Foreword

This Bangladesh Standard was adopted by the Bangladesh Standards and Testing Institution on ..... after the draft finalized by the Soap and Detergent Sectional Committee and approved by the Chemical Divisional Committee.

Epoxy resin is one of the most important resins used by paint industries in high performance paint application. It may be corrosion resistance or chemical resistance coating.

Due to its growing demand the sectional committee decided to formulate this standard. While revising this standard the sectional committee gave due consideration to the views of the producers, consumers and technologists and felt that it should be related to the prevailing trade and manufacturing practices followed in this field in the country.

In the preparation of this standard, assistance derived from the following publications is acknowledged with thanks:

IS 14925:2001 Epoxy Resin for Paints - Specification; Bureau of Indian Standards.

IS : 354 (Part 1) - 1987 (Reaffirmed 2002) Methods of Sampling and Test for Resins for Paints Part 1 General Test Methods; Bureau of Indian Standards.

IS : 354 (Part 4) - 1986 (Reaffirmed 2012) Methods of Sampling and Test for Resins for Paints Part 4 Special Test Methods for Epoxy Resins

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 BDS 103.

Bangladesh Standard

Specification for Epoxy Resin for Paints

1. Scope

1.1 This standard prescribes the requirement and methods of sampling and test for epoxy resin used in paint industry.

2. Normative references

2.1 The following Bangladesh Standards are necessary adjuncts to this standard. For undated references the latest edition of the publication referred to applies.

BDS 103 Methods of rounding off numerical values.

BDS 356 Acetic acid glacial

BDS 392 Test sieves

BDS 1701 Glossary of terms for paints

BDS 1765 Methods for Random Sampling

BDS 833 Water for laboratory use.

3. Terminology

3.1 For the purpose of this standard definitions given in BDS 1701 and the following shall apply.

3.2 Ambient Temperature

It is a temperature between 21°C and 38°C.

3.3 Epoxide Equivalent

The mass of an epoxide compound, in grams, which contains one mole of epoxide group.

4. Classification

4.1 A condensation product of epichlorohydrin and bispheneol A in alkaline condition, which leads to formation of epoxy group containing resins. These resins shall have different molecular weight or epoxy equivalent weight depending on stochiometric ratios of above mentioned reactants. The manufacturer shall declare molecular distribution and average molecular weight.

4.2 Depending upon the applications in paint system epoxy resins can be classified into three types based on average molecular weight or epoxy equivalent weight when determined through the method specified in Annex A, along with physical state of resin.

Type	Grade	Physical State	Average Molecular Weight	Epoxy Equivalent Weight
A	-	Liquid	360-380	180-200
B	Grade 1	Low Molecular weight, Solid	900	425-550
	Grade 2		1400	850-1000
C	Grade 1	High Molecular weight, Solid	2900	1700-2300
	Grade 2		3750	2400-3500

5. Requirements

5.1 Epoxy resin shall comply with the requirements specified in Table 1.

Table 1 Requirement for Epoxy Resins

(Clauses 5.1 and 5.2)

Sl. No.	Characteristics	Type A	Type B		Type C		Method of Test
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i.	Colour (on 'Gardner' scale), Max.	3	4	4	4	6	A
ii.	Viscosity a) As such at 25°C ± 0.05°C (Cps) b) 40 percent in butyl carbitol at 25°C ± 0.05°C (Cps) c) 50 Percent in 1:1 mixture of O. Xylene/D.A.A. at 25°C ± 0.05°C (Cps)	 8000-15000 - -	 - 80-170 100-150	 - 470-750 -	 - 1600-3400 -	 - 4000-11000 -	B
iii.	Softening point (ball and ring), °C	-	60-70	90-100	117-127	130-145	C
iv.	Epoxide equivalent, g/mole	180-200	425-550	850-1000	1700-2300	2400-3500	D
v.	Hydrolyzable chlorine content by mass, Max.	0.5	0.2	0.2	0.2	0.2	E
vi.	Relaive density (at 27 ± 2°C)	1.15-1.20	-	-	-	-	F

5.2 Keeping Quality

The material when stored in normal storage condition at the ambient temperature shall retain its property for atleast 12 months from the date of manufacture as prescribed in Table 1.

6. Form of Supply

6.1 Epoxy resin shall supply as 100 percent solids or any other percentage solution in any suitable solvent as agreed to between the manufacturer and the purchaser.

7. Packing and Marking

7.1 Packing

The material shall be packed in sound, clean and dry containers as agreed to between the manufacturer and the purchaser.

7.2 Marking

The containers shall be marked with the following information:

a) Name and type of the material;

b) Type and grade;

c) Mass of the material;

d) Month and year of manufacturer;

e) Batch No. or lot No. in code or otherwise; and

f) Name of the manufacturer and/or any recognized trade-mark.

g) Any other information required by statutory authorities.

7.2.1 The product(s) may also be marked with the BSTI Certification mark.

NOTE – The use of the BSTI Certification Mark is governed by the provisions of the Bangladesh Standards and Testing Institution Act 2018 and the Rules and Regulations made thereunder. Details of the conditions under which license for the use of the BSTI Certification Mark may be granted to manufacturers or Processors may be obtained from the Bangladesh Standards and Testing Institution.

8. Quality of Reagents

8.1 Unless specified otherwise pure chemicals and distilled water (see BDS 833) shall be used.

NOTE— 'Pure chemicals' shall mean chemical that do not contain impurity which affect the results of the analysis.

