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Adhesives — Guide to the selection of standard laboratory ageing conditions for testing bonded joints

*Adhésifs — Guide pour la sélection de conditions normales d'essai de
vieillessement en laboratoire des assemblages collés*



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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9142 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Annexes A, B, C, D, E and F of this International Standard are for information only.

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Adhesives — Guide to the selection of standard laboratory ageing conditions for testing bonded joints

1 Scope

This International Standard describes laboratory ageing conditions under which adhesive joints may be exposed to various environmental influences — climatic or chemical — for the purpose of assessing the effects of such influences on certain properties.

The ageing conditions are applicable to bonded assemblies and may be used to constitute a set of tests for the evaluation of an adhesive.

The results obtained using the procedures described in this International Standard are not necessarily applicable to the determination of the service life of a bonded assembly because there is no direct relation between the test results and the behaviour of a bonded assembly over a period of time under service conditions. However, for certain specific applications, experience with the procedures may enable a correlation to be established.

NOTE 1 The ageing conditions should be related to the specific application of the adhesives.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 175:1981, *Plastics — Determination of the effects of liquid chemicals, including water.*

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 483:1988, *Plastics — Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value.*

ISO 3205:1976, *Preferred test temperatures.*

ISO 3768:1976, *Metallic coatings — Neutral salt spray test (NSS test).*

ISO 4587:1979, *Adhesives — Determination of tensile lap shear strength of high strength adhesive bonds.*

ISO 4588:1989, *Adhesives — Preparation of metal surfaces for adhesive bonding.*

ISO 4892:1981, *Plastics — Methods of exposure to laboratory light sources.*

ISO 6238:1987, *Adhesives — Wood-to-wood adhesive bonds — Determination of shear strength by compression loading.*

3 Principle

Definition of the ageing conditions that relate most closely to the application for the adhesive by reference to the preferred conditions set out in annex A to annex F.

4 Materials

4.1 **De-ionised or distilled water**, having a conductivity less than 200 $\mu\text{S}/\text{m}$.

4.2 **Materials for ageing tests** under special environmental conditions (see appropriate annex).

5 Apparatus

5.1 **Conditioning chamber**, meeting the requirements of ISO 483, capable of being maintained at a temperature of $(23 \pm 2)^\circ\text{C}$ and a relative humidity of $(50 \pm 5)\%$.

5.2 Dry-heat chamber, ventilated and adjustable to a temperature between 20 °C and 200 °C.

5.3 Humid chamber, meeting the requirements of ISO 483, adjustable to a relative humidity between 25 % and 100 %. The chamber shall be equipped with

- a) a device to enable the temperature to be measured to within 1 °C;
- b) a device to enable the relative humidity to be measured to within 3 % R.H.

5.4 Cold chamber, adjustable to a temperature of (-40 ± 3) °C.

5.5 Controlled-pressure chamber, capable of operating at a pressure of 0,6 MPa¹⁾ and at an under-pressure of 0,092 MPa.

6 Specimens

6.1 Number and type

The number of test specimens, and the type used, will depend on the properties to be measured and the required ageing conditions. The test specimens shall be prepared in accordance with the requirements of the appropriate ISO standard test method. Prepare a sufficient number of specimens to provide homogeneous sets of samples for testing and to provide control samples. The following standards, which do not constitute an exhaustive list, are applicable: ISO 4587, ISO 4588, ISO 6238.

6.2 Conditioning

Specimens shall be conditioned in the conditioning chamber (5.1) for a minimum duration of 24 h before being subjected to ageing. The conditioning shall be carried out after the adhesive has been cured in accordance with the materials specification or the methods specified by the manufacturer of the adhesive.

7 Procedure

7.1 Tests before ageing

Determine the properties required for the sample under test by the appropriate test method, using specimens conditioned in accordance with 6.2.

¹⁾ 1 MPa = 10 bar

7.2 Ageing

Expose conditioned specimens to the environmental conditions as defined in annex A to annex F, as required.

7.3 Tests after ageing

Determine the properties after ageing in accordance with the procedure(s) of the appropriate standard(s). Unless there are indications to the contrary in the annexes, condition test specimens in the conditioning chamber (5.1)

- for (3 ± 1) h in the case of specimens exposed at a relative humidity higher than 50 %,
 - for at least 24 h for all other specimens,
- before determining the properties.

In addition to the test results, all changes visible during the ageing and testing periods, including mode of failure, shall be noted.

7.4 Controls

For control purposes, a representative group of specimens shall be kept in the conditioning chamber (5.1) for the period of time covered by the ageing conditions selected.

8 Expression of results

For properties that can be measured, the results obtained are expressed as either

- absolute values or
- a relative variation with respect to the initial value.

For properties that cannot be measured, the nature of the effects observed shall be noted, e.g. rupture between the substrate and adhesive, failure of cohesion of the adhesive joint due to partial or total corrosion of the substrate beneath the adhesive, etc.

NOTE 2 It may be worth verifying that the variation in a measured property is caused only by the ageing process from the following three values:

- A* the value of the property measured before accelerated ageing;
- B* the value of the same property measured on the control specimen which has undergone only intrinsic ageing at (23 ± 2) °C and (50 ± 5) % R.H. (see 7.4);
- C* the value of the property measured after ageing.

