IS 12994 (1990): Epoxy adhesives, room temperature curing General purpose [PCD 12: Plastics]

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IS 12994 : 1990
(Superseding IS 8230 : 1976)
(Reaffirmed 2005)

भारतीय मानक

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Indian Standard

EPOXY ADHESIVES, ROOM TEMPERATURE CURING, GENERAL PURPOSE — SPECIFICATION

UDC 665·93 : 620·113 : 620·16

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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Price Group 4

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 5 March 1990, after the draft finalized by the Adhesives Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

IS 8230 : 1976 'Steel filled epoxy resin based adhesives' covering five grades was published. Lately this classification has been found arbitrary and overlapping with other close types. Furthermore, it is felt that steel filled epoxy resins are not so commonly used as adhesives but are rather in vogue for face casting of foundry patterns or filling blowholes. They are very rarely used for joining surfaces now.

Therefore, the Committee decided to prepare a separate specification for epoxy resins based adhesives in place of steel filled epoxy resin based adhesives.

Epoxy resin based adhesives are of the thermosetting type. They set by cross-linking with a curing agent or a hardener. Hardeners are added in a prescribed ratio to the resin just before application in two-component systems. Latent hardeners may be pre-mixed with the resin to give one-component systems. Two-component systems cure at room temperature or at elevated temperature, depending on the hardener used. One-component systems are invariably cured at elevated temperature. Joints cured at elevated temperature are resistant to higher temperature than those cured at room temperature.

Expoxy resin based adhesives because of their very high adhesive strength find application in bonding metals, glass, ceramics, concrete, wood and certain types of plastics. They are therefore widely employed in aircraft construction, automobile fabrication, glued rail joints, electronic assemblies and in other engineering industries, besides being used for repairs of different kinds.

Epoxy resin based adhesives may be modified with a wide variety of fillers such as finely divided chalk, talc, graphite or other minerals, metal powders or glass fibre to obtain certain desired properties. Filled adhesives are normally used when the gap between the adherends is wider than 200 μm. Metal powders improve thermal conductivity. Silver copper or graphite powders impart good electrical conductivity to the adhesive.

Considerable assistance has been derived from BS : 2782 : Part 8 : Method 835C : 1980 'Determination of gelation time of polyester and epoxide resins using a gel timer' and is prescribed in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

EPOXY ADHESIVES, ROOM TEMPERATURE CURING, GENERAL PURPOSE — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for liquid and paste type epoxy adhesives for performance: (a) up to 50°C, and (b) up to 100°C.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
3434 : 1984	Glossary of terms for adhesives and pressure sensitive adhesive tapes
4905 : 1968	Methods for random sampling
8230 : 1976	Steel filled epoxy resin based adhesives

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 3434 : 1984 shall apply.

4 TYPES

4.1 For certain applications where very thin glue lines are required, a liquid adhesive is preferred. It can be applied by brush or by special dispensing machines. Other applications involving thicker glue lines require a filled, paste-type adhesive, usually thixotropic in nature, which may be applied by spatula.

4.2 Most room-temperature curing adhesives are suitable for joints which are subjected to a maximum operating temperature of 50°C in-use. Specially formulated adhesives may be used in joints subjected to an operating temperature of 100°C.

4.3 There shall be four types of epoxy adhesives:

Type L 50 :	Liquid adhesives for performance up to 50°C.
Type P 50 :	Paste adhesives for performance up to 50°C.
Type L 100 :	Liquid adhesives for performance up to 100°C.
Type P 100 :	Paste adhesives for performance up to 100°C.

5 REQUIREMENTS

5.1 Properties of Uncured Components

5.1.1 *Solids Content*

Each component of the adhesive shall be free from volatile solvents, and the non-volatiles in each component shall be not less than 99·5 percent when tested in accordance with Annex A.