9. Sampling

9.1 Representative samples of the material shall be drawn as prescribed in annex G.

Annex A

[Clause 5.1 and Table 1, Sl. No. (i)]

Colour Test (Gardner Scale Method)

A-1 Apparatus

A-1.1 Gardner colour standards

a) Reference standards - Reference standards are glass colour standards having chromaticity coordinates and luminous transmittances as specified in Table 2. Eighteen glass colour standards are required. The glass colour standards shall be checked by the method given below:

Select a dual beam spectrophotometer with a sufficiently small light beam at the sample position so that all rays will pass through the standard to be calibrated. Alternatively equip the spectrophotometer with a condensing lens to achieve this. Place the standards in turn in the sample position of the spectrophotometer. If the comparator is provided with a separate green filter in front of the light source, place this filter in the reference beam of the dual beam spectrophotometer during calibration of each standard. Obtain spectral transmittance data for each glass reference standard. From the spectral transmittance data for each reference standard, calculate the CIE tristimulus values, X, Y, Z, and the chromaticity coordinates x and y for CIE illuminant C.

b) Working standards - Working standards are 18 glass or liquid colour standards having chromaticity coordinates that differ from those of the reference standards by not more than one-third of the difference in x or y (see Table 2) between adjacent reference standards. In any one set, no two standards shall be closer together than two-thirds of the difference in x or y between corresponding reference standards. The luminous transmittances shall be as specified in Table 2. The liquid working standards are freshly prepared coloured solutions contained in glass test tubes. Potassium chloroplatinate solutions are used for the lighter standards (1 to 8), and solutions of ferric chloride and cobalt chloride in hydrochloric acid are used for the darker standards (9 to 18).

c) The compositions of the liquid colour standards prepared are as follows:

i. **Gardner colour standards 1 to 8 -** into each of a series of one-mark volumetric flasks of the capacities indicated in Table 3, transfer from a burette the corresponding volumes of potassium hexachloroplatinate solution (see Note 4) as shown in Table 3, dilute each to the mark with the hydrochloric acid solution (see Note 3) and mix well.

ii. **Gardner colour standards 9 to 18 -** Into a series of 100-mL one-mark volumetric flasks, transfer from burettes the volumes of the ferric chloride solution (see Note 5) and cobalt chloride solution (see Note 6) as shown in Table 4. Dilute each to the mark with the hydrochloric acid solution (see Note 3) and mix well.

NOTE 1 - In case of dispute, only glass reference standards shall be used.

NOTE 2 - The chromaticity coordinates of the glass standards and those of the liquid standards differ slightly. However, the difference does not affect the accuracy of the estimation.

NOTE 3 - Hydrochloric acid (1:17) solution. Mix one volume of concentrated hydrochloric acid (specific gravity approximately 1.19 g/mL) with 17 volumes of water.

NOTE 4 - Potassium hexachloroplatinate solutions. Dissolve 790 mg of potassium hexachloroplatinate (K_2PtCl_6) in the hydrochloric acid solution (see Note 3) in a 100-mL one-mark volumetric flask. Warm the solution until all the potassium hexachloroplatinate is dissolved. Cool to 20°C room temperature, dilute to the mark with the same hydrochloric acid solution and mix well.

NOTE 5 - Cobalt chloride solution. Dissolve 40 g of cobalt chloride hexahydrate ($CoCl_2.6H_2O$) in 120 g of the hydrochloric acid solution (see Note 3).

NOTE 6 - **Ferric chloride solution.** Dissolve 1000 g of ferric chloride hexahydrate ($FeCl_3.6H_2O$) in 240 g of hydrochloric acid solution (see Note 1). Heat gently if necessary. Adjust the concentration so that the solution has exactly the same colour as a freshly prepared 30 g/L solution of potassium dichromate in concentrated sulphuric acid (specific gravity approximately 1.84 g/mL).

A-1.2 Glass test tubes – clear, colourless, round, of inside diameter 10.65 ± 0.025 mm, outside diameter about 12.5 mm and outside length about 114 mm.

NOTE - Test tubes that approximate to the stated diameter and that are, preferably, not less than 10 mm or not more than 11 mm inside diameter may be used. In such cases, the results should be multiplied by a correction factor equal to I10.65/d, where d is the inside diameter of the test tube.

A-1.3 Colour comparator - constructed to illuminate uniformly, and to permit simultaneously visual comparison of light transmitted through two colour standards and through a test tube in a transverse direction.

The apparatus may be of any design but should have the following characteristics:

a) **Illumination -** CIE Illuminant C.

b) **Surrounding field -** The field should not differ significantly in brightness from the samples and standards and shall be essentially achromatic.

c) **Field of view -** Two standards and a sample shall always be in the field of view.

d) **Arrangement of standard and sample -** There shall be a perceptible gap between sample and standard, but this shall be as small as possible.

A-2.2 Procedure - Fill a clean test tube (A-1.2) to a height of at least 70 mm with the sample, passing it through a sintered glass filter if there is any visual turbidity. Place the sample tube in the sample compartment of the comparator (A-1.3). Switch on the light source and compare simultaneously the colour of the sample with the colours of two adjacent standards, at a viewing distance between 30 and 50 cm. Determine which standard most closely matches the sample in brightness and saturation, ignoring any differences of hue.

A-2.3 Expression of Results

A-3.1 Reporting - Report the number of the standard most closely matching the colour of the sample. If more precise measurements are needed, report as either lighter than, matching or darker than the standard and expressed as a Gardner Colour number and also state the type of standard used (that is), glass colour standard or liquid colour standard.