This can be done by calculating the following ratios:

- a) $[(A - B)/A] \times 100$ (this ratio represents the intrinsic ageing during the period concerned);
 - b) $[(A - C)/A] \times 100$ (this ratio represents the overall ageing due to intrinsic ageing and the accelerated ageing under the conditions specified in this International Standard);
 - c) $[(B - C)/A] \times 100$ (this ratio represents the degree of ageing due to ageing under the conditions specified in this International Standard).
- a) a reference to this International Standard;
 - b) a description of the type of ageing carried out, with reference to the relevant annex(es) and the duration of exposure and number of test cycles;
 - c) the results of observations made in accordance with 7.3 (last paragraph) and clause 8;
 - d) details of any operations not laid down in this International Standard, as well as any incidents liable to have had an effect on the results.

9 Test report

The test report shall include the following information:

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Annex A (informative)

General conditions of ageing

Whatever the type of ageing carried out, the temperature and duration of exposure, as well as the relative humidity, shall, as far as possible, be selected from the values given below. Other values may be used if this is absolutely essential for technical reasons, but they shall be selected initially from the values given in ISO 3205 and ISO 291 (the latter gives temperatures only).

A.1 Preferred temperatures

Preferred temperatures are given, together with tolerances, in table A.1.

A.2 Preferred exposure periods

Hours: 1 - 2 - 3 - 4 - 6 - 16 - 24 - 48 - 72 - 96

Weeks: 1 - 2 - 3 - 4 - 6 - 8 - 12 - 16 - 26 - 52

The number of cycles shall be selected to approach, as closely as possible, these preferred exposure periods.

A.3 Preferred relative humidities

< 15 %; (25 ± 5) %; (50 ± 5) %; (65 ± 5) %; 90 % to 100 %.

Table A.1 — List of preferred temperatures and tolerances

Values in degrees Celsius

Temperature	Tolerances (to be used only in the absence of special indications calling, in particular, for stricter tolerances)
- 40	± 3
- 20	± 3
+ 20	± 2
+ 23	± 2
+ 30	± 2
+ 40	± 2
+ 50	± 2
+ 55	± 2
+ 60	± 2
+ 70	± 2
+ 90	± 2
+ 100	± 2
+ 105	± 3
+ 125	± 3
+ 175	± 3

Annex B

(informative)

Atmospheric conditions for single-variable ageing

In this type of ageing, the effect on the test specimen of only one environmental variable (e.g. temperature) is considered, the other variables being fixed.

B.1 Set of conditions B1

The specimen is exposed to dry heat in the dry heat chamber (5.2), the temperature and period of exposure being selected from the lists of preferred values in annex A.

B.2 Set of conditions B2

The specimen is exposed to cold in the cold chamber (5.4), the temperature and period of exposure being selected from the lists of preferred values in annex A.

Annex C

(informative)

Atmospheric conditions for multi-variable ageing

In this type of ageing, the simultaneous effect of two or more environmental variables on the specimen is considered. In some cases, transfers from one chamber to another are necessary; unless otherwise specified, such transfers shall be carried out in less than 3 min.

Set of conditions C1

The specimen is exposed in the humid chamber (5.3) to the simultaneous action of heat and humidity. The temperature, exposure period and humidity are selected from the lists of preferred values in annex A.

NOTE 3 A further-accelerated ageing test using humid heat may be carried out at $(70 \pm 2)^\circ\text{C}$ and 90 % to 100 % humidity for 1 000 h.

Annex D
(informative)

Atmospheric conditions for cyclic ageing

In this type of ageing, the specimen is exposed for several successive periods, under single-variable and/or multi-variable conditions, on a cyclic basis. In some cases, transfers from one chamber to another are necessary; unless otherwise specified, such transfers shall be carried out in less than 3 min.

The following cycles may be used:

Cycle D1 — Heat and humidity cycle

Cycle D2 — Heat, cold (thermal shock) and humidity cycle

Cycle D3 — Heat, cold and humidity cycle

Cycle D4 — Humid-heat and cold cycle

Cycle D5 — Dry-heat, humid-heat and cold cycle

Cycle D6 — Immersion in water and drying

Cycle D7 — Alternating immersion in water and drying

Cycle D8 — Immersion in boiling water and drying

Cycle D9 — Immersion in boiling water followed by drying, and then dry heat and re-immersion in boiling water

Cycle D10 — Immersion in boiling water followed by drying, and then re-immersion in boiling water

Cycle D11 — Immersion in water, exposure to cold and exposure to dry heat

Cycle D12 — Immersion in salt solution and drying

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D.1 Cycle D1 — Heat and humidity cycle

Humidity and period of exposure are fixed, while the temperature is varied. Two temperatures θ_A and θ_B are selected from table A.1 in annex A.

The cycle requires two humid chambers (5.3) as follows:

- chamber A, kept at temperature θ_A °C and a relative humidity of no lower than 90 %;
- chamber B, kept at temperature θ_B °C and a relative humidity of not more than 30 %.

The cycle consists of the following exposure periods:

- a) 24 h in chamber A;
- b) 24 h in chamber B;
- c) 72 h in chamber A;
- d) 48 h in chamber B.

For the number of cycles, see annex A, clause A.2.

For conditioning after ageing, see 7.3.

A typical cycle, in which θ_A is taken as (23 ± 2) °C and θ_B as (55 ± 2) °C, is shown in figure D.1.

