INTERNATIONAL STANDARD 1268

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes

Matières plastiques — Préparation de plaques ou de panneaux en stratifiés verre textile-résine basse-pression pour la réalisation d'éprouvettes

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

International Standard ISO 1268 was drawn up by Technical Committee ISO/TC 61, *Plastics*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 1268-1970, which had been approved by the Member Bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Iran	Sweden
Bulgaria	Israel	Switzerland
Canada	Japan	Turkey
Czechoslovakia	Korea, Dem. P. Rep. of	United Kingdom
Egypt, Arab Rep. of	Korea, Rep. of	U.S.A.
France	Netherlands	Yugoslavia
Germany	Poland	
Greece	Romania	

The Member Body of the following country had expressed disapproval of the Recommendation :

Italy

Printed in Switzerland

Plastics — Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the procedure for the preparation of test plates or panels by bonding glass cloth or mats with low-pressure thermosetting resins. In this International Standard the term "low-pressure thermosetting resins" relates to resins which cure at or above room temperature either without loss of reaction products or which are not significantly altered by loss of reaction products during cure and which can be processed with a pressure up to 3,5 MPa. The purpose of this International Standard is to standardize the preparation of the plates or panels from which test specimens subsequently are machined. Standard specimens prepared in this manner may be used either for evaluating the components; i.e. the glass reinforcement, finishes, resins, catalysts, curing agents, etc., or for verifying the overall quality of the finished "composite" products.

2 PRINCIPLE

Preparation of the plates or panels by one of the following methods :

2.1 Method A

The glass reinforcement impregnated with liquid resin containing a suitable catalyst or curing agent is moulded in a press under conditions of temperature and pressure appropriate for the resin and curing system.

2.2 Method B

The glass reinforcement impregnated with resin partially cured to the B stage (prepreg), but capable of further curing, is moulded in a press at a mould temperature higher than room temperature.

2.3 Method C

The glass reinforcement is manually laminated or laid up with simultaneous impregnation with a resin containing a suitable catalyst or curing agent. The curing of the resin proceeds at room temperature or at elevated temperature. No press is required for preparation of the laminate.

3 EQUIPMENT FOR THE MAKING OF PLATES OR PANELS BY METHODS A AND B

3.1 Press

For moulding the plates or panels, use may be made of any hydraulic or mechanical press which permits

a) clamping the mould as specified in 3.2;

b) maintaining the moulding force with an accuracy of ± 5 % over the period of time required for the curing of the resin;

c) maintaining the closing speed of the mould in accordance with the requirements of 4.1.4. Generally the closing speed should be low and adjustable.

3.2 Mould

3.2.1 One of two types of moulds is chosen, depending on

a) the nature of the glass reinforcement;

b) the type of resin and curing system;

c) the purpose of testing.

3.2.2 The mould type 1 (see figure 1) or type 2 (see figure 2), shall comply with the following conditions, but in other respects the design is optional :

a) the working surface of the mould shall be a square, the dimensions of which are within the limits specified for types 1 and 2 (between 150 mm × 150 mm and 400 mm × 400 mm);

b) the active surfaces shall be hardened, ground and hard-chromium plated; the active surface of the lower die shall bear a mark which, when impressed on the moulded plate or panel, will identify the surface formed by the lower die. Care shall be taken to ensure that such marks do not influence the results of subsequent testing (types 1 and 2, see note below);

c) the type 1 mould is a two-part (open type) mould consisting of two plane parallel plates separated at constant distance during the moulding operation by means of spacers. This type of mould ordinarily is used for specification purposes;

d) the type 2 mould is a three-part positive type consisting of a removable chase (frame, for example F in figure 2) and an upper and lower die. The cavity shall have such dimensions that the material required for the prescribed plate thickness can be introduced in a single charge. The tool shall be provided with suitable guiding (for example K in figure 2) so that a clearance, b, of 0,15 ± 0,05 mm can be maintained around the perimeter of the punch. It is recommended that the inner walls of the frame be tapered as shown in figure 2, where $c = 6$ mm and $\alpha = 1°$;

e) the temperature of any spot of the active surface of the mould shall not differ by more than ± 2 °C from the value indicated by the temperature check sensitive element built into the mould.

