INTERNATIONAL STANDARD

ISO 14615

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Adhesives — Durability of structural adhesive joints — Exposure to humidity and temperature under load

Adhésifs — Durabilité des joints adhésifs structuraux — Exposition à l'humidité et à la température sous contrainte

Reference number
ISO 14615:1997(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. 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 14615 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

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Adhesives – Durability of structural adhesive joints – Exposure to humidity and temperature under load

1 Scope

This International Standard describes the equipment and the test procedure used to evaluate the durability of adhesive-bonded joints exposed simultaneously to heat, humidity and load.

This method is intended for the evaluation of structural and semi-structural adhesives used in hot and/or humid environments. It is particularly suitable for metal, composite and plastic adherends.

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 using the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4588:1995, *Adhesives - Guidelines for the surface preparation of metals.*

ISO 9142:1990, *Adhesives - Guide to the selection of standard laboratory ageing conditions for testing bonded joints.*

ISO 10365:1992, *Adhesives - Designation of main failure patterns.*

ISO 13895:1996, *Adhesives - Guidelines for the surface preparation of plastics.*

3 Principle

This method consists of testing lap-shear or T-joint specimens under static load at elevated temperature and high humidity. The specimens are placed in a tube equipped with a pre-calibrated spring system for loading the specimens. The times to failure of the loaded specimens are recorded.

4 Apparatus

4.1 Humidity cabinet

The cabinet shall be such that the relative humidity can be maintained at 95% to 100% and that the temperature can be cycled between 42°C and 48°C (see 4.2). The cabinet shall be insulated and shall have an open, heated water reservoir in the base from which distilled or deionised water is pumped to spray bars at each side of the cabinet. The top of the cabinet shall have holes through which an appropriate number of specimen-loading tubes can be inserted so that they hang within 100 mm of the cabinet walls.

4.2 Heater and cooling unit

The heater shall be of sufficient capacity to raise the cabinet temperature from 42°C to 48°C in 30 min. The radiator-type cooling unit shall be of sufficient capacity to lower the cabinet temperature from 48°C to 42°C in 30 min.

The heater and a cooling fan mounted on the radiator shall be regulated by a programmed controller linked to a temperature sensor located inside the cabinet at least 100 mm from the cabinet walls.

The controller shall be such that the temperature in the operating range is accurate to +/- 0,25°C and the temperature gradient in the cabinet does not exceed +/- 0,5°C.

4.3 Water circulation system

The heated water from the reservoir shall be circulated through the radiator into two spray bars mounted on opposite sides near the top of the cabinet (see figure 1), from where it is sprayed on to the cabinet walls.

The water level in the reservoir shall be maintained at a constant level.

Note 1: It is therefore desirable tc have an automatic self-fill system and overfill alarm.

Note 2: It is recommended that equipment be fitted to prevent the growth of harmful bacteria. For example, the water could be made to circulate through a UV-irradiation unit (see figure 2) consisting of two concentric tubes. The outer tube is made of UPVA or stainless steel. The inner tube consists of a short-wavelength UV source of appropriate wattage in a quartz envelope to ensure maximum penetration of the UV light. The tubes are changed at 4000 h intervals or earlier if so specified by the manufacturer.

4.4 Specimen-loading tube

The specimen-loading tube shall have a tubular main body, which contains the specimens to be tested, and a spring assembly which can be compressed and locked in place to apply the desired load. The tubular body shall be perforated for ease of inspection of the specimens and to allow moisture to enter.

The spring assembly shall consist of a main spring and a secondary spring and is normally designed to apply loads from approximately 1 MPa to 7 MPa to the specimens. However, for some types of testing different load ranges may be appropriate.