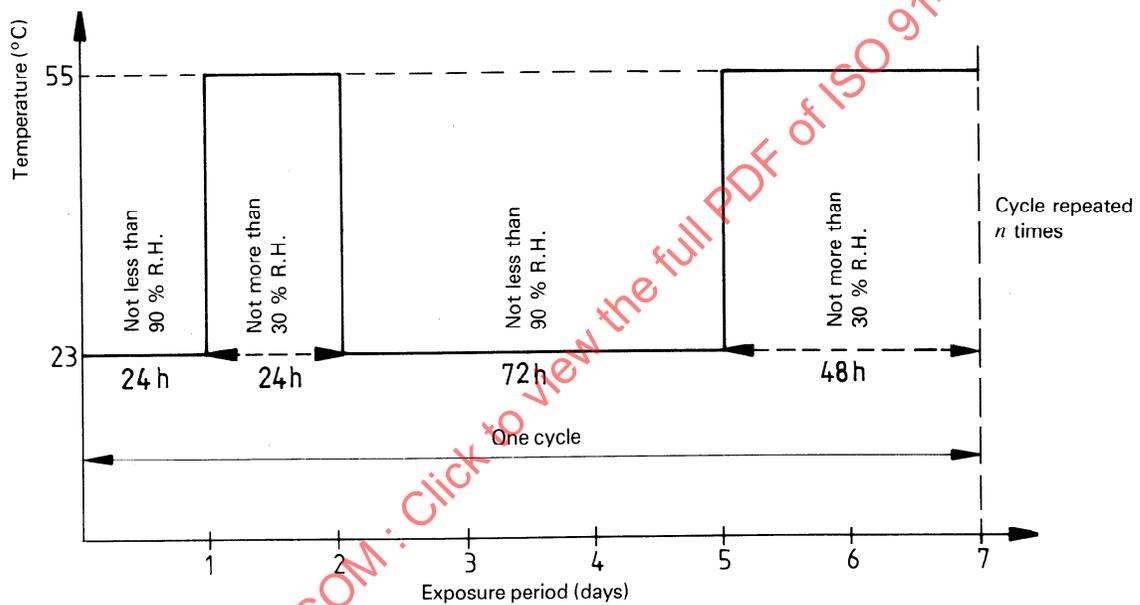


Figure D.1 — Typical conditions for cycle D1, in which θ_A and θ_B are taken as (23 ± 2) °C and (55 ± 2) °C, respectively

D.2 Cycle D2 — Heat, cold (thermal shock) and humidity cycle

This cycle requires three chambers, as follows:

- a humid chamber A (5.3), kept at a temperature θ_A of either $(40 \pm 2)^\circ\text{C}$ or $(70 \pm 2)^\circ\text{C}$ and at a relative humidity not lower than 90 %;
- a cold chamber B (5.4), kept at a temperature θ_B of either $(-20 \pm 3)^\circ\text{C}$ or $(-40 \pm 3)^\circ\text{C}$;
- a humid chamber C (5.3), kept at a temperature θ_C of either $(70 \pm 2)^\circ\text{C}$ or $(100 \pm 2)^\circ\text{C}$ and at a relative humidity of $(50 \pm 5) \%$.

The cycle consists of the following exposure periods:

- a) (16 ± 1) h in chamber A;
- b) (3 ± 1) h in chamber B;
- c) (5 ± 1) h in chamber C.

For the number of cycles, see annex A, clause A.2.

For conditioning after ageing, see 7.3.

A typical cycle, in which temperature θ_A is taken as 40°C , θ_B as -20°C and θ_C as 70°C , is shown in figure D.2.

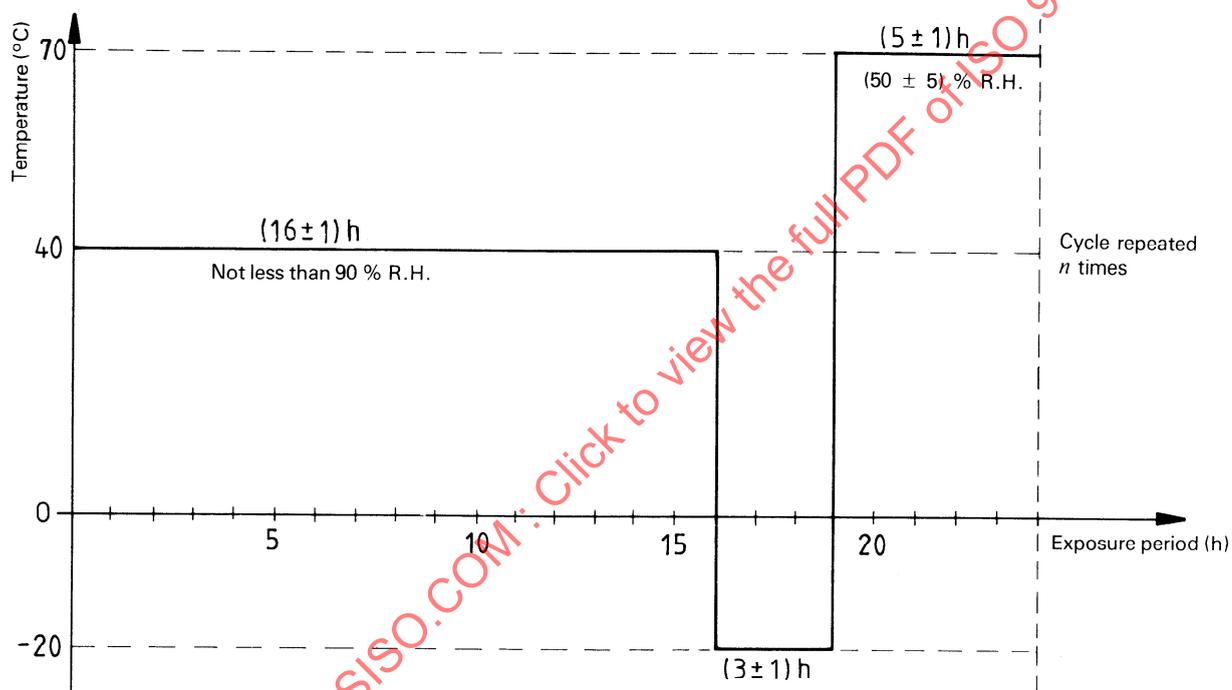


Figure D.2 — Typical conditions for cycle D2, in which θ_A , θ_B and θ_C are taken as 40°C , -20°C and 70°C , respectively

D.3 Cycle D3 — Heat, cold and humidity cycle

This cycle requires a programmable humid chamber (5.3) capable of maintaining the required conditions of temperature and humidity and able to change the conditions in the time specified in each case.