NOTE – The two main surfaces of the moulded plate or panel are not exactly equivalent with regard to all of their properties. During the period between filling and closing of the mould the lower surface of the test plate is heated longer and to a higher temperature than the upper surface. It may therefore be advisable in preparing test methods, specifications, property schedules, etc. to state the particular surface to which specifications or properties should be referred.

4 WORKING PROCEDURES

4.1 Method A

4.1.1 Shear, cut or chop the glass reinforcement into squares, the sides of which are 3 to 6 mm less than the dimensions of the cavity of the tool.

4.1.2 Dry the glass reinforcement thus prepared at 80 °C for a period of 1 h, except in the case of sized or finished glass reinforcements requiring lower temperature. After drying, cool it for 1 h in a desiccator over a drying agent and then impregnate it.

4.1.3 Prepare the laminate at a relative humidity not exceeding 65 % unless otherwise agreed. The impregnation of the glass reinforcement with a resin containing the required catalyst or curing agent may be conducted outside the mould on a suitable foil or film (placed on a heated support pad, if necessary) by freely spreading the resin with a spatula on individual layers of the glass reinforcement, or may be carried out in the mould cavity. An excess of at least 20 % of resin as compared with the resin content of the cured product (in the finished plate) is consumed in making the plate. After lamination of the required number of layers, place the impregnated glass reinforcement in the cavity of the mould (if impregnation has been conducted outside the mould) and heat to curing temperature.

Unless otherwise specified, not more than 50 % of the gel time of the resin at the temperature of the impregnation should elapse before the above operation is completed.

Unless otherwise specified, mould release agents facilitating the removal of the moulded plates should be used only if those agents are not soluble in the resin and if such substances have been proved to exert no influence on the properties of either the plate or the test specimens to be made from it. The observance of this point is particularly important if the specimens are to be tested for electrical properties, flame resistance, or freedom from taste and odour. Polytetrafluorethylene and silicones can normally be used.

Using the type 1 mould, it is recommended that the impregnated glass reinforcement be covered with a second layer of foil or film before it is placed between the plates of the mould. The impregnated glass reinforcement thus is pressed between the two layers of foil or film, both of which should be at least 30 % larger than the dimensions of the glass reinforcement. Strip these films or foils from the plate after it has been moulded and cooled.

4.1.4 Moulding procedure depends considerably on the properties of the resin. Unless otherwise specified, the mould, heated to the required temperature, shall be closed and the moulding pressure shall be attained before the gelification of the resin at the given moulding temperature starts. The time between the closing of the mould and attainment of the required moulding force shall be specified (this time is reckoned from the first deflection of the pressure measuring instrument to the attainment of the value required) with as uniform a rise of force as possible. The moulding force shall be kept constant (see 3.1) for the prescribed period of time.

4.1.5 When the type 2 mould is used, the glass content of the moulded plate and its thickness are functions of temperature, pressure, and other factors depending upon the properties of the glass reinforcement and of the resin. For this reason it will be necessary, before making the number of plates or panels required for preparation of the test specimens, to determine by experiment the number of layers of glass reinforcement and the moulding pressure necessary to obtain plates or panels of the required thickness. When the type 1 mould (see figure 1) is used, the number of layers can be calculated (see 4.3.3) and the thickness of the plate can be governed by the use of appropriate spacers. The magnitude and tolerances of the thickness, within and between plates, shall be in agreement with the requirements of the testing method. Where the glass content is not subject to separate agreement, the laminate produced shall contain 60 ± 2 % (m/m) of glass in the case of fabrics and 40 ± 4 % (m/m) in the case of mats. Unless otherwise specified, the principal directions of all layers of the reinforcement shall be the same. In other cases the orientation of every individual layer of the reinforcement shall be stated using schematic drawing.