Table 2 Colour Specifications of Reference Standards

Sl. No.	Gardner Colour Standard Number	Chromaticity Coordinate		Luminous Transmittance Y, Percent	Transmittance Tolerance (±)
		X	Y		
(1)	(2)	(3)	(4)	(5)	(6)
1	1	0.3177	0.3303	80	7
2	2	0.3233	0.3352	79	7
3	3	0.3329	0.3452	76	6
4	4	0.3437	0.3644	75	5
5	5	0.3558	0.3840	74	4
6	6	0.3767	0.4061	71	4
7	7	0.4044	0.4352	67	4
8	8	0.4207	0.4498	64	4
9	9	0.4343	0.4640	61	4
10	10	0.4503	0.4760	57	4
11	11	0.4842	0.4818	45	4
12	12	0.5077	0.4638	36	5
13	13	0.5392	0.4458	30	6
14	14	0.5646	0.4270	22	6
15	15	0.5857	0.4089	16	2
16	16	0.6047	0.3921	11	1
17	17	0.6290	0.3701	6	1
18	18	0.6477	0.3521	4	1

Table 3 Composition of Potassium Hexachloroplatinate Solutions

(Gardner Colour Standards 1 to 8)

Gardner Colour Standard Number	Potassium Hexachloroplatinate Solution, mL	Nominal Capacity of the One-Mark Volumetric Flask, mL
(1)	(2)	(3)
1	3.48	50
2	5.47	50
3	8.42	50
4	6.58	25
5	9.60	25
6	5.36	10
7	8.10	10
8	10.00	10

Table 4 Composition of Iron and Cobalt Solutions

(Gardner Colour Standards 9 to 18)

Gardner Colour Standard Number	Ferric Chloride Solution mL	Cobalt (Ii) Chloride Solution mL	Hydrochloric Acids Solution mL
(1)	(2)	(3)	(4)
9	3.8	3.0	93.2
10	5.1	3.6	91.3
11	7.5	5.3	87.2
12	10.8	7.6	81.6
13	16.6	10.0	73.4
14	22.2	13.3	64.5
15	29.4	17.6	53.0
16	37.8	22.8	39.4
17	51.3	25.6	23.1
18	100.0	0.0	0.0

Annex B

[Clause 4.2 and Table 1, Sl. No. (iv)]

Determination of Viscosity

B-0 Outline of the Method - Viscosity of resin solutions may be determined by (a) bubble tube method, or (b) Brookfield viscometer

B-0.1 In bubble tube method, the resin is dissolved in solvent and the bubble seconds are determined. Brookfield method measures viscosity in terms of Poises.

B-1 Bubble Tube Method

B-1.1 Apparatus

B-1.1.1 Bath - a constant temperature bath.

B-1.1.2 Holder for viscosity tubes - Preferably a mechanical holder capable of holding the tube in exact vertical position after being placed in constant temperature bath.

B-1.1.3 Mechanical shaker

B-1.1.4 Timing device - Suitable timing device like stop-watch capable of reading to a precision of 0.1 second.

B-1.1.5 Viscosity tubes - of clean glass and flat bottom, having 10.650 ± 0.025 mm inside diameter and 114 ± 1 mm length and having circular markings at 27.0 ± 0.5 mm, 100.0 ± 0.5 mm and 108.0 ± 0.5 mm from the bottom outside the tube.

B-1.1.6 When kinematic viscosity is to be determined directly, viscosity tubes of the following dimensions and details shall be used:

Flat bottom, open mouth glass tube having inside diameter of 11.50 ± 0.25 mm and length 101.50 ± 1.25 mm. It is fitted with a cork so that the distance from the bottom of the cork to the bottom of the tube is 90.0 ± 1.0 mm. The tube is to be filled with the material under test up to a height so that the air space left will give a bubble of 15.0 ± 1.0 mm length when it will ascend through the material.

B-1.2 Reagent

B-1.2.1 Solvent - as agreed to between the purchaser and the supplier,

B-1.3 Procedure

B-1.3.1 Preparation of resin solutions - The procedure employed for dissolving solid resins will depend upon the chemical nature of resin. The solution may be obtained either by cold cut or hot cut method as agreed to between the purchaser and the supplier. The two procedures should not be interchanged:

a) Cold-cut method - Weigh a 250-mL screw cap bottle with a cellulose film (100 x 100 mm) to nearest 0.05 g. Weigh into the bottle the required amount of solvent (40 g for a 60 percent solution). Weigh in a beaker the required amount of resin (60 g for 60 percent solution). Transfer the resin slowly into the bottle containing solvent, swirl gently to wet. Place the cellulose film over the mouth of the bottle and screw cap tightly. Shake the flask in the shaker overnight. Check the mass of the bottle and solution at the completion of shaking. If the loss is appreciable, discard solution and prepare fresh one.

b) Hot-cut method - Weigh accurately a long necked 250-mL flask with ground stopper. Weigh into the flask the required amount of solvent and resin. Connect the flask to a suitable air or water condenser and gently warm over a suitable oil or liquid paraffin bath with occasional swirling to prevent any charring. Usually the solution will be complete in about 15 minutes. When solution is complete, cool the flask, disconnect and weigh. If the loss of solvent is over 0.1 g, add the necessary amount of solvent to compensate loss, swirl and mix.

NOTE - If any air bubbles are present, allow the solution to stand until they disappear.

B-1.3.2 The resins supplied as solutions may be employed as such for determination of viscosity. However, if dilution is necessary, such as reduction to a specified solids content, use a suitable solvent as agreed to between the purchaser and the supplier.