The cycle consists of the following operations:

- expose for (15 ± 1) h at a temperature of either (40 ± 2) °C or (70 ± 2) °C and at a relative humidity not lower than 90 %;
- change, in (60 ± 20) min, the temperature to (-20 ± 3) °C or (-40 ± 3) °C and expose for (2 ± 1) h;

c) change, in (60 ± 20) min, the temperature to (70 ± 2) °C or (10 ± 2) °C and the relative humidity to (50 ± 5) % and expose for (4 ± 1) h;

d) change, in (60 ± 20) min, the temperature to (40 ± 2) °C or (70 ± 2) °C and the relative humidity to not lower than 90 %.

At the end of the final cycle, change from the conditions in d) to the conditioning atmosphere (see 5.1) in (60 ± 20) min.

For the number of cycles, see annex A, clause A.2.

For conditioning after ageing, see 7.3.

A typical cycle, in which the temperature of the first stage is taken as 40 °C, that of the second stage as -20 °C and that of the third stage as 70 °C, is shown in figure D.3.

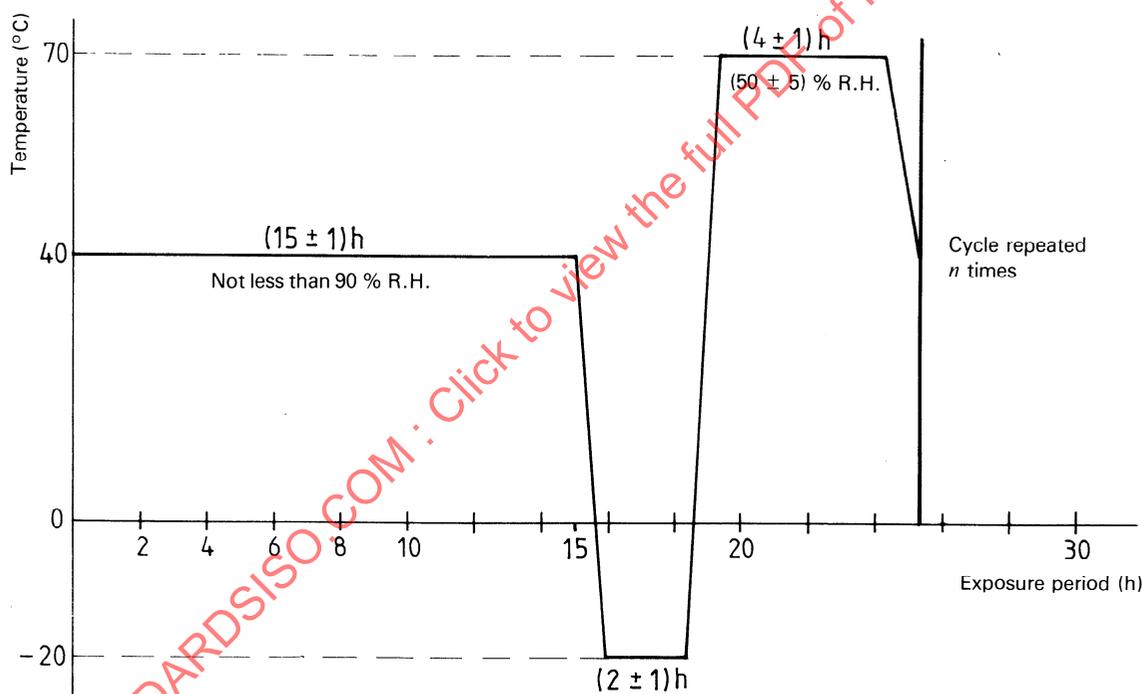


Figure D.3 — Typical conditions for cycle D3, in which the temperatures of the first, second and third stages are 40 °C, -20 °C and 70 °C, respectively

D.4 Cycle D4 — Humid-heat and cold cycle

This cycle requires a programmable humid chamber (5.3) capable of maintaining the required conditions of temperature and humidity and able to change the conditions in the time specified in each case.

The cycle consists of the following operations:

- a) increase the temperature from ambient to $(70 \pm 2)^\circ\text{C}$ in 30 min (this operation is carried out at the beginning of the first cycle only);

- b) expose for $5\text{ h} \pm 20\text{ min}$ at $(70 \pm 2)^\circ\text{C}$ and at a relative humidity not lower than 90 %;
- c) change the temperature, in $(60 \pm 20)\text{ min}$, to $(-40 \pm 3)^\circ\text{C}$ and expose for $5\text{ h} \pm 20\text{ min}$;
- d) change the temperature, in $(60 \pm 20)\text{ min}$, to $(70 \pm 2)^\circ\text{C}$.

For the number of cycles, see annex A, clause A.2.

For conditioning after ageing, see 7.3.

A typical cycle is shown in figure D.4.