4.1.6 The temperature and time of curing shall be stipulated by agreement, depending on the type of resin, catalyst, or curing agent. The temperature stipulated shall

be so maintained during the curing cycle that the temperature values indicated by the temperature check sensitive elements remain within the ranges shown in the diagram in figure 3; line 1 represents temperature fluctuations during curing times up to 30 min; line 2 over 30 min.

4.1.7 After completion of the moulding process take the test plate from the mould and cool in such a way that any deformation, damage, etc. is avoided.

4.1.8 Unless otherwise stipulated by agreement, use the plates without further treatment for making test specimens by machining. If not specified in the relevant International Standards on testing, the types, sizes and orientations of the specimens with respect to the orientation of the glass reinforcement shall be stipulated by separate agreement. The margins of the plate up to at least 15 mm from its edges shall be discarded.

4.2 Method B

4.2.1 Shear, cut or chop the glass reinforcement, impregnated with resin partially cured to the B stage and no longer sticky, into squares, the sides of which are 3 to 6 mm less than the dimensions of the cavity of the tool.

4.2.2 If necessary, store the test pieces, prior to moulding, in a desiccator or at reduced temperature.

4.2.3 Lay the required number of layers of impregnated glass reinforcement freely on top of each other and then place in the cavity of the mould.

4.2.4 The rest of the working procedure is identical with that described in 4.1.

4.3 Method C

4.3.1 Shear, cut or chop the glass reinforcement to dimensions not less than 400 mm × 400 mm and dry as indicated in 4.1.2.

4.3.2 Carry out the impregnation of the glass reinforcement with liquid resin on a smooth rigid support pad, heated if necessary, and provided with a suitable release agent (see 4.1.3). Cover the support pad with a thin layer of catalyzed resin, then lay out the first layer of the glass reinforcement and cover again with a layer of resin, and so forth. Roll each layer, or each second layer (depending on the thickness of the glass reinforcement), before being covered with resin, with a special roller (see note below) so as to remove all visible air bubbles and to impregnate the glass reinforcement thoroughly with resin over the whole area. The rolling at the same time removes excess resin. An excess of at least 20 % of resin as compared with the resin content in the cured product shall be used for a lamination of the required thickness. Not more than 50 % of the gel time of the resin at the temperature of the impregnation should elapse before the end of the lamination. Pour the remaining resin over the last layer of glass reinforcement and then cover with a plane glass or similar plate. Provide this plate with a release agent. Press it down by hand without enclosure of bubbles until it contacts three spacers suitably positioned to control the thickness of the plate and leave it in that position until the resin is cured. Where rigid glass plates cannot ensure a smooth surface free from bubbles, flexible plates or foils may be used, in which case care shall be exercised to keep the thickness tolerance within the prescribed limits. Prepare the laminate at a relative humidity not exceeding 65 % unless otherwise agreed.

NOTE – Well-suited for this purpose are soft-faced rollers; for example, rollers covered with mohair tissue. A rod or paddle covered with polytetrafluorethylene may be used instead. Air bubbles in light-coloured resins can be seen more easily if the support pad for the lamination is dark-coloured or translucent and lighted from below.

4.3.3 The number of layers of glass reinforcement shall be such that the glass content of the cured plate is 50 ± 2 % (*m*/*m*) in the case of glass fabrics and 30 ± 4 % (*m*/*m*) in the case of mats. By agreement the plates may also contain other proportions of glass. For one lot, however, the glass content shall be constant within the range which shall be specified. The magnitude and tolerances of the thickness, within and between plates, shall be in agreement with the requirements of the testing method. For the determination of the required number of layers of glass reinforcement it will be necessary to make a preliminary experiment or to calculate this number by the use of the formula

$$n = \frac{e\rho_g \rho_n b}{g\,[b\rho_n + \rho_g(1-b)]}$$

where

n is the number of layers;

e is the thickness of the plate, in centimetres;

ρ_g is the density of the glass in grams per cubic centimetre;

ρ_n is the density of the cured resin in grams per cubic centimetre;

b is the glass content, by mass, expressed as a fraction of one;

g is the mass of glass reinforcement per unit area in grams per square centimetre.

