भारतीय मानक
Indian Standard

IS 18174 : 2023

पॉलिथर पोलिओल्स — विशिष्टि

Polyether Polyols — Specification

ICS 83.080.10

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March 2023 **Price Group 5**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Polyol is an organic compound containing multiple hydroxyl groups. Polyols are reacted with compounds containing isocyanate groups to produce polyurethanes (