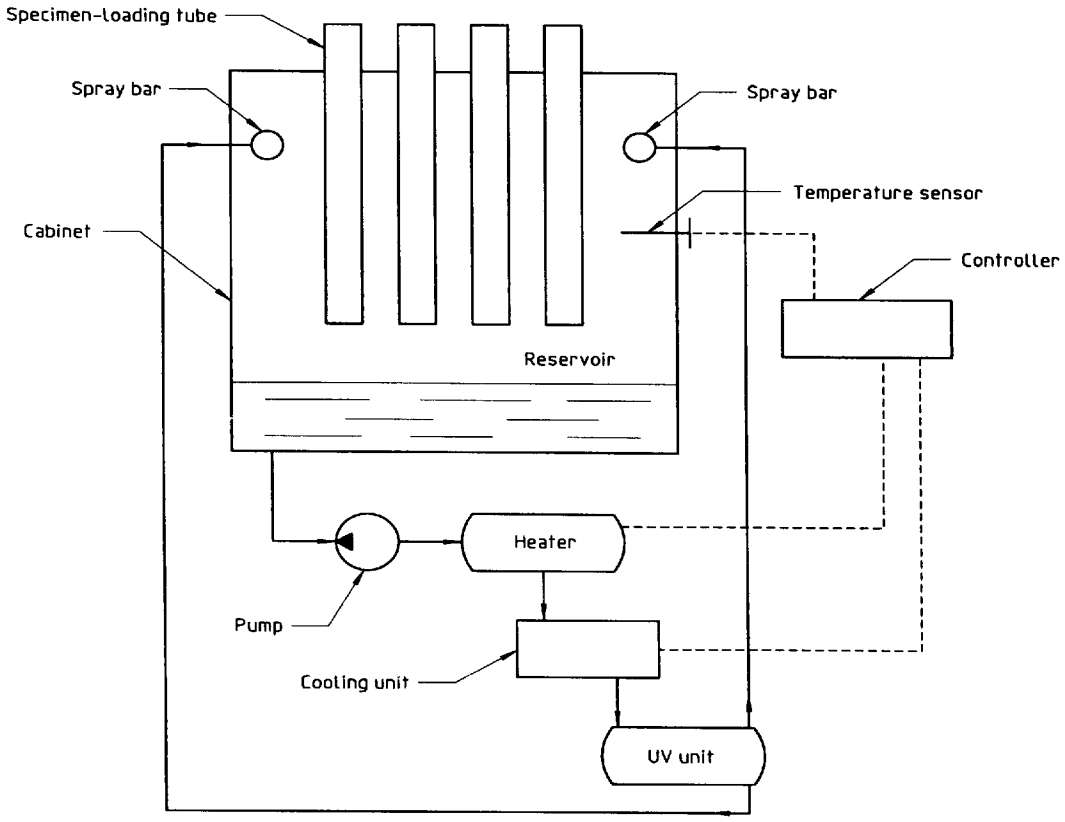

Figure 1 — Schematic diagram of apparatus

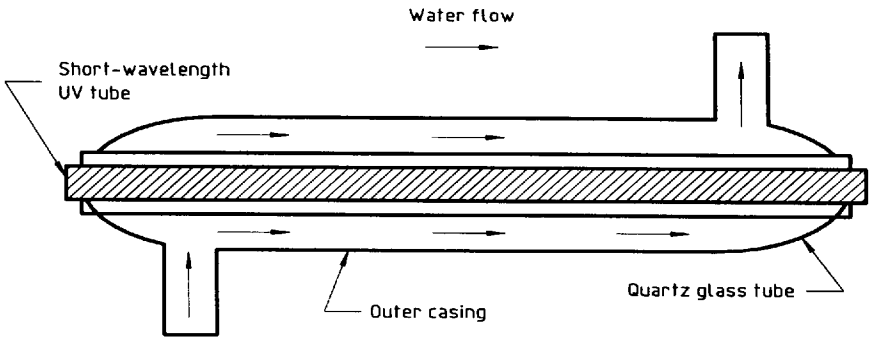

Figure 2 — Ultraviolet unit

A typical specimen-loading tube design[1)] is shown in figures 3 and 4. Aluminium or stainless steel is a convenient material for its construction.

Note 3: Load levels will need to be established for the particular system under test. They will generally lie in the range between 10% and 50% of the joint failure load.

1) One possible supplier is:
Stoke Golding Applied Research, 29 Billington Road West, Elmesthorpe, Leicestershire LE9 7SD, UK