4.3.4 Unless otherwise specified, either leave the laminated plate or panel, prepared as described in 4.3.2, for 48 h at room temperature, or place it, together with its support pad, in an oven or heated air bath. Curing at room

temperature may be accomplished at temperatures ranging from 20 to 25 °C. In the case of curing in an oven or heated air bath the temperature shall be checked by means of two sensitive elements situated not more than 50 mm above opposite corners of the plate. Temperatures indicated by these elements shall not vary more than shown in the graph, figure 3.

4.3.5 In the case of curing at room temperature, the plate may be taken off the support pad after 48 h but, unless otherwise specified, it shall then be left freely exposed for 21 days at room temperature in a standard atmosphere, according to ISO/R 291[1] on a plane surface. If less than 21 days elapse between curing of the plate at room temperature and the making of test specimens, the time elapsing between the making of the test specimens and their testing shall be exactly specified. The margins of the plate up to at least 20 mm from the edge shall not be used for making test specimens and the working portions of the specimens shall not be taken any closer than 40 mm from the edge.

4.3.6 In the case of curing at elevated temperatures, take the plate or panel, together with its support pad, out of the air bath at the end of the curing time and leave to cool freely for at least 60 min. After removal from the support pad the plate may be used for making test specimens with the exclusion of margins as specified in 4.3.5.

5 REPORT OF PREPARATION OF TEST PLATE OR PANEL

The report of preparation of the test plate shall include the following particulars :

a) place and date of production of test plate;

b) description of materials used for preparation of plate (nature and type of resin, catalyst, curing agent or other additives including the amounts used, nature and type of glass reinforcement, nature of finish, etc.);

c) description of production equipment (type of press, mould, method of checking temperatures and pressures, etc.);

d) working procedure (designation of method as given in this International Standard, moulding pressure (or force), temperature, curing time, release agent used, post-curing, etc.);

e) date indicating when test pieces are to be made;

f) dimensions of plate, mass of plate and its glass content;

g) special notes.