B-1.3.3 Place the flask containing the resin solution in the bath at 27 ± 2°C. Fill a viscosity tube with the solution exactly to 100 mm mark, taking care to avoid any of the sample on the tube wall above 108-mm mark. Insert a new cork stopper so that the bottom of the cork is level with the 108-mm mark. This will give a bubble of uniform size and reproducible pressure. Insert the tube in the holder with stoppered-end down, and place the assembly in the bath at 27 ± 2°C for about 10 to 15 minutes to attain temperature equilibrium. With the assembly still in the bath, push the holder to return the tube to upright position and start the time device when the top of the bubble becomes tangent with the 100 mm line. Record the time when the bubble again becomes tangent to 27-mm mark to nearest 0.1 second. Record at least three determinations which agree within 1.0 second. Report the average of these values as result.

B-1.3.4 Report - Report the 73-mm bubble travel time in seconds as viscosity at 27 ± 2°C, and report solution concentration, solvent used and method of making the solution

NOTE - When kinematic viscosity is required, it shall be obtained by multiplying time obtained in seconds using a tube described in 8.1.1.6 by a factor 1.2.

B-2 Brookfield Viscometer Method

B-2.0 General - Resistance offered to the flow of a liquid is quantified into a factor called viscosity. Viscosity of a solution is directly related to molecular weight, percentage solid content

in solution, nature of solvent, temperature of measurement, nature of polymer and method of determination. The absolute unit of viscosity is poise. Occasionally, it is also referred in the units of kinematic viscosity, namely, stokes or centistokes.

$$\frac{\text{Viscosity in poise}}{\text{Density}} = \text{Viscosity in stokes}$$

Viscosities are also expressed in units known as Pa.s.

1 Pa.s. = 10 Ps = 1000 cPs.

B-2.0.1 Absolute viscosity of liquid materials, solutions both clear and opaque can be measured by this method. Kinematic viscosities obtained by this method are more direct and accurate for Newtonian type of fluids.

B-2.1 Apparatus

B-2.1.1 Brookfield viscometer - The Brookfield viscometer - Model RVF or suitable other models as agreed to between the purchaser and the supplier. For viscosities between 0 to 2000000 cPs as maximum range and 0 to 500 cPs as minimum range. An eight-speed RVF model known as RVT, has maximum and minimum ranges of 0 to 8000000 cPs and 0 to 100 CPS.

NOTE - Brookfield viscometer is available from Brookfield Engineering Laboratories Inc., Stoughton, Rassachuseths, USA 02072.

B-2.1.2 Temperature controlled bath with a variance limit as ± 0.5°C using either oil or water.

B-2.1.3 Thermometer - 110°C with 0.5°C division.

B-2.1.4 Glass beaker - 600 mL of 104 mm diameter. .

B-2.1.5 Glass rod - useful to stir the material in beaker.

B-2.2 Preparing the Sample

B-2.2.1 Fill the 600 mL beaker to 3/4th its height, with the test sample and keep the same in the constant temperature bath maintained at 27 ± 0.2°C, so that the test solution layer is below the layer of liquid in the bath. Carefully stir the test material and check its temperature with the thermometer (stirring should not introduce air bubbles in the medium). When the temperature of the test material has reached the desired value, leave it in the bath without stirring for 15 minutes to allow normalization. Select a suitable spindle for measuring the viscosity, and bring its temperature along with the spindle guard to the measurement temperature. Fix the spindle with the guard to the instrument. Remove the stirring glass rod and thermometer from the material in the beaker.

B-2.3 Procedure

B-2.3.1 Place the 600 mL beaker with test solution under the viscometer and lower the instrument such that the spindle dips up to the mark given on the spindle for the purpose. Take care to avoid air bubble sticking to the spindle.

B-2.3.2 Select a proper speed of the motor such that the reading is always in the middle of the dial range. Setting the speed, start the motor and allow the rotation to continue till the needle remains steady on the dial, note the value. After the reading, allow the spindle to rotate for two more cycles and check if the readings obtained correspond with the former. If not, repeat the

rotations checking till two successive constant readings are obtained. It is advisable to confirm the readings with two more speeds, one chosen above and another below the one used for the above determination. Note all the three values.

B-2.3.3 Convert the readings to viscosity values in centipoise (Pascal seconds) with the aid of conversion table provided with the instrument. The three values obtained as above should not differ at best by more than 1.0 cPs or 1 percent of the full scale.

The value obtaind may also be confirmed with other spindles for which speeds are chosen such that the dial readings are in the middle of the scale.

B-2.4 Report

B-2.4.1 A report on viscosity shall include the following:

a) Viscosity in centipoise;

b) Speed of rotation;

c) Spindle number;

d) Temperature of the sample to the nearest 0.5°C;

e) Method of sample preparation;

f) Model of Brookfield; and

g) In case of solution, the percentage solid and the solvent.

Annex C

[Clause 4.2 and Table 1, Sl. No. (iv)]

Determination of Softening Point (Ring and Ball Method)

C-1.0 Outline of the Method - The sample is enclosed in a horizontal ring and supports a steel ball. The temperature at which the sample sags or flows through a specified distance is the softening temperature.

C-1.1 Apparatus - Shall be as follows (see Fig. 1 and 2).

C-1.1.1 Ball - of steel, 9.50 mm in diameter and weighing 3.50 ± 0.05 g.

C-1.1.2 Ring - tapered ring as shown in Fig. 10 and of following dimensions:

Depth	6.35 ± 0.10 mm
Inside diameter at bottom	15.88 ± 0.10 mm
Inside diameter at top	17.46 ± 0.10 mm
Outside diameter	20.60 ± 0.10 mm

For convenience of mounting the ring in a support of the type shown in Fig. 1 and 2, the outside diameter of the ring at the bottom may be smaller, but shall not be less than 18.0 mm.

C-1.1.3 Ball guide - a convenient form of ball centering guide as shown in Fig. 2.

