

$$\bar{\alpha} = \frac{1}{L_o} \times \frac{\triangle L}{\triangle T}$$

is equal to the true coefficient of contraction

$$\alpha(T) = \frac{1}{L} \times \frac{dL}{dT}$$

i.e. one is in a situation where $\alpha(T)$ = constant in the temperature interval in question.

If, on the other hand, the true coefficient varies with the temperature within the interval considered, $\bar{\alpha} = \alpha(T)$, one must be careful not to use the value $\bar{\alpha}$ obtained between two extreme temperatures to forecast the behaviour of the cellular material under other conditions, even, for example, between one of the extreme temperatures and a temperature which falls within the interval. In fact, in the case of a cellular product other than polystyrene, it has been found, for example, on the basis of curves of the true coefficient of expansion, that:

$\bar{\alpha}$ between $+20$ and $-190 \, °C = 8 \times 10^{-5} \, K^{-1}$

$\bar{\alpha}$ between $+20$ and $-50 \, °C = 15,5 \times 10^{-5} \, K^{-1}$

If, therefore, in the case of certain cellular materials, it is desired to know the mean coefficient of expansion between any two temperatures, method B cannot generally be used for this purpose. Nevertheless, it may be adapted to this problem by replacing the measuring micrometer by a displacement pick-up capable of supplying an electrical signal that will be picked up on a recorder as a function of the temperature of the test specimen, the said temperature being controlled by a liquid bath surrounding the test specimen in accordance with a programme that has been pre-set on a thermostat.

Annex

Suitable apparatus for method A

(This annex forms an integral part of the Standard.)

A suitable apparatus is shown in figures 1 and 2 and consists of the following:

A.1 Test chamber, consisting of a box (see figure 1) made from a good sub-ambient temperature insulating material. The internal dimensions of the box shall be approximately 400 mm × 1 000 mm × 150 mm with a wall thickness of at least 100 mm. In the front of the box, two tapered viewing holes of about 50 mm diameter and having centres 900 ± 10 mm apart shall be cut so that the ends of the test specimen can be viewed under all temperature conditions. These holes shall be closed on the inside by thin glass sheet; plugs to fill the holes shall be provided. In the back of the box a cooling coil shall be fitted consisting of three turns of copper tubing of 5 mm nominal bore running the length of the box; the top limb shall be pierced with holes of 1,5 mm diameter. The coil shall be closed at one end and the other end shall be connected to a liquid nitrogen container by a heavily insulated tube (see figure 2). A fan shall be installed in the box to minimize any temperature variation.