5.1.2 *Viscosity*

The viscosity of the individual components shall comply with the following requirements:

Type L 50 and L 100 :	10 000 mPa s *Max* when tested in accordance with **B-1.**
Type P 50 and P 100 :	1 000 000 mPa s *Max* when tested in accordance with **B-2.**

5.2 Properties of Uncured Mixture

5.2.1 *Gelation Time of Liquid Adhesives*

The gelation time of the mix of resin and hardener of Type L 50 and L 100 prepared in the ratio recommended by the manufacturer shall not be less than 30 minutes at a bath temperature of 27 ± 1°C, when tested in accordance with Annex C.

5.2.2 *Spreadability of Paste Adhesives*

A mass of 25 g of the adhesive of Type P 50 and P 100, when uniformly mixed with the prescribed hardener in the ratio recommended by the manufacturers, shall remain spreadable at a temperature of 27 ± 1°C for a minimum of 15 minutes.

5.2.3 *Sagging*

The uncured adhesive of Type P 50 and P 100, when uniformly mixed in the ratio recommended by the manufacturer, shall not run, drip or sag when tested in accordance with Annex D.

5.3 Properties of Cured Adhesive Joints

The tensile shear strength of joints prepared and tested in accordance with Annex E shall be not

less than the following minimum values, after curing the joints for 24 hours at 27 ± 1°C.

	Types L 50 and P 50	*Types L 100 and P 100*
At 27°C (N/mm²)	14	14
At 50°C (N/mm²)	10	14
At 100°C (N/mm²)	—	12

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in containers as agreed to between the purchaser and the supplier.

6.2 Marking

The containers shall be marked legibly with the following information:

a) Name and type of the material;

b) Indication of the source of manufacture;

c) Batch number in code or otherwise to enable the lot of supply to be traced back from records; and

d) Shelf life.

7 SAMPLING

7.1 General Requirements of Sampling

7.1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

7.1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

7.1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

7.1.4 The samples shall be placed in clean, dry air-tight glass or other suitable containers.

7.1.5 Each sample container shall be sealed with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material and its shelf life.

7.2 Scale of Sampling

7.2.1 *Lot*

All the containers in a single consignment of the material of the same type, same grade and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

7.2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, samples shall be tested from each lot separately.

7.2.2 The number of containers to be sampled from the lot shall depend upon the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

Table 1 Number of Containers to be Selected

Lot Size (1)	Sample Size (2)
Up to 150	3
151 to 300	4
301 to 500	5
501 to 1 000	6
1 001 to 3 000	7
3 001 and above	8

7.2.3 The containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 : 1968.

7.3 Samples and Referee Samples

7.3.1 Draw with an appropriate sampling instrument a small portion of the material. The total quantity of material drawn from a container shall be sufficient to make triplicate determinations for all the characteristics given in the specification.

7.3.1.1 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but approximately equal quantity of material shall be taken from each selected container and shall be well mixed up together so as to form a composite sample. The quantity of the material in the composite sample shall be sufficient for making triplicate determinations for all the characteristics to be tested on the composite sample. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee. These parts shall be transferred to clean, dry air-tight containers supplied by the manufacturer which are then sealed with all the particulars given in **7.1.4**.

7.3.1.2 The quantity of material remaining in the selected container (after a small quantity needed for composite sample has been taken out) shall be divided into three equal parts. These parts shall be transferred to clean, dry containers which are then sealed with all the particulars given in **7.1.4**. The sample in each such sealed container shall constitute an individual test sample. These individual samples shall be separated into three identical sets of test samples in such a way that

each set has a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the supplier and the third as the referee sample.

7.3.2 The referee samples shall consist of a set of individual samples and a composite sample. They shall bear seals of purchaser and the supplier so as to be used in case of a dispute between the two.

7.4 Number of Tests

7.4.1 Tests for determination of solid content, viscosity, gelation time and tensile shear strength shall be conducted on each of the individual samples separately.

7.4.2 Tests for the determination of sagging and spreadability of paste adhesives shall be conducted on the composite sample.