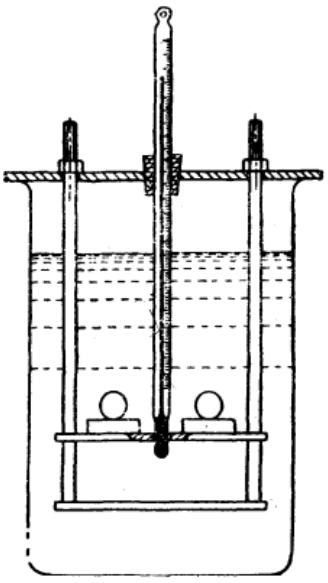

Fig. 1 Assembly of Apparatus for Determination of
Softening Point (Ring and Ball - Two Rings)

C-1.1.4 Support - a suitable one which fulfils the following conditions:

a) Rings shall be supported in a horizontal position with the upper surface of the rings 5.0 cm below the surface of the bath liquid;

b) There shall be a distance of exactly 2.5 cm between the bottom of the rings and the surface of the bottom plate of the support or bottom of the bath; and

c) The thermometers shall be suspended so that the bottom of the bulb is level with the bottom of the rings, and within 1 cm of the rings, but not touching them.

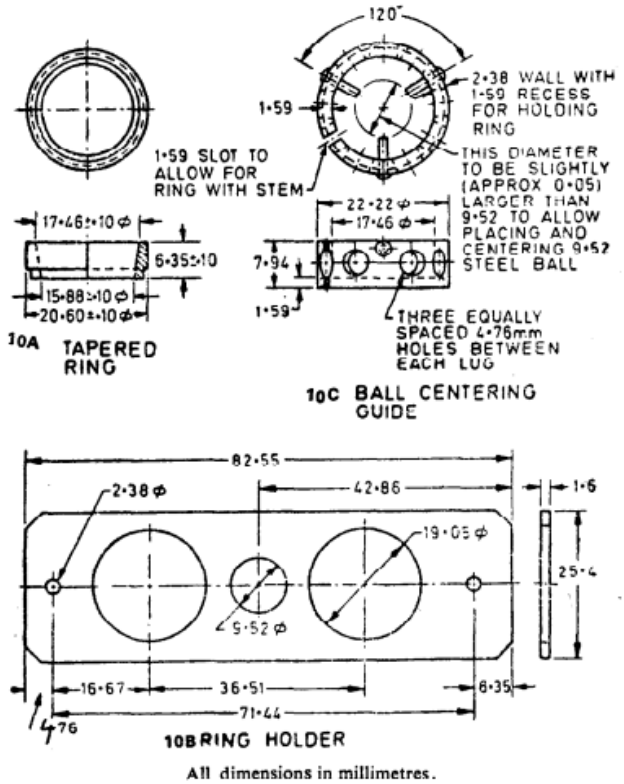

Fig. 2 Components of Ring and Ball Apparatus

C-1.1.5 Thermometer - of range -2°C to + 160°C with 0.5°C graduations.

C-1.1.6 Bath - made of glass and of suitable size. The bath liquid shall be water for materials having softening point below 80°C and glycerine for materials having softening point above 80°C. But both the liquids shall not be a solvent for the material under test.

C-1.1.7 Stirrer - mechanical.

C-1.2 Procedure

C-1.2.1 Preparation of test specimen - The material shall be prepared as follows by either of the methods:

a) Pour method - Melt the sample just above its softening point. Stir thoroughly avoiding trapping of air bubbles. Place ring on a brass plate, amalgamated to prevent adherence of sample. Pour enough sample into ring to provide an excess when cool. After cooling for about 30 minutes, remove excess sample with knife, slightly heated. Proceed with test as in C-1.2.2 within 4 hours.

b) Powder method - Break sample into pieces not larger than 3.0 mm. Mix thoroughly and quarter down to a portion of 50 to 75 g. Grind to powder and separate the portion passing through 300-micron BDS Sieve and retained on 75-micron BDS Sieve. Assemble mortar and add powdered resin to about half-full sieve. Insert pestle and compact powder by striking pestle with a mallet. Remove ring and scrape off excess resin from top. If top and bottom surfaces are not smooth and flush with ring, discard sample and repeat preparation with a clean ring and fresh powder.

C-1.2.2 Two procedures are prescribed depending upon the softening point of the resin. Tests shall be carried out in duplicate.

a) **For materials of softening point above 80°C -** Assemble the apparatus and fill the bath to a height of 5 cm above the upper surface of the rings with glycerine at a temperature of 35°C. Maintain the bath at 35°C for 15 minutes, after which place a ball previously raised to 35°C in each ball guide by means of forceps. Apply heat to the bath and stir the liquid so that the temperature rises at uniform rate of 5 ± 0.5°C per minute until the material softens and allows the ball to pass through the ring. The rate of temperature-rise shall not be averaged over the period of test and any test in which the rate of temperature rise does not fall within the specified limits after the first three minutes, shall be rejected. Record, for each ring and ball, the temperature shown by the thermometer at the instant the material surrounding the ball touches the bottom plate of the support or the bottom of the bath. Repeat the test if the difference between duplicate determinations exceeds 1 °C.

b) **For materials of softening point below 80°C -** Follow the procedure prescribed in C-1.2.2 (a) except that freshly boiled distilled water is used in the bath in place of glycerine and starting temperature of test is 5°C instead of 35°C.

C-1.3 Report - Report, to the nearest 0.5°C, the mean of the temperatures recorded in duplicate determinations as softening point.