A.2 Cooling system

Liquid nitrogen shall be supplied to the cooling coil by pressurizing the container with compressed nitrogen (see figure 2). The rate of flow of liquid nitrogen to the coil shall be regulated by a gas valve controlled by a thermostat which shall provide venting of the pressurizing air to atmosphere to reduce the pressure in the liquid nitrogen container and also to provide a vent for any boil-off from the liquid nitrogen container. A buffer vessel shall be provided in the compressed air system to eliminate "hunting" of the temperature control and a manometer with a range of about 20 kPa* to indicate the air pressure to the required accuracy.
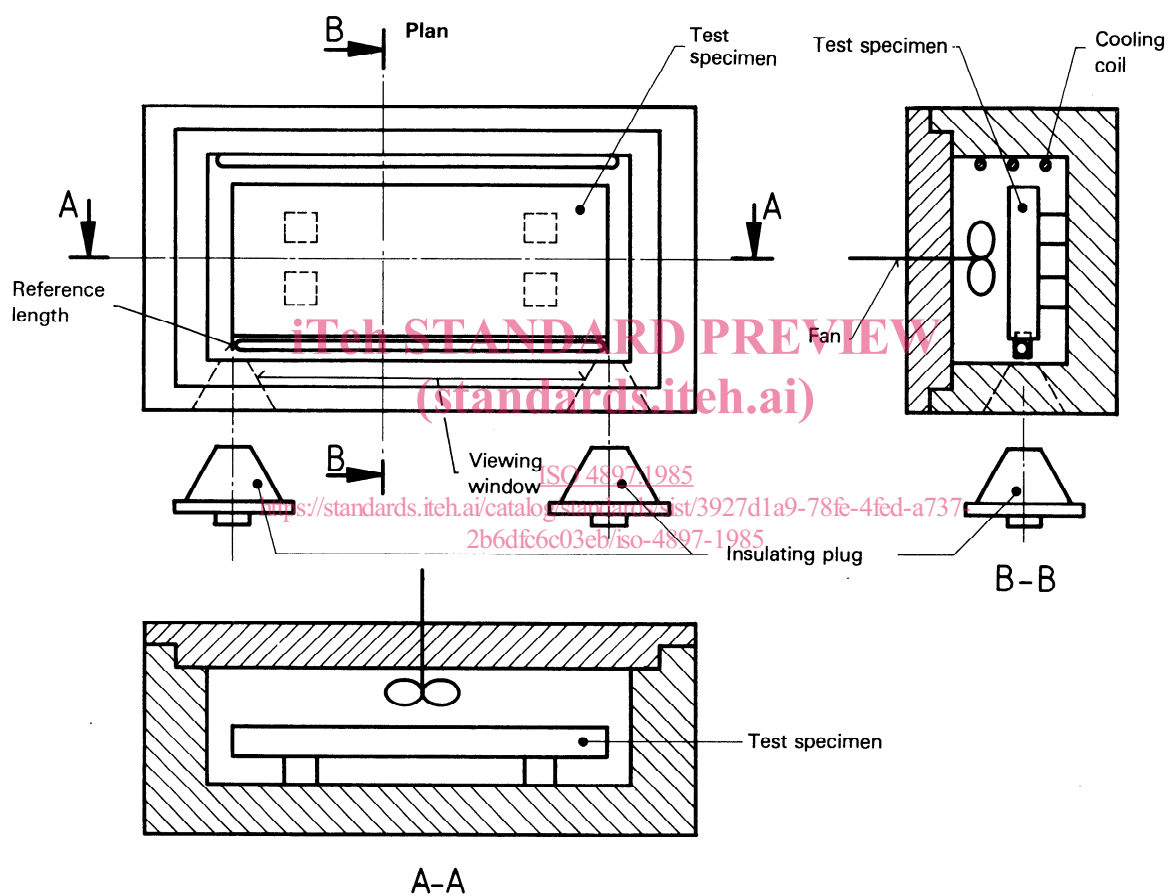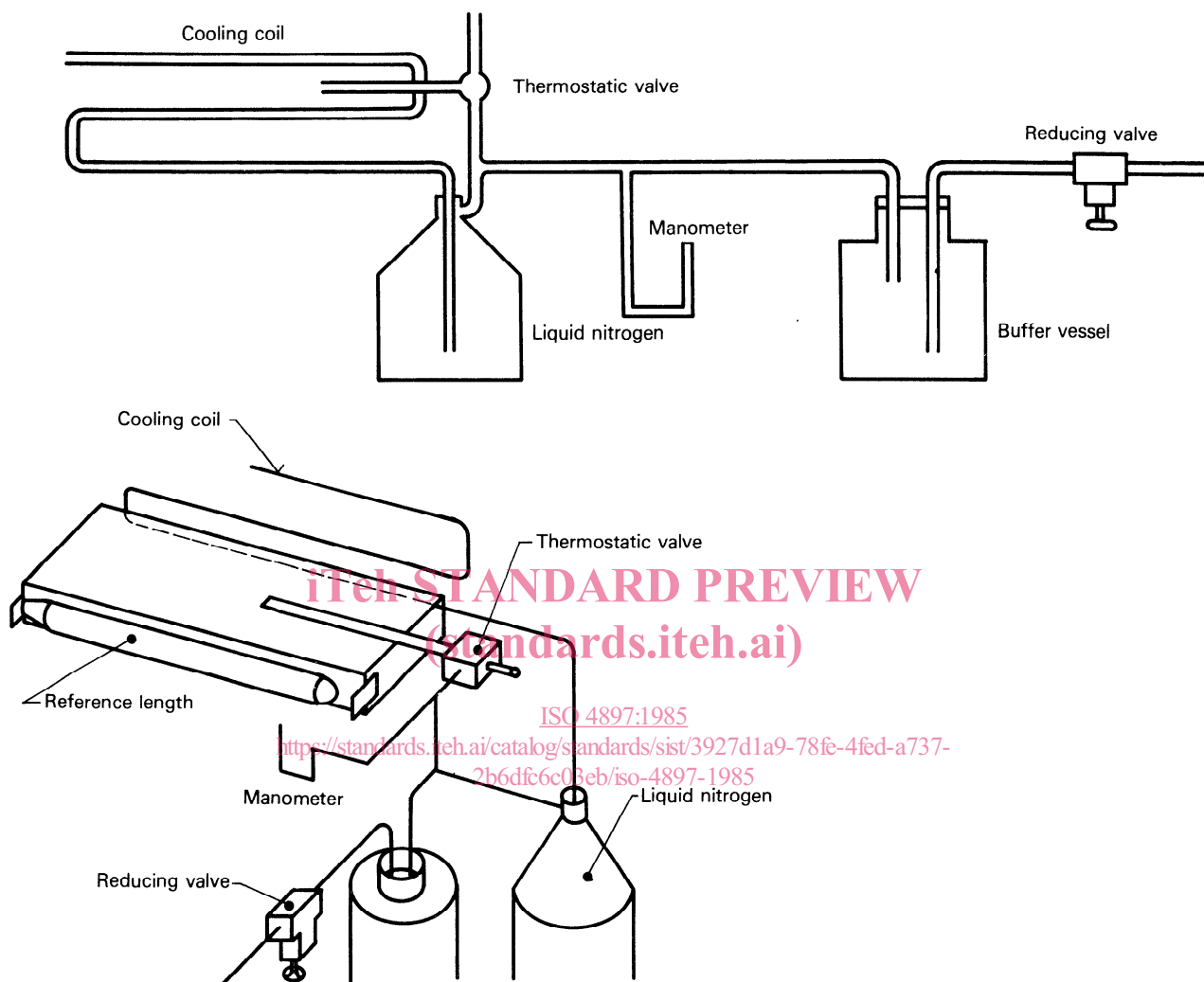
---

* 1 kPa = 1 kN/m^2

Figure 1 — Suitable apparatus for determination of coefficient of linear thermal expansion at sub-ambient temperatures for method A

NOTE — Attention is drawn to the fact that this particular cooling system is the subject of a patent.

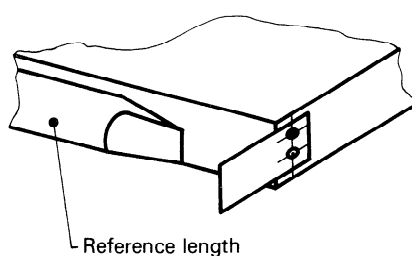**Figure 2 — Layout of suitable cooling system for method A**

Figure 3 — Suitable fiducial lines on test specimen for method A