7.5 Criteria for Conformity

7.5.1 The lot shall be declared as conforming to the requirements of the relevant material specification, if the following are satisfied.

7.5.1.1 From the test results for each selected container, average ($\bar{X}$) and range (R) shall be computed for each of the characteristics to be tested on individual samples, as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{Difference in the largest and smallest test results}$$

If $\bar{X} - 0{\cdot}6R$ is greater than or equal to the minimum specification limit and $\bar{X} + 0{\cdot}6R$ is less than or equal to the given maximum specification limit, the lot shall be deemed to have satisfied these requirements.

7.5.1.2 All the test results on the composite sample shall meet the relevant requirements given in the individual material specification.

ANNEX A

(*Clause* 5.1.1)

DETERMINATION OF SOLIDS CONTENT

A-1 DETERMINATION OF SOLIDS CONTENT

A-1.1 Weigh out about 5·00 g of the material in a flat bottom weighing bottle with a lid. Keep the bottle with the lid open in an oven at 105°C for 60 minutes. Cool in a dessicator and re-weigh. From the decrease in weight, the non-volatile content is calculated as follows.

A-1.2 Calculation

$$\text{Residue on evaporation, percent by mass} = \frac{100\ M_2}{M_1}$$

where

M_1 = mass in g of sample taken, and

M_2 = mass in g of residue.

Express the result to the first decimal place.

ANNEX B

(*Clause* 5.1.2)

DETERMINATION OF VISCOSITY

B-1 DETERMINATION OF VISCOSITY

B-1.0 Outline of the Method

Viscosity of the solution may be determined by Hoeppler falling ball method or by Brookefield viscometer. In case, viscosity is determined by Brookefield viscometer, model LV using spindle 2 at a speed of 1.5 rev/min is recommended. Determination of viscosity by Hoeppler's method is as follows.

B-1.1 Determination of Viscosity by Hoeppler Falling Ball Method

B-1.2 Apparatus

The Hoeppler apparatus (*see* Fig. 1) consists of a number of tubes and balls, The choice of the ball and tube is made as follows.

B-1.2.1 For resins of viscosity 2 000 mPa or less, a ball of about 10 mm diameter is used. (Factor: about 25).