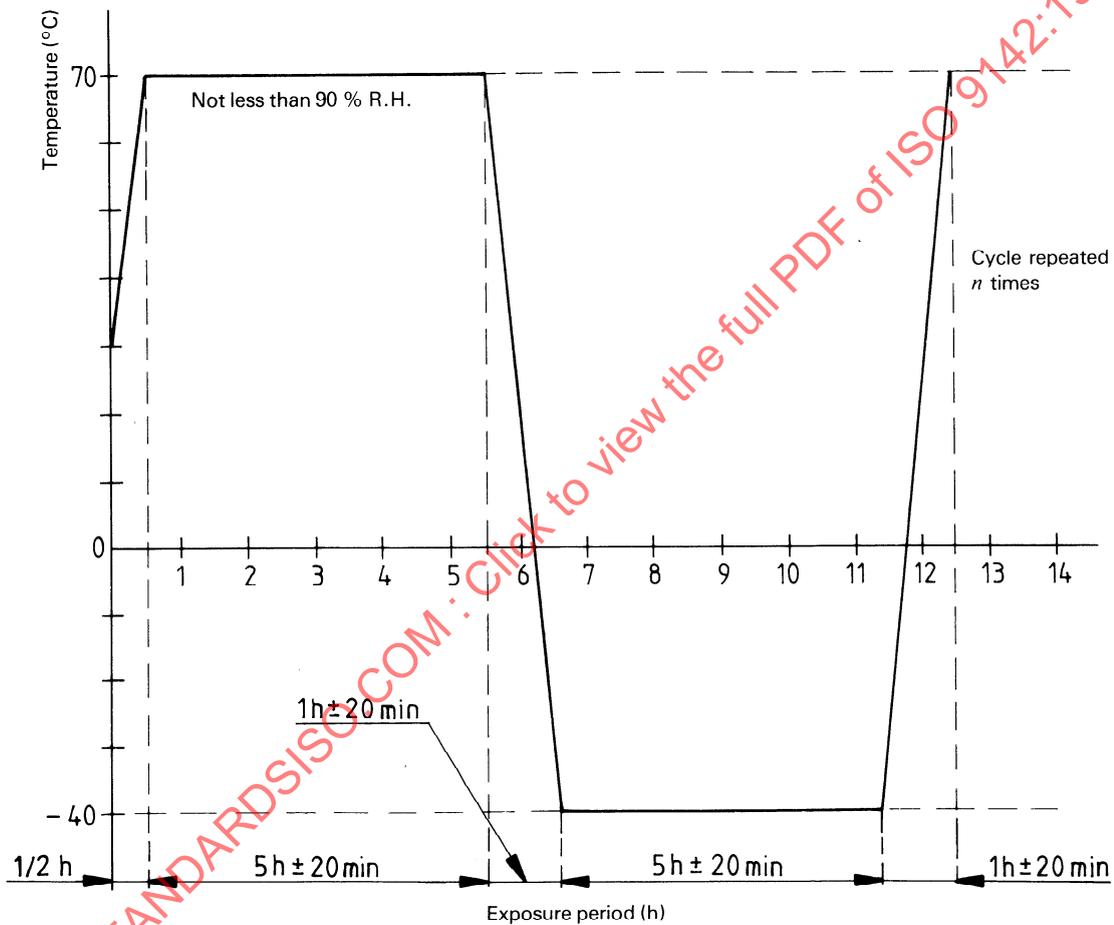


Figure D.4 — A typical humid-heat and cold cycle

D.5 Cycle D5 — Dry-heat, humid-heat and cold cycle

This cycle requires three chambers as follows:

- a dry-heat chamber A (5.2), kept at a temperature of (55 ± 2) °C and at a relative humidity lower than 15 %;
- a humid chamber B (5.3), kept at a temperature of (40 ± 2) °C and at a relative humidity not lower than 90 %;
- a cold chamber C (5.4), kept at a temperature of (-20 ± 2) °C.

The cycle consists of the following exposure periods:

- a) (48 ± 1) h in chamber A;
- b) (48 ± 1) h in chamber B;
- c) (24 ± 1) h in chamber C;
- d) (48 ± 1) h in chamber B.

For the number of cycles, see annex A, clause A.2.
For conditioning after ageing, see 7.3.