Annex D

[Clause 4.2 and Table 1, Sl. No. (iv)]

Determination of Epoxide Equivalent of Epoxy Compounds

D-1 Principle

D-1.1 Method A

Epoxide groups react with nascent hydrogen bromide produced by the action of a standard 0.1 mol/1solution of perchioric acid on tetraethylammonium bromide. The end-point is determined either by using crystal violet as indicator or, for dark-coloured products, by a potentiometric method. This method is recommended for normal reactive epoxy resin.

D-1.2 Method B

The amino nitrogen of the epoxyamine is titrated with a standard solution of perchloric acid. The value thus obtained is used as a correction in the calculation of the epoxide equivalent as obtained in Method A. This method is recommended for slow reactive epoxy resin.

NOTE — Safety goggles and safety screen may be used while carrying out tests through the above methods.

D-2 Reagents

D-2.1 Acetic Acid, Glacial — Conforming to BDS 356.

D-2.2 Acetic Anhydride

D-2.3 Suitable Solvent for Solution of the Sample — for example chloroform, dibutyl phthalate, chlorobenzene.

D-2.4 Potassium Hydrogen Phthalate — Dry the potassium hydrogen phthalate for 2 hours at 120°C before use.

D-2.5 Crystal Violet, Indicator Solution — Dissolve 100 mg of crystal violet in 100 mL of acetic acid.

D-2.6 Perchloric Acid, 0.1 mol/L Standard Solution — To 8.5 mL of a 70 percent (m/m) aqueous solution of perchloric acid, add 300 mL of acetic acid followed by 20 mL of acetic anhydride. Dilute to 1 Iitre with acetic acid and mix thoroughly.

Standardize this solution by titrating it against 200 mg of potassium hydrogen phthalate dissolved in 50 mL of acetic acid, using the crystal violet indicator solution (see Note 1).

Use 4 to 6 drops of the crystal violet indicator solution and continue the titration until a stable green colour is obtained. Note the temperature, t_s, of the solution of perchloric acid at the time of standardization (see Note 2). The concentration, C, in mol/L of the standard perchloric acid solution, is given by the formula:

$$C = \frac{m}{V \times 0.20422}$$

Where,

m = is the mass (in g) of potassium hydrogen phthalate, and

V= is the volume (in mL) of perchloric acid used in the titration.

NOTES

1. If a potentiometric method is used, it is necessary to standardize the perchloric acid in the same way as used for the test.

2. The use of a correction factor is necessary if the temperature of the perchloric acid at the time of its standardization is different from that at the time of the test. This is because of the significant coefficient of expansion of the standard perchloric acid solution (1.07×10^{-3} $°C^{-1}$), which corresponds to a volume variation of 0.1 percent °C.

D-2.7 Tetraethylammonium Bromide Reagent Solution – Dissolve 100 g of tetraethylammonium bromide in 400 mL of acetic acid. Add a few drops of the crystal violet indicator solution; if it changes colour, bring it back to the original colour with the perchloric acid standard solution.

NOTE — For some epoxy compounds of low reactivity, the use of tetrabutylammonium iodide is advised, either as the solid or as a 10 percent solution in chloroform; in this case, light should be excluded as much as possible. Solutions of tetrabutyl ammonium iodide in chloroform are unstable and should be freshly prepared for each titration.

D-3 Apparatus

D-3.1 Balance — Accurate to within 0.1 mg.

D-3.2 Conical Flask — 100 mL or 200 mL, with ground glass neck and ground glass stopper.

D-3.3 Micro-burette — of capacity 10mL.

D-3.4 Magnetic Stirrer — With polytetrafluorethylene-coated bar.

D-3.5 Thermometer — Calibrated to permit temperature measurements to within = 0.1 °C.

D-3.6 Pipette, **One-mark, 10 mL Capacity**

In addition the following apparatus is required if a potentiometric end-point is to be used.

D-3.7 pH-millivoltmeter — preferably of the compensating type.

D-3.8 Glass and Calomel Reference Electrodes — The electrode system shall have a salt bridge of lithium chloride in glacial acetic acid. If the electrodes are not in constant use, wash the glass electrode with butanone, rinse with water, soak the electrode for at least 10 minutes in a solution of dilute hydrochloric acid (9 mL of hydrochloric acid made up to 100 mL with water) and rinse again with water and butanone.

If the electrodes are in constant use, it is sufficient to soak the electrodes in water between carrying out tests.

D-4 Procedure

D-4.1 Method A

Weigh into the flask, to the nearest 0.2 mg, a quantity of sample containing from 0.6 millimoles to 0.9 millimoles of epoxide groups. (This corresponds to a mass of between 0.6 x EE mg and 0.9 x EE mg, where EE is the epoxide equivalent of the epoxide compound.)

Add 10 mL of the solvent chosen for solution of the sample; then dissolve the sample by stirring and, if necessary, heat slightly. Cool the solution to room temperature. Add 20 mL of the acetic

acid and, with the pipette, 10 mL of the tetraethylammonium bromide reagent. Then add 4 to 6 drops of the crystal violet indicator solution.

Titrate with the standard perchloric acid solution immediately, in a closed system while stirring the contents of the flask by means of the magnetic stirrer. Continue the titration until a stable green colour is obtained.

Note the temperature, r, of the standard perchloric acid solution.

At the same time carry out a blank test, omitting the sample.

If a potentiometric end point is used, carry out the above procedure using the electrodes and omitting the indicator solution. Take the mid-point of the inflection on the titration curve as the end point.

D-4.2 Method B

Follow the procedure for Method A, including the blank test. Also carry out a second test with the sample, but without the addition of the tetraethylammonium bromide reagent.