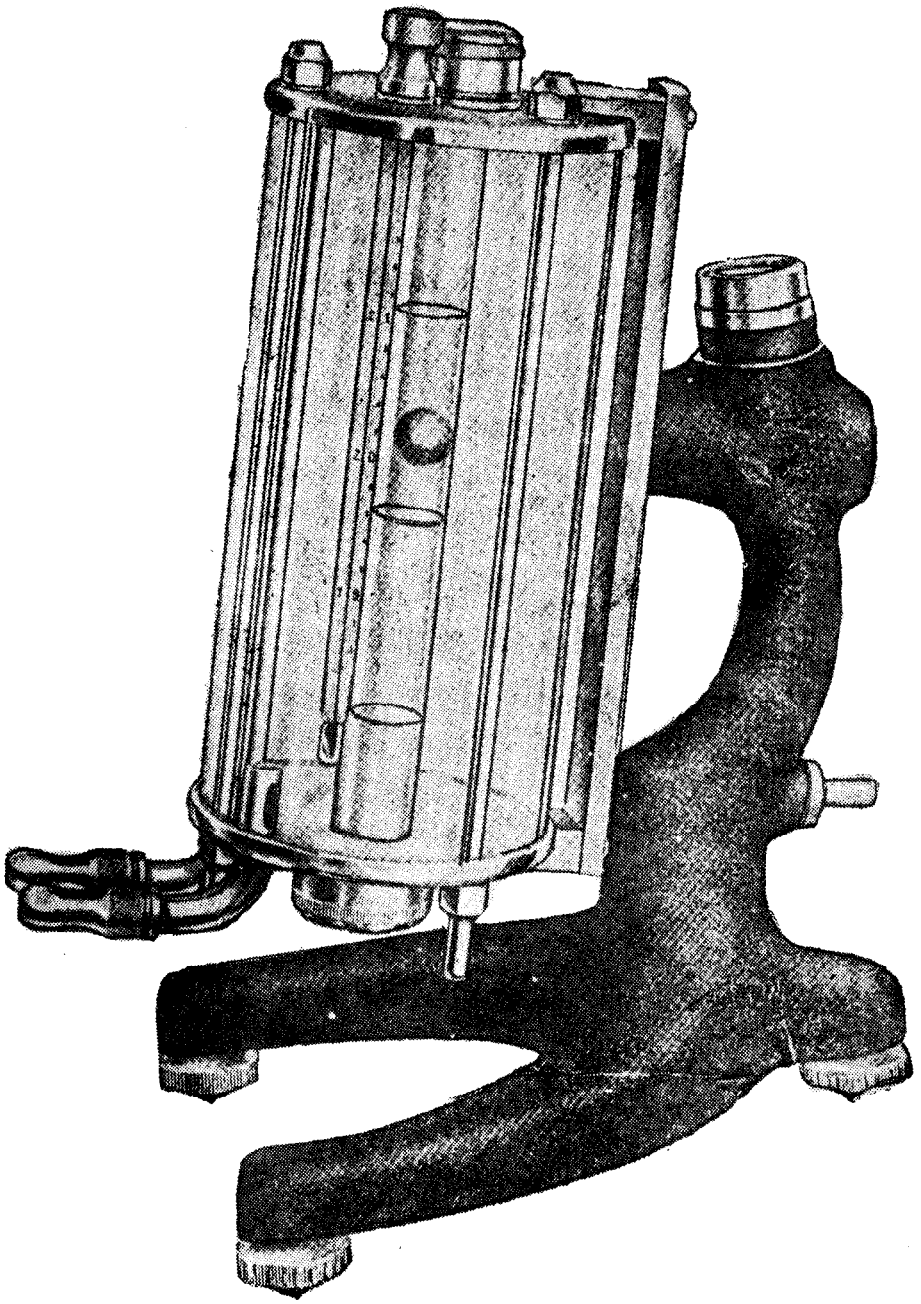


FIG. 1 HOEPPLER VISCOMETER

B-1.2.2 For resins of viscosity above 2 000 mPa, a ball of about 9 mm diameter is used (Factor : about 75).

B-1.3 Other Equipment Required

B-1.3.1 *Support for the Glass Tube*

B-1.3.2 *Stop Watch*

B-1.3.3 *Thermostatic Bath,* precision ± 0·1°C.

B-1.3.4 *Rubber Stopper*

B-1.4 Calibration of the Glass Tube

B-1.4.1 The glass tube is closed at one end with a rubber stopper and a liquid of known viscosity is filled in from the other end. When the liquid is free of air bubbles (very small air bubbles will not affect the results very much), a steel ball is introduced into the tube and the second rubber stopper inserted to close the tube completely.

The filled tube is kept into the thermostatic bath for about 10 minutes to allow the liquid in it to attain the required temperature. During this time, the tube is turned to an angle of 180° about 5 times. At the end of this period, the experiment may be commenced.

After turning the tube to an angle of 180°, the time taken for the ball to fall from one graduation mark to the other is measured accurately with a stop watch.

B-1.4.2 *Calculation*

If the viscosity of the known liquid was 20 000 mPa and the average time taken for the ball to fall is 190 seconds, the factor would be:

$$f = \frac{20\,000}{190} = 105{\cdot}2$$

B-1.5 Viscosity Measurement

B-1.5.1 The same experiment is repeated using the resin under test in place of the liquid of known viscosity. At least 5-6 measurements are made and the average taken.

B-1.5.2 *Calculation*

Viscosity in mPa $= f \times t$

where t is the time in seconds for the fall of the ball from one graduation mark to the other.