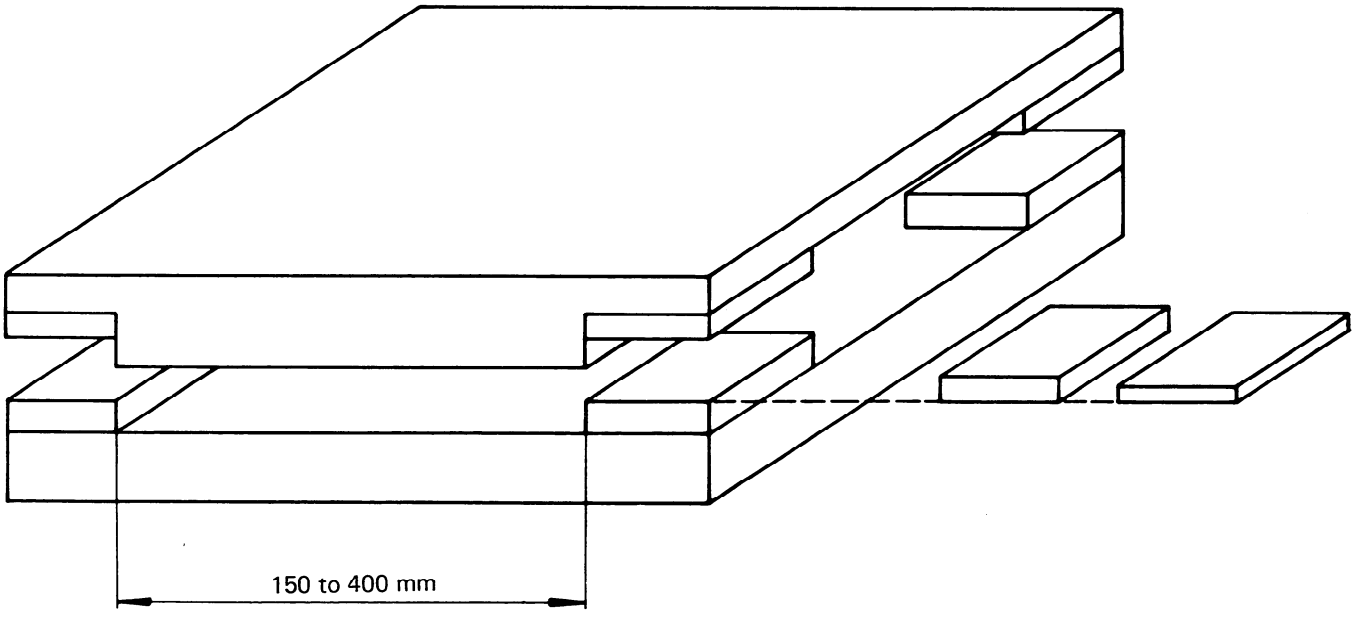
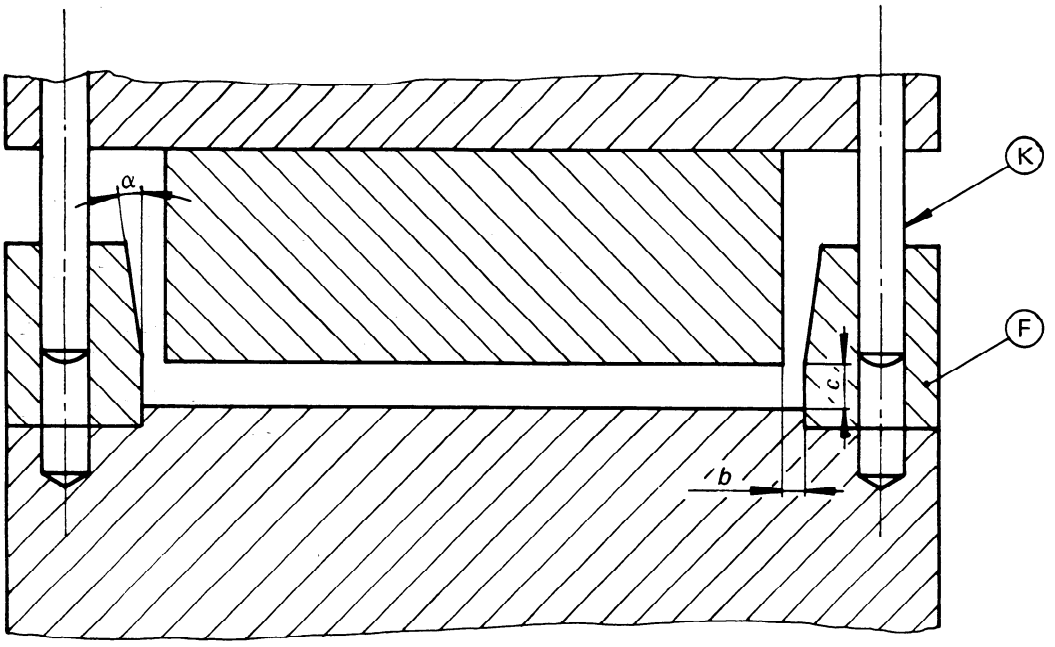