D-5 Expression of Results

D-5.1 Method A

The epoxide equivalent, EE, in grams per mole, is given by the formula:

$$EE = \frac{1000 \times m}{(V_1 - V_0)(1 - \frac{t - t_s}{1000}) \times C}$$

Where,

m = is the mass (in g) of the test sample;

V_0 = is the volume (in mL) of the standard perchloric acid solution used in the blank test;

V_1 = is the volume (in mL) of the standard perchloric acid solution used in the test on the sample;

t = is the temperature (in °C) of the standard perchloric acid solution at the time of tests;

t_s = is the temperature (in °C) of the standard perchloric acid solution at the time of standardization; and

C = is the concentration (in mol/L) of the perchloric acid solution at the time of standardization.

The result is sometimes expressed as epoxide index (in moles of epoxide groups per kg) calculated as follows:

$$\text{Epoxide index} = \frac{1000}{\text{EE}}$$

D-5.2 Method B

The epoxide equivalent, EE, in grams per mole, is given by the formula:

$$EE = \frac{1000 \times m}{(V_1 - V_0 - V_2 \frac{m}{m_1})(1 - \frac{t}{1000}) \times C} - t_s$$

Where

m = is the mass (in g) of the test sample;

m_1 = is the mass (in g) of the test sample used in the second test, that is, without the addition of the tetraethylammonium bromide reagent;

V_0 = is the volume (in mL) of the standard perchloric acid solution used in the blank test;

V_1 = is the volume (in mL) of the standard perchloric acid solution used in the first test on the sample;

V_2= is the volume (in mL) of the standard perchloric acid solution used in the second test on the sample, that is, without the addition of the tetraethylammonium bromide reagent;

t = is the temperature (in °C) of the standard perchloric acid solution at the time of the tests;

t_s = is the temperature (in °C) of the standard perchloric acid solution at the time of standardization; and

C = is the concentration (in mol/L) of the perchloric acid solution at the time of standardization.

The result is sometimes expressed as epoxide index, expressed in moles of epoxide groups per kilogram, calculated as follows:

$$\text{Epoxide index} = \frac{1000}{\text{EE}}$$

D-6 Test Report

The test report shall include the following particulars:

a) A complete identification of the material tested, including type, source, manufacturer's code numbers etc;

b) The epoxide equivalent;

c) The solvent used for solution and the reagent used if it is not tetraethylammonium bromide:

d) Whether a visual or potentiometric end-point was used; and

e) Any other factor likely to have affected the result.

Annex E

[Clause 4.2 and Table 1, Sl. No. (iv)]

Determination of Hydrolyzable Chlorine Content

E-0 Outline of the Method — The material is refluxed in the presence of a known amount of 0.1 N alcoholic potassium hydroxide solution. The amount of potassium hydroxide consumed in the hydrolysis is a measure of the hydrolyzable chlorine content.

E-1 Apparatus

E-1.1 Reflux Apparatus

E-2 Reagents

E-2.1 Standard Hydrochloric Acid — 0.1 N.

E-2.2 Phenolphthalein Indicator Solution — 0.1 percent.

E-2.3 Alcoholic Potassium Hydroxide Solution — Dissolve 5.5 to 6.0 g of potassium hydroxide in 1000 mL of ethyl alcohol.

E-2.4 Toluene

E-3 Procedure — Weigh 6 to 8 g of the material accurately and transfer to a 250-mL long-necked flask. Pipette 50 mL of alcoholic potassium hydroxide solution into the flask and add 15 mL of toluene. Stopper the flask, swirl to mix the contents. Attach the reflux condenser and reflux gently for 15 ± 1 minutes. At the end of this period, remove the flask, cool to room temperature. Rinse down the condenser, remove the condenser and add a few drops of the phenolphthalein indicator. Titrate the solution against standard hydrochloric acid to an end point when the colour of the solution changes from pink to colourless.

NOTE - About 100 mL of methyl ethyl ketone may be added to the material, if required, to ensure a homogeneous solution during titration of excess potassium hydroxide solution.

E-4 Calculation

$$\text{Hydrolyzable chlorine (as Cl), percent by mass} = \frac{(V_1 - V_2)N \times 3.55}{M}$$

Where

V_2 = volume in mL of standard hydrochloric acid required for titration of the material,

V_1 = volume in mL of standard hydrochloric acid required for titration of the blank,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for test.

Annex F

[Clause 4.2 and Table 1, Sl. No. (iv)]

Determination of Relative Density

F-0 Outline of the Method - This method prescribes the determination of relative density of materials which are highly viscous, solids or semisolids and free-flowing liquids at 27°C, by pyknometers.

F-1 Apparatus

F-1.1 Pyknometer - conforming to Type 4 (for free-flowing liquids) and Type 6 (for viscous, semi-solid or solid materials) as shown in Fig. 3.

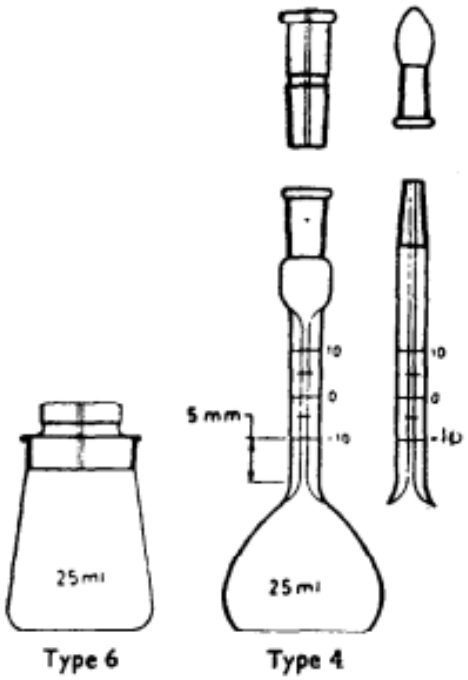

Fig. 3 Pyknometer

F-2 Procedure

F-2.1 Calibration - Clean the pyknometer first by soaking in potassium chromate solution and then washing thoroughly with water, alcohol and ether. Dry the pyknometer thoroughly and weigh accurately. Fill the pyknometer with freshly boiled water, taking care that no air-bubbles are entrapped. Keep the pyknometer in a water-bath maintained at 27 ± 2°C till constant temperature is attained (approximately 30 minutes). Remove the pyknometer from bath, wipe dry with a clean cloth and weigh immediately.