B-2 DETERMINATION OF VISCOSITY OF THIXOTROPIC PASTES WITH BROOKEFIELD VISCOMETER

B-2.0 Outline of the Method

Viscosity of thixotropic pastes with Brookefield viscometer and Helipath stand.

B-2.1 Apparatus

B-2.1.1 *Water Bath,* approximately 20 cm high edges — plastic.

B-2.1.2 *Recirculating Thermostat*

B-2.1.3 *Thermometer,* 0 — 5°C with 1/10° graduation.

B-2.1.4 *Brookefield Viscometer,* Type HBT with T Spindel.

B-2.1.5 *Helipath Stand*

B-2.2 Procedure

B-2.2.1 The paste is first tempered for 7 days that is the paste is left undisturbed in a beaker (500 to 1 000 g) in a water bath at 27 ± 0·1°C.

B-2.2.2 The specified *T Spindel* is connected to the Brookefield viscometer and with the *Helipath* stand so fixed that the cross arm of the spindel just touches the substance. Now the viscometer is run at the speed of 20 rev/min and the operation carefully watched. After the cross arm causes no further visible deformation of the liquid surface during the rotation (usually after 2 turns), the reading of the next 5 turns is noted.

B-2.2.3 The extreme values caused by air bubbles or agglomerations may be deleted. There must be at least 3 values within ±1° remaining otherwise the measurement is to be repeated.

B-2.2.4 From these 3 values, the mean is calculated and from that the viscosity of the given paste is calculated according to the instructions for the apparatus.

ANNEX C

(*Clause* 5.2.1)

DETERMINATION OF GELATION TIME OR POT LIFE

C-0 GENERAL

C-0.1 Gelation time is the time required for an epoxide resin system, maintained at the specified temperature to reach the gel stage in the process of hardening.

C-1 APPARATUS

C-1.1 Heating Bath

Heating bath should be capable of being maintained within ±0·2°C of the specified temperature.

C-1.2 Ancillary Containers

These containers should be of suitable shape and capacity for premixing the reagents.

C-1.3 Reciprocating Device/Gel Timer

The device consists of a stainless steel plunger rod, about 100 mm in length, 3 mm in diameter at one end of which is attached a stainless steel disc of 1·5 mm thickness and 22·1 to 22·3 mm diameter. The combined mass of the rod, disc, and connecting link shall be 16·2 + 0·02 g. The rod is connected, with its axis vertical, to a mechanical device driven by an electric motor so that the disc can be moved up and down in a vertical plane with an amplitude of 12·7 mm and at such a rate that the disc travels 12·7 mm in half minute so that one up and down movement, that is, one reciprocation, is completed in one minute (*see* Fig. 2).

C-1.4 An automatic timing device, for example, a system for counting the number of reciprocations may be used.