A typical cycle is shown in figure D.5

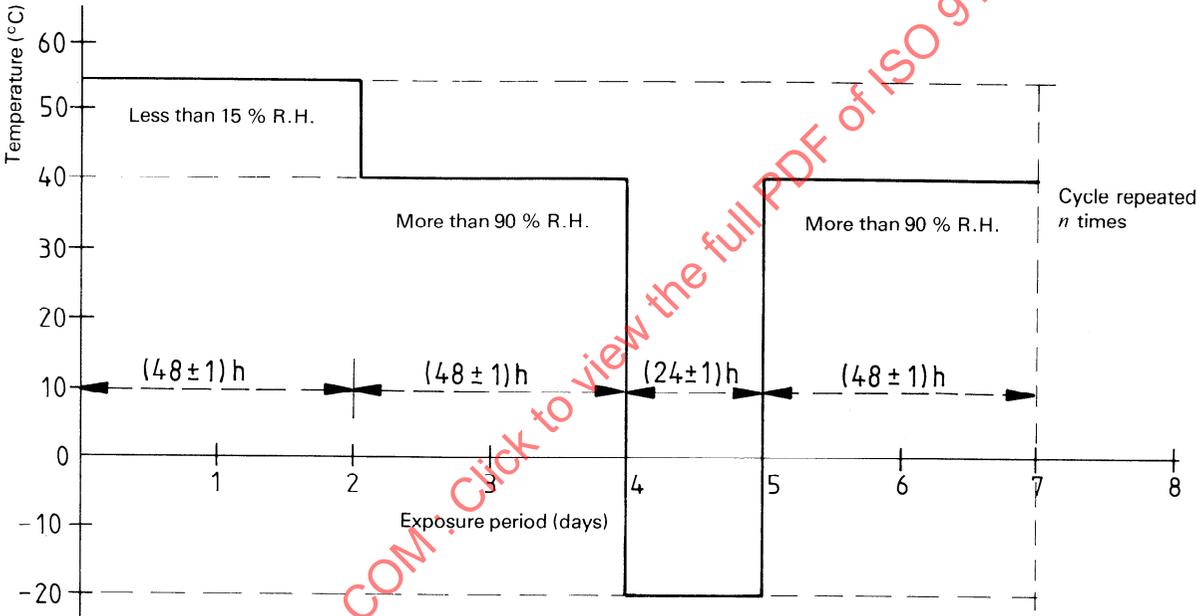


Figure D.5 — A typical dry-heat, humid-heat and cold cycle

D.6 Cycle D6 — Immersion in water and drying

NOTE 4 This cycle is recommended for ageing porous materials (e.g. wood).

The cycle requires two chambers, as follows:

- a controlled-pressure chamber A (5.5), kept at a temperature of $(23 \pm 2)^\circ\text{C}$ and the pressure of which is increased to 0,6 MPa and lowered to an underpressure of 0,092 MPa;
- a humid chamber B (5.3), kept at a temperature of $(23 \pm 2)^\circ\text{C}$ and at a relative humidity of $(30 \pm 5)\%$.

The cycle consists of the following operations:

- a) expose the test specimens, immersed in water, for 15 min in chamber A at an underpressure of 0,092 MPa;

- b) change the pressure to 0,6 MPa and expose for 2 h;
- c) change the pressure to 0,092 MPa and expose for 15 min;
- d) change the pressure to 0,6 MPa and expose for 2 h;
- e) transfer to chamber B and expose for 10 days.

For conditioning after ageing, see 7.3.

For materials whose moisture content changes slowly (e.g. wood), mechanical tests shall be carried out, if required, either taking account of the moisture content of the material at the time of test or extending the conditioning time to obtain a given moisture content.

A typical cycle is shown in figure D.6.

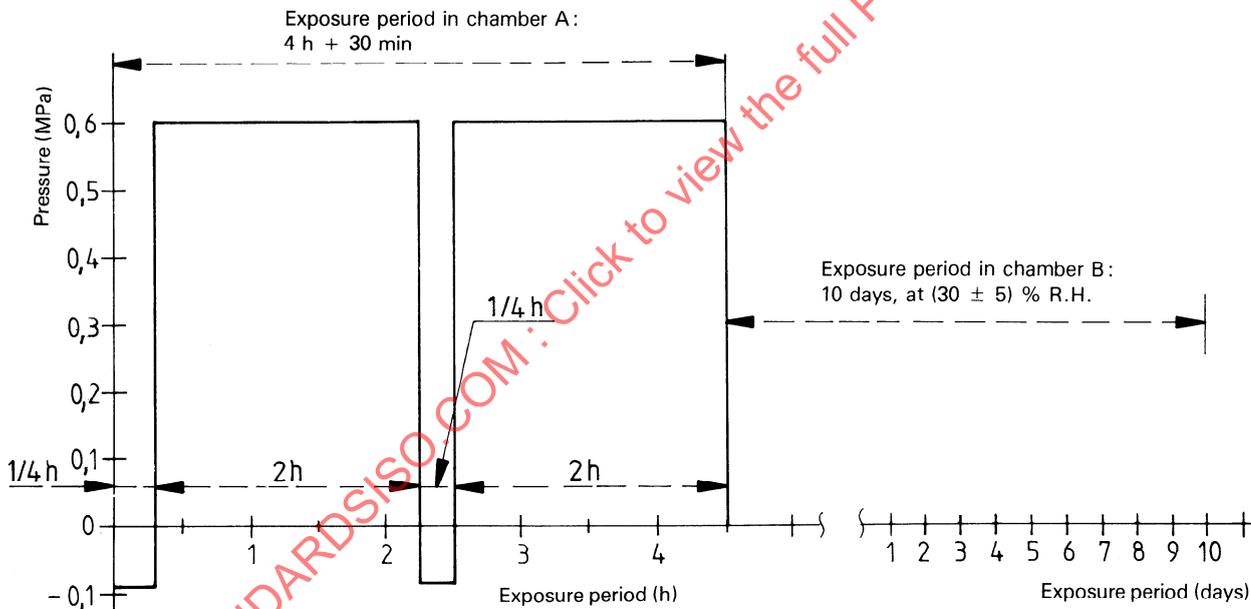


Figure D.6 — A typical cycle for immersion in water and drying

D.7 Cycle D7 — Alternating immersion in water and drying

NOTE 5 This cycle is recommended for ageing porous materials (laminates, etc.).

The cycle requires two chambers, as follows:

- a dry-heat chamber A (5.2), kept at a temperature of $(23 \pm 2) ^\circ\text{C}$;
- a humid chamber B (5.3), kept at a temperature of $(55 \pm 2) ^\circ\text{C}$ and at a relative humidity of less than or equal to 25 %.

The cycle consists of the following operations:

- a) expose the test specimens, immersed in water, for 64 h in chamber A;

- b) expose for 8 h in chamber B;
- c) expose, re-immersed in water, for 16 h in chamber A;
- d) expose for 8 h in chamber B;
- e) expose, re-immersed in water, for 16 h in chamber A;
- f) expose for 8 h in chamber B;
- g) expose, re-immersed in water, for 16 h in chamber A.

Conditioning after ageing: none; test the specimens immediately on completion of stage g), i.e. in the wet condition.

A typical cycle is shown in figure D.7.