FIGURE 1 – **Type 1 mould**

1) ISO/R 291, *Plastics – Standard atmospheres for conditioning and testing.*



Section A-A

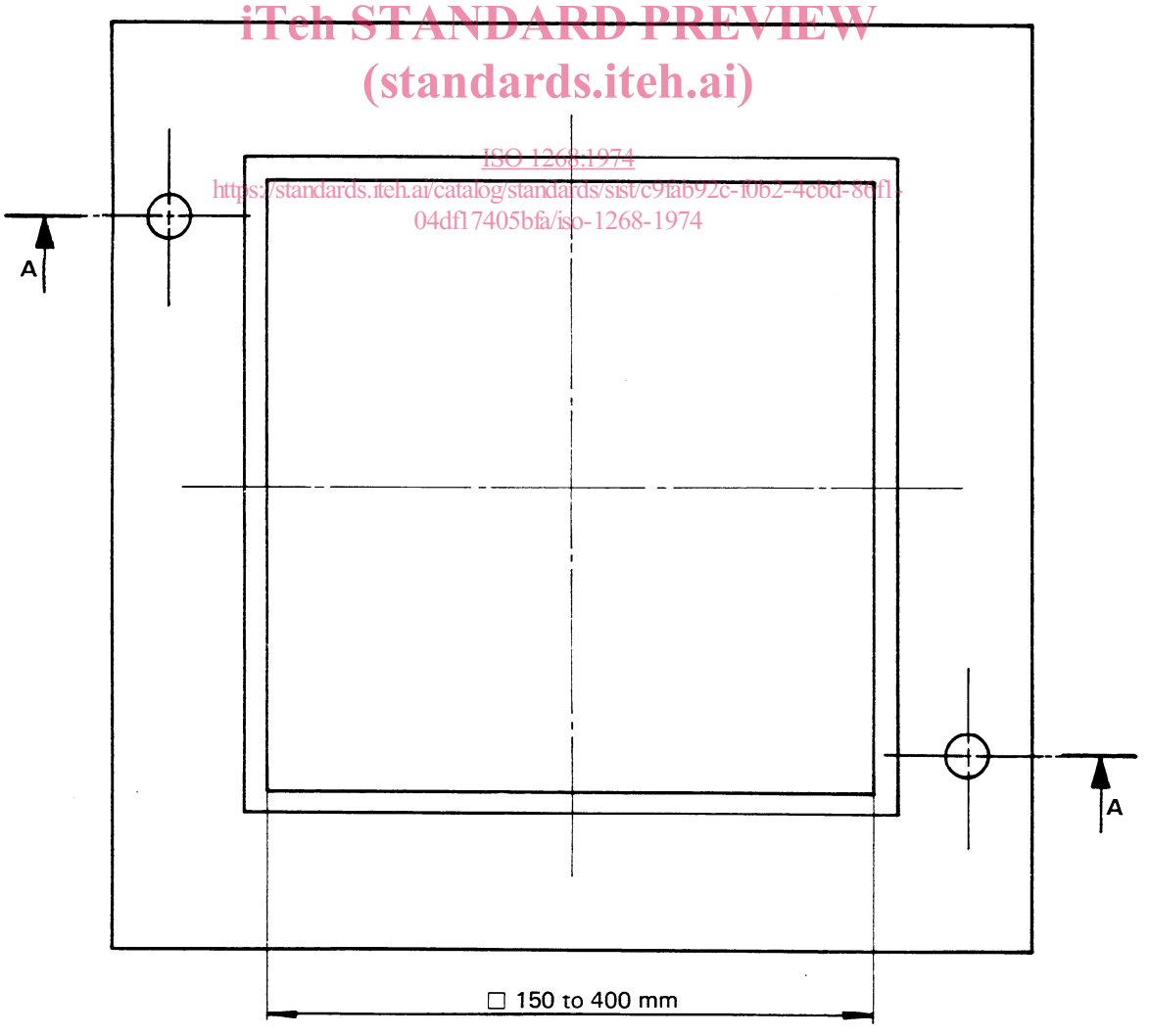