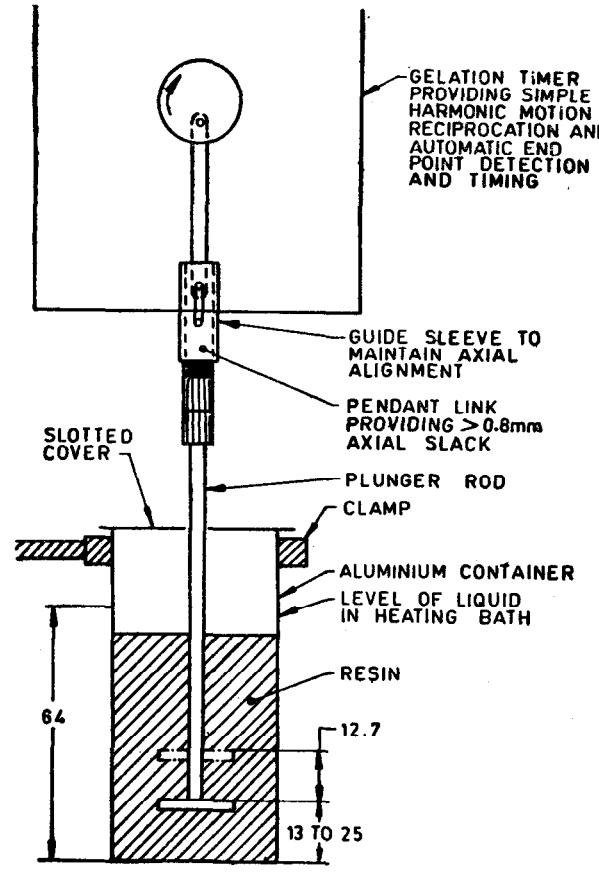
C-2 PROCEDURE

C-2.1 Assemble the apparatus in such a manner that the rod and disc may move vertically in the middle of the empty aluminium container when it is immersed in the bath to a depth of at least 64 mm.

C-2.2 Mix the ingredients of the resin system in the ancillary container in the proportions and in the manner prescribed by the supplier. The mass of the final blend shall be between 120 to 150 g. Start the motor as soon as the mixing is done to bring the contents to the test temperature. Stir the complete mixer vigorously (*see* Note below) for two minutes.

NOTE — Stirring should be sufficiently vigorous to obtain good mixing and temperature equilibration within the period specified but not so vigorous as to entertain air bubbles into the mixture as these could interfere significantly with the gelation period.

The temperature of the ingredients before and while mixing shall be such that the final temperature of the mixuture does not exceed the test temperature and is not more than 2°C below the test temperature for temperature up to 40°C, Weigh 100 ± 2 g of the mixture into the aluminium container quickly, which shall be immersed in the bath. Adjust the apparatus so that the disc is immersed in the resin during complete



All dimensions in millimetres.

FIG. 2 SUITABLE ARRANGEMENT FOR GEL TIMER

reciprocation and is 13 to 25 mm above the bottom of the container. Report the gelation time of the resin under test as the number of reciprocations recorded in the interval in minutes between the starting and stopping of the motor.

CAUTION — Epoxide resins give off epichlorhydrin when heated. The vapour is toxic and care should be taken not to inhale it.

C-3 REPORT

C-3.1 Report as follows:

a) The gelation (or gel) time of the material; and

b) The temperature at which the test was carried out.

ANNEX D

(*Clause* 5.2.3)

TEST METHOD FOR SAGGING

D-1 TEST METHOD FOR SAGGING

D-1.1 Prepare smooth aluminium panels 25 mm × 150 mm in size. The panels shall be clean, dry and free of all dust, grease and other foreign matter. To one side of the panel, apply a 1 mm thick coating of the ready-to-use uncured adhesive to a length of 100 mm. Hold the panel in a vertical position along the 150 mm axis. Any evidence of running, sagging or film deformation shall be a cause for rejection. Three panels shall be prepared and tested for this test.

ANNEX E

(*Clause* 5.3)

DETERMINATION OF SHEAR STRENGTH

E-1 GENERAL

E-1.1 This method covers the determination of the comparative shear strengths of adhesives for bonding metals when tested on a standard specimen under specified conditions.

E-1.2 Unless otherwise agreed by the manufacturer and the purchaser, the bonding conditions shall be prescribed by the manufacturer, which includes procedure for preparation of surfaces prior to application of the adhesives, complete blending directions, conditions of application, curing conditions and conditioning procedure before testing including the length of time, temperature and relative humidity.

E-2 APPARATUS

E-2.1 Tensile Testing Machine

Any suitable motor-driven, tensile testing machine may be used. The capacity shall be such that any reading taken during or on completion of the test shall fall within the loading range (loading range being the range within which the indicated load shown by calibration is correct within ±1·5 percent). The speed of the moving head of the tensile testing machine when running free shall be 250 ± 50 mm per minute. The machine should be equipped with a suitable device for maintaining the joints at 100°C during test.