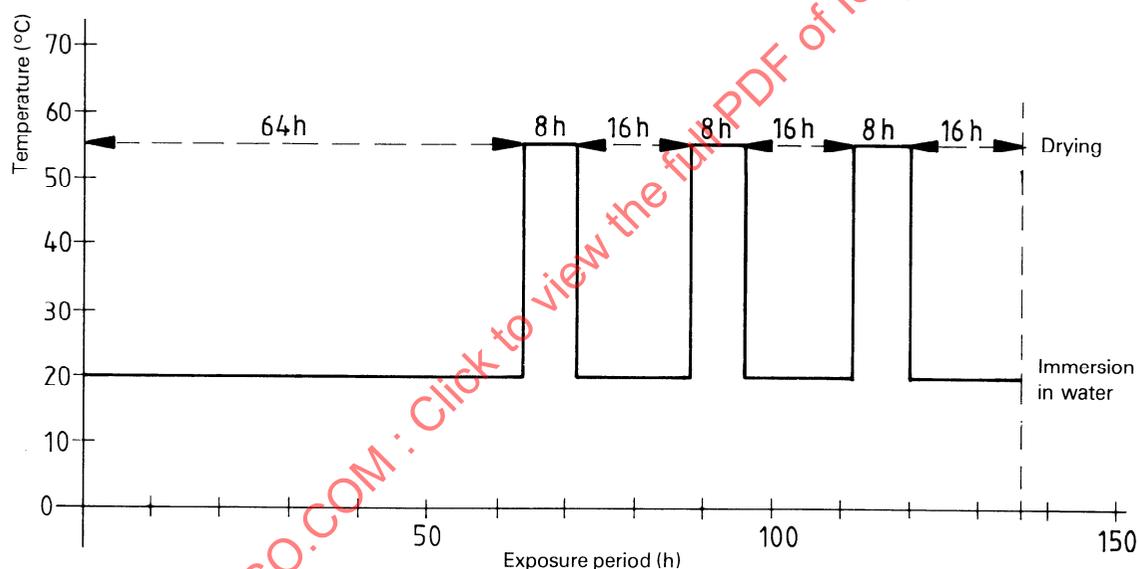


Figure D.7 — A typical cycle for alternating immersion in water and drying

D.8 Cycle D8 — Immersion in boiling water and drying

NOTE 6 This cycle is recommended for ageing porous materials, in particular laminates bonded with phenolic adhesives.

The cycle requires three test chambers, as follows:

- a dry-heat chamber A (5.2), kept at a temperature of $(100 \pm 2) \text{ }^\circ\text{C}$ (since water boils at $100 \text{ }^\circ\text{C}$ only at an atmospheric pressure of 0,1 MPa, the temperature of the boiling water is a variable and shall be reported);
- a humid chamber B (5.3), kept at a temperature of $(55 \pm 2) \text{ }^\circ\text{C}$ [when testing laminates, a temperature of $(50 \pm 2) \text{ }^\circ\text{C}$ shall be used in order to ensure commonality with existing procedures] and at a relative humidity of $(30 \pm 5) \%$;

- a dry-heat chamber C (5.2), kept at a temperature of $(23 \pm 2) \text{ }^\circ\text{C}$.

The cycle consists of the following operations:

- a) expose the test specimen, immersed in water, for $(4 \pm 0,1) \text{ h}$ in chamber A;
- b) expose for 20 h in chamber B;
- c) expose, re-immersed in water, for $(4 \pm 0,1) \text{ h}$ in chamber A;
- d) expose for 1 h in chamber C.

Conditioning after ageing: none; test the specimens immediately on completion of stage d), i.e. in the wet condition.

A typical cycle is shown in figure D.8.