FIGURE 2 – **Type 2 mould**

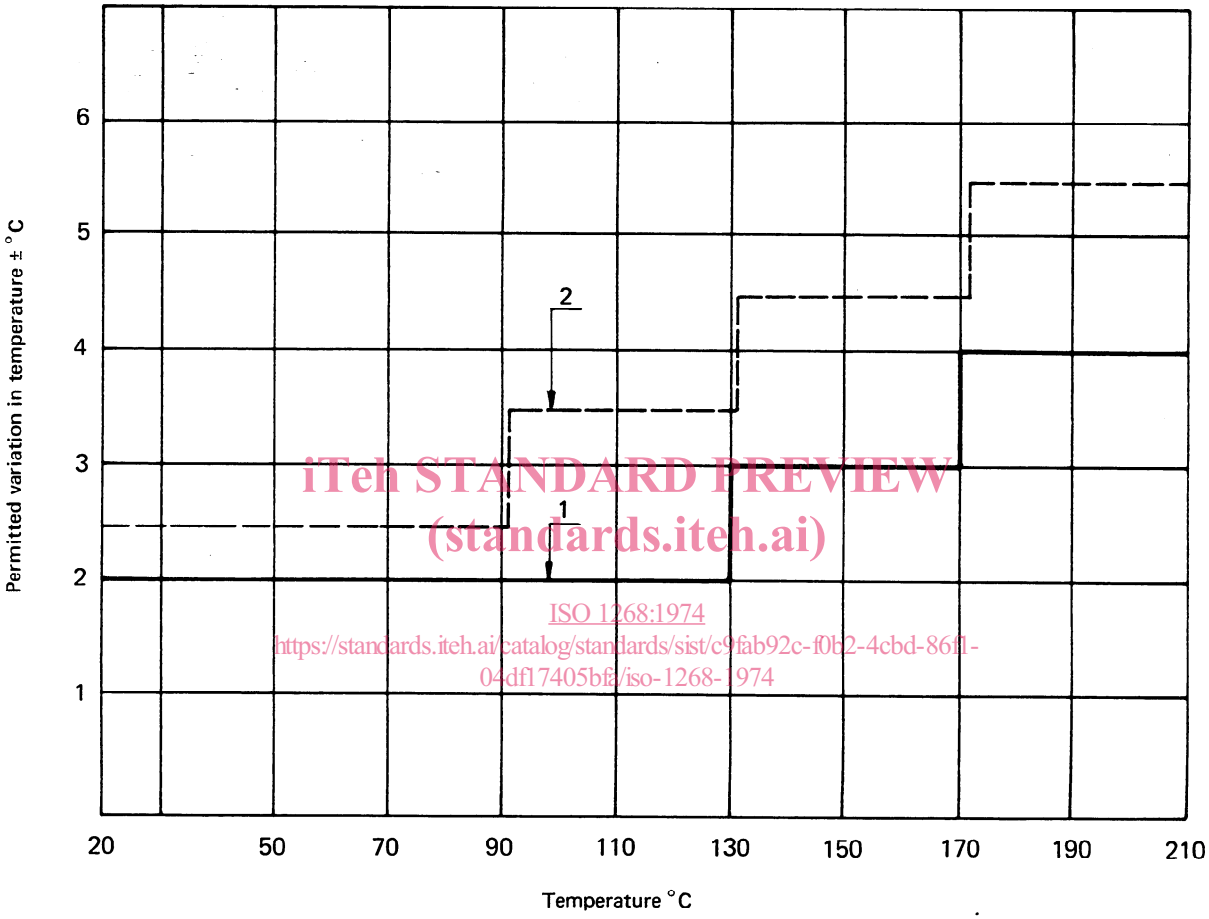


FIGURE 3 – **Permissible variation of curing temperatures (see 4.1.6)**