E-3 NUMBER OF TEST SPECIMENS

E-3.1 Test shall be carried out on three test specimens.

E-4 PREPARATION OF TEST SPECIMENS

E-4.1 Metal to Metal Specimens

Each set of specimens shall consist of 2 strips of any high strength aluminium alloy measuring 150 × 25 × 1·5 mm ground on one side with a grinding belt or by hand with emery paper of 20 to 30 grit. The surfaces to be bonded shall thereafter be degreased with a wad soaked in trichloroethylene.

E-4.2 One-component adhesives may be applied directly on the end of the test strip. In the case of two-component adhesives, the resin and hardener shall be accurately weighed and well mixed by means of a glass rod or spatula prior to application on the test strip. Film adhesives are just sandwiched between the two strips. The thickness of the adhesive applied is usually 100 to 300 microns.

The two adherends are clamped together over the bonded area which should have 10 mm overlap. Pressure of approximately 25 kg is applied by means of clamps. Excessive pressure should be avoided to ensure that the joint is not starved of adhesive. The specimens bonded with a cold-setting adhesive shall be left undisturbed at 27 ± 2°C for a minimum period of 24 hours. Those bonded with a hot-setting adhesive shall be cured in a thermostatically controlled oven (sensitivity ± 2°C) in accordance with the curing schedule prescribed by the manufacturer (*see* Fig. 3).

E-5 CONDITIONING

E-5.1 Condition the test specimens for 24 hours at 27 ± 2°C and 65 ± 5 percent relative humidity.

E-6 PROCEDURE

E-6.1 Fix the two free ends of the test specimen in the two jaws of the testing machine which shall be 125 mm apart. For testing shear strength at elevated temparature, a special thermostatically controlled heating device shall be installed around the specimen in the machine. The specimen shall be kept for at least 30 minutes in this device to enable the adhesive to attain the required temperature.

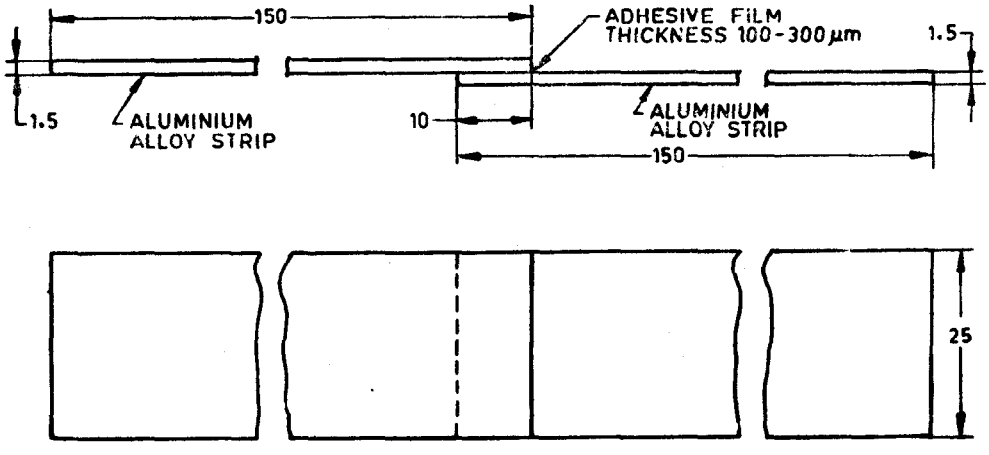
E-7 REPORT

E-7.1 Report the shear strength in N/mm², calculated from the load required to separate or break the joint and the area of the joint for each test specimen, and the mean of the three values.

E-7.2 The report shall also include the following information:

a) Complete identification of the adhesive tested; and

b) The nature of failure, whether it is failure in the cohesion of the adhesive, contact failure or adhesion to the metal.



All dimensions in millimetres.

FIG. 3 METAL TO METAL TEST SPECIMEN FOR SHEAR STRENGTH

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standards

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