F-2.2 For Liquids that Flow Readily - Fill a clean, dry pyknometer with the material to be tested, proceed as prescribed in F-2.1 and record the mass. Calculate the relative density of the material as the ratio of the mass of the material and equal volume of water.

NOTE - If air-bubbles are entrapped, centrifuge the material either before or after filling the pyknometer.

F-2.3 For Viscous, Semi-Solid or Solid Materials - Fill a clean, dry pyknometer one-half full with the material to be tested. Precautions shall be taken to prevent the material from touching the sides of the pyknometer above the final level and to prevent inclusion of air-bubbles. It is advisable to warm the bottle before filling. Cool the bottle with its contents to 27 ± 2°C in a water-bath. Remove the pyknometer, wipe dry and weigh immediately. Fill the pyknometer with water and quickly push under water contained in a beaker at 20°C. Insert the stopper while the pyknometer is under water and transfer the pyknometer to a water-bath maintained at 27 ±2 °C. Allow the pyknometer to attain the temperature of bath (approximately 30 minutes). At the end of this period, carefully blot dry the top of the stopper, being careful not to draw water from stopper opening. Remove the pyknometer from bath, wipe dry and weigh.

F-2.3.1 Calculation - Calculate relative density as follows:

$$\text{Relative density} = \frac{M_3 - M_1}{(M_2 - M_1) - (M_4 - M_3)}$$

Where

M_3 = mass in g of the pyknometer plus material,

M_1 = mass in g of empty pyknometer,

M_2 = mass in g of the pyknometer plus water, and

M_4 = mass in g of the pyknometer plus material plus water.

Annex G

[Clause 4.2 and Table 1, Sl. No. (iv)]

Method of Sampling

G-1 General Requirements - In drawing and storing samples, the following precautions and directions shall be observed.

G-1.1 Samples shall not be taken in an exposed place.

G-1.2 Sampling instruments shall be clean and dry at the commencement of sampling.

G-1.3 The sampled material shall be placed in clean, dry and air-tight containers on which the material has no action.

G-1.4 Each sample container shall be sealed after filling and marked with full details of sampling.

G-2 Scale of Sampling

G-2.1 Lot - All the containers in a consignment of resins of the same type and same batch of manufacture shall be grouped to constitute a lot.

G-2.2 The number of containers to be selected for sampling shall be as given in Table 5.

Table 5 Scale of Sampling

No. of Containers in the Lot (1)	**No. of Containers to be Selected for Sampling** (2)
Up to 50	3
51 to 100	4
101 to 200	5
201 to 300	6
301 and above	7

G-2.2.1 These containers shall be selected at random from the lot, preferably with the help of random number tables (see BDS 1765).

G-3 Procedure for Taking Samples from Containers

G-3.1 Solid Resins - If the material is supplied in the form of large lumps packed in containers, a suitable sampling implement is required to withdraw samples from the selected containers. It is recommended that a portable steel spike, with a wooden handle, as shown in Fig. 4, be used. The total mass of the spike shall be 2 kg and the mass of the steel shank shall be 15 kg.

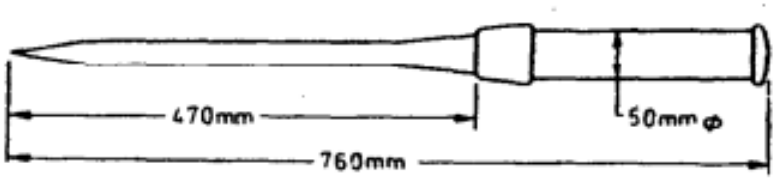

Fig. 4 Steel Spike

G-3.1.1 From each of the selected containers, remove the top surface to a depth of 10 cm with the help of steel spike. From the exposed surface draw a sample of 500 g of resin at different depths using the steel spike. The collected material shall be broken to small lumps and kept in separate sample containers. These individual samples representing each of the selected containers in the lot shall be examined for colour and visible impurities.

G-3.1.2 From each of the individual samples, approximately equal quantities of resin shall be taken and mixed together to give a composite sample weighing 1 kg. This composite sample shall be used for conducting various tests mentioned in material specifications.

G-3.2 Resin Solution - If the resin is supplied in the form of solution, each of the containers selected from the lot shall be opened and individually examined for clarity, sedimentation/settling and layer separation. After these visual tests are conducted, approximately equal quantities of resin solution shall be taken from the selected containers at different depths for checking solids and viscosity in order to ascertain the homogeneity of solution and these portions shall be mixed together to give a composite sample of 1 litre.

G-4 Number of Tests and Criteria for Conformity

G-4.1 Tests for colour, visible impurities and, if required by the purchaser, test for relative density shall be conducted on each of the individual samples. Tests for all other characteristics shall be conducted on the composite sample.

G-4.2 The lot shall be deemed to conform to the specification if all the test results (see G-4.1) satisfy the corresponding requirements when tested as prescribed in this standard.