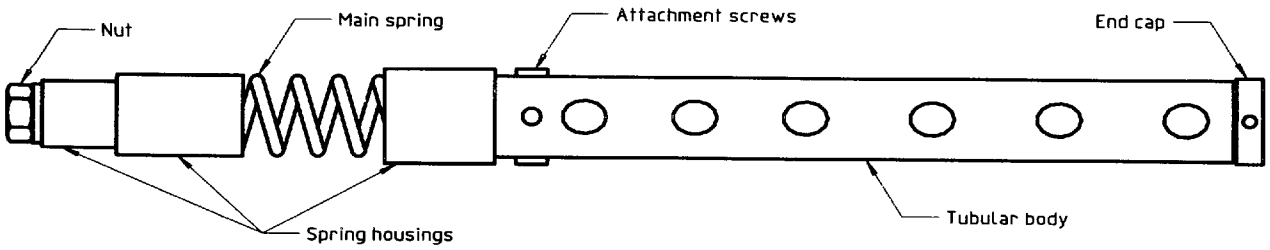

Figure 3 — Specimen-loading tube

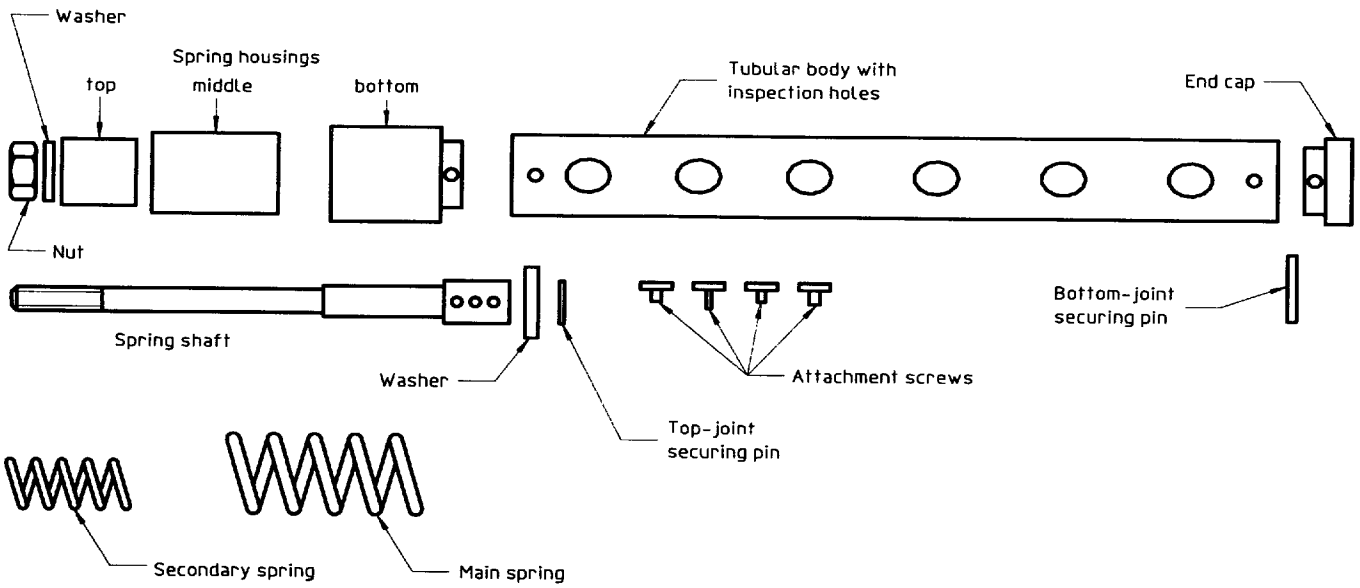

Figure 4 — Components of specimen-loading tube

The load is applied to a series of three to six specimens contained within the main body of the tube.

Care shall be taken to ensure that the thermal capacity of the tubes does not exceed the capacity of the cabinet to maintain the correct humidity and temperature cycle.

5 Test specimens

5.1 Specimens of the general dimensions shown in figures 5 and 6 shall be prepared individually and shall consist of two adherends prepared and bonded together in accordance with either the adherend- or adhesive manufacturer's instructions.

5.2 Any surface treatment used shall be such that consistent results are obtained with the bonded assembly. Any surface contaminant, e.g. oil or lubricant, being evaluated shall be applied to all specimens in a manner which ensures uniformity between specimens. The preparation of the adherend surfaces shall be in accordance with either the adherend- or the adhesive manufacturer's instructions or, in the case of metals, with ISO 4588 and, in the case of plastics, with ISO 13895.