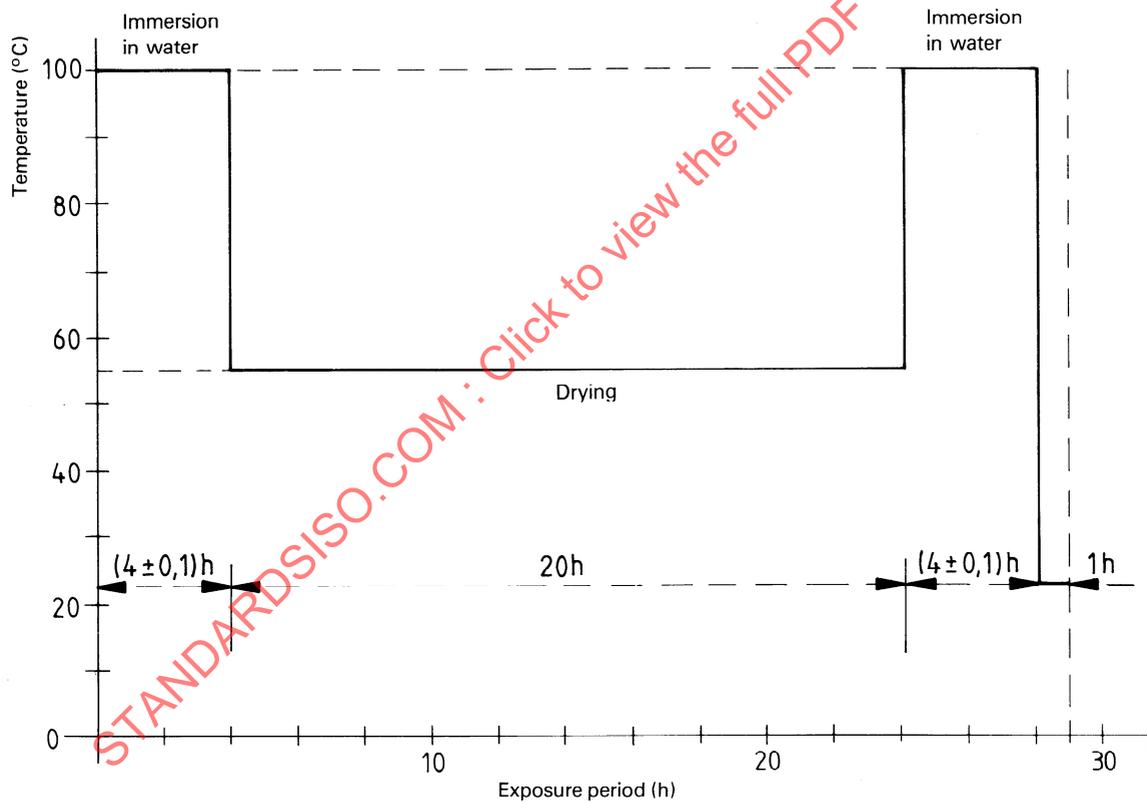


Figure D.8 — A typical cycle for immersion in boiling water and drying

D.9 Cycle D9 — Immersion in boiling water followed by drying, and then dry heat and re-immersion in boiling water

NOTE 7 This cycle is recommended for testing wood laminates.

The cycle requires three dry-heat chambers (5.2), as follows:

- chamber A, kept at a temperature of $(100 \pm 2)^\circ\text{C}$ (since water boils at 100°C only at an atmospheric pressure of 0,1 MPa, the temperature of the boiling water is a variable and shall be reported);
- chamber B, kept at a temperature of $(23 \pm 2)^\circ\text{C}$ and provided with a controlled air-flow of $(1,75 \pm 0,3) \text{ m/s}$;

- chamber C, kept at a temperature of $(105 \pm 3)^\circ\text{C}$ and provided with a controlled air-flow of $(1,75 \pm 0,3) \text{ m/s}$.

The cycle consists of the following operations:

- a) expose the test specimens, immersed in water, for 10 min in chamber A;
- b) expose for 4 min in chamber B;
- c) expose for 57 min in chamber C.

For the number of cycles, see annex A, clause A.2.

Conditioning after ageing: at the end of the final cycle, transfer to water at $(23 \pm 2)^\circ\text{C}$ and maintain for at least 3 days. Testing shall be carried out on wet specimens.

A typical test cycle is shown in figure D.9.

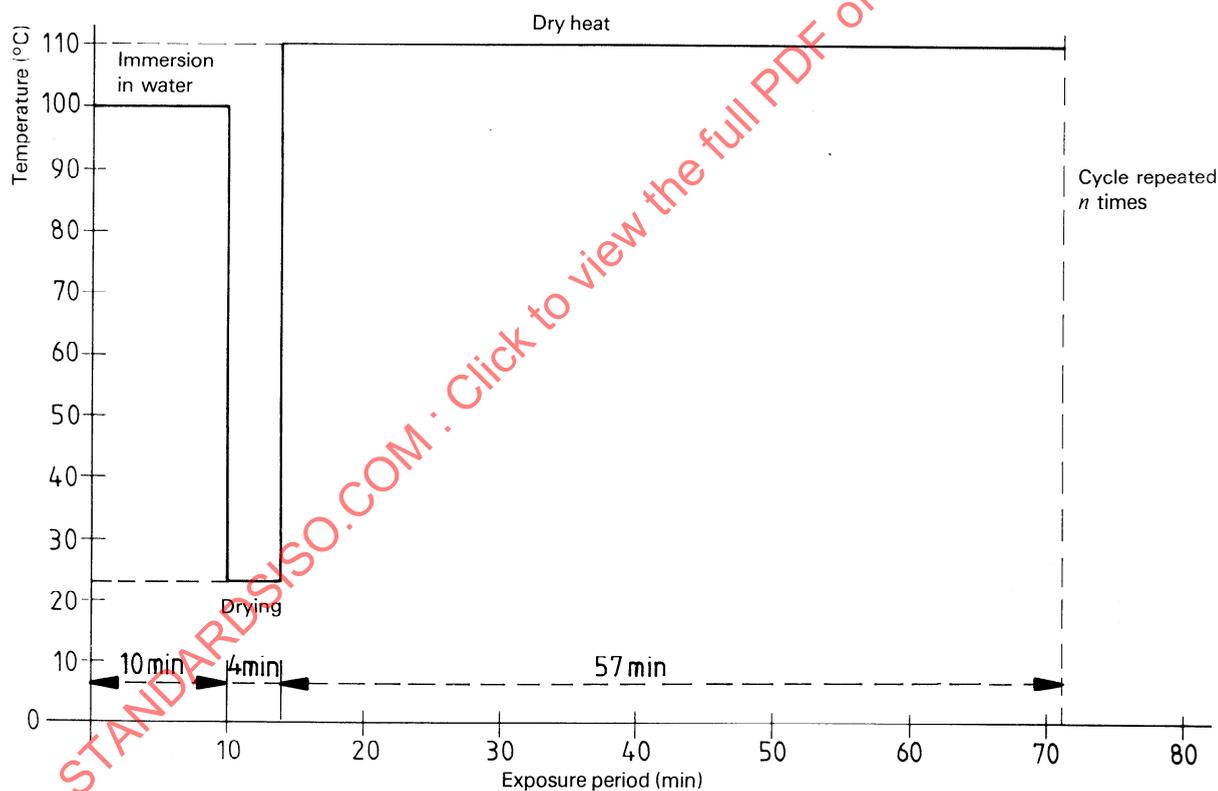


Figure D.9 — A typical cycle for immersion in boiling water followed by drying, and then dry heat and re-immersion in boiling water