Dimensions in millimetres

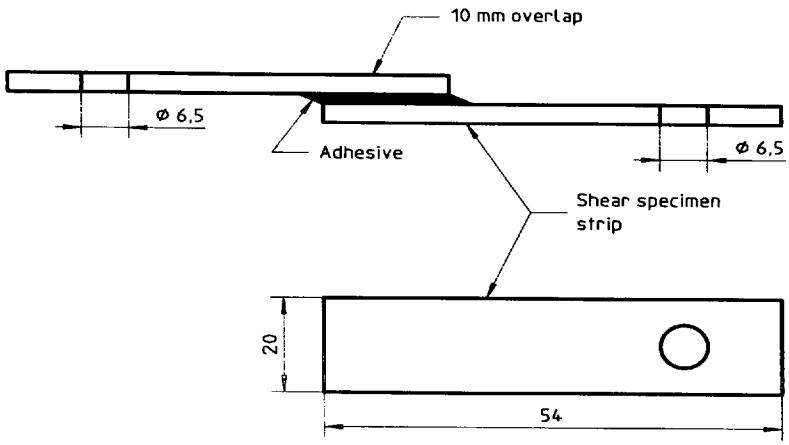

Figure 5 — Typical lap-shear joint

Dimensions in millimetres

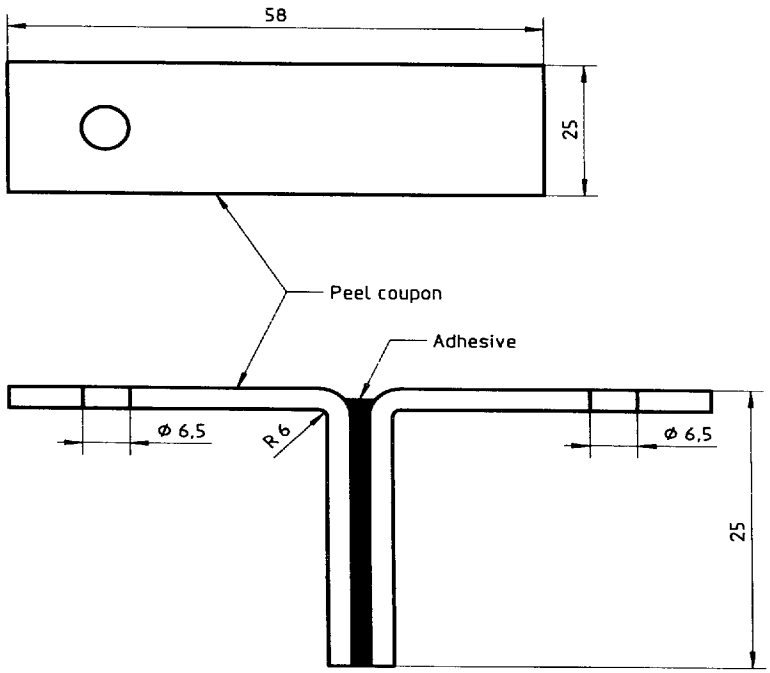

Figure 6 — Typical T-peel joint

The adhesive shall be applied in accordance with the manufacturer's instructions to obtain an optimum bond with minimum variation.

The test is most suitably carried out with lap-shear specimens of the type shown in figure 5. Simple T-joints of the type shown in figure 6 can also be used. In the case of the T-joint specimens, some modification of specimen-loading tube design will be required to allow the joints to lie centrally within the tube. The greater compliance of such joints also requires a more extensive spring system for loading.

In the case of lap-shear joints, it may sometimes be useful to drill holes through the centre of the joints after preparation of the specimens, in order to accelerate the test. Care shall be taken not to

damage the joints when the holes are drilled. The location, size and number of drill holes shall be either as specified or the person preparing the specimens shall ensure that drill hole location, size and number are identical for each system. In some cases, it may be desirable to drill the specimen before assembling the adherends.

The fillet area of a T-joint is particularly critical, and the shape and size of the fillet shall be checked. Consistency can be obtained by using a suitably shaped filleting tool which can be moved along the joint line after adherend assembly and before adhesive cure.

Bond thickness is usually a significant factor and shall be controlled by appropriate means such as glass spheres or spacer wires.

Note 4: Direct comparisons between different systems can be made only when specimen construction, adherend materials and test conditions are identical.

5.3 The adherends shall be chosen from materials representative of those used in the manufacturing industry, and their thickness shall normally lie within the range 0,6 mm to 3 mm.

5.4 Specimens shall be prepared individually. The lap-shear joint configuration shall be

a) either 20 mm wide with a 10 mm overlap (preferred)

or

b) any other convenient width and overlap, provided that the test equipment is suitably adapted and the values are given in the test report.

5.5 The number of specimens tested in each specimen-loading tube shall be between three and six. The number tested shall be recorded.

6 Procedure

6.1 Spring calibration

The main and secondary springs used in the specimen-loading tube shall be calibrated on a tensile-testing machine and individually numbered. A calibration chart shall be prepared for each main spring, relating the applied load to the distance separating the middle and bottom spring housings. It is preferable to maintain particular spring and tube combinations.

The secondary spring shall be chosen such that, when fully compressed in the top spring housing, it applies a load of 1 MPa.

6.2 Assembly

Assemble a series of at least three suitable specimens by bolting them together with stainless-steel or polyamide bolts. Use solid strips of material to complete the series if necessary.

Insert the series into the specimen-loading tube by securely pinning it first to the the bottom of the spring shaft. With the bottom spring housing and washer in position on the shaft, slide the specimens into the specimen-loading tube. Secure the bottom spring housing to the tube using the attachment screws, which prevent the specimens from twisting when they are under load. Place the end-cap over the end of the series of specimens and securely pin.

Locate the main spring over the shaft and then the middle spring housing. Place the secondary spring and its housing over the shaft and finally the washer and nut.

With the tube held firmly in a vice, turn the nut until the top spring housing is just tight. Using an interior calliper and a micrometer, take a reading of the spacing between the middle and bottom spring housings and record. Consult the spring calibration chart and, using the chart and the reading obtained, set the callipers for the spacing required at the desired load. Tighten the nut on the spring shaft until the calliper is just tight.

6.3 Introducing the tubes into the cabinet

Introduce the specimen-loading tubes through the holes in the top of the cabinet so that they hang vertically. Seal any unused holes in the top of the cabinet and any gaps between the tubes and the cabinet top.

6.4 Temperature cycle

Unless another temperature cycle is specified by the referring standard, selected for instance from those defined in ISO 9142, the cabinet temperature shall be continuously cycled between 42°C and 48°C, taking 60 min +/- 5 min to complete a full cycle from 42°C to 48°C and back to 42°C.

6.5 Testing

Record the date and time of the start of the test.

Continue the test until a joint fails and record the time to failure. Check the tubes daily, or at shorter periods if appropriate (the spring assembly will be loose when a joint has failed).

When a failure occurs, disassemble the tube and replace the failed specimen with a solid strip of appropriate length. Re-assemble the tube and place under load as before. Introduce the tube into the cabinet again and record the time to failure of the next joint. Continue this procedure until at least three of the joints in the series have failed.

Record the failure pattern in accordance with ISO 10365.

Record the load applied.

Note 5: Control specimens may be included in any comparative investigation, at the discretion of the individual user.

Note 6: The creep/relaxation history of the individual specimens will be different, owing to the replacement of the failed specimens and reloading of the remaining ones. However, since the test may last several weeks, the effect of relaxation is not significant.

7 Expression of results

Express the results as the time to failure, in days or hours, of the joints under a given load. Report the individual result for each specimen or the mean value of the at least three times to failure determined.

Note 6: It may be convenient to test specimens in a series of tubes under different loads and to plot a graph of failure time against applied load.

Note 7: The strengths of unaged controls will need to be known in order to choose appropriate loads. The test procedure described in this International Standard will be one of a number of tests being carried out, and this information will normally be available from other tests.

8 Precision

The precision of this test method is not known because inter-laboratory data are not available. When inter-laboratory data are obtained, a precision statement will be added at the following revision.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for complete identification of the adhesive tested, including type, source and manufacturer's code number;

c) all details necessary for complete identification of the adherends used, including the dimensions, the methods used to prepare the surfaces prior to bonding;

d) details of any conditioning of the adherends or bonded specimens;

e) the temperature cycle used;

f) the average thickness, to the nearest 0,02 mm, of the layer of adhesive in the bond, and the method of measurement used;

g) the width and overlap of the adherends in lap-shear specimens, and the width of the joint in T-joint specimens;

h) the number of specimens tested ;

i) the load applied;

j) the times to failure;

k) the failure patterns observed;

l) the dates over which the test took place.

ICS 83.180

Descriptors: plastics, adhesives, adhesive bonded joints, tests, adhesion tests, determination, durability, test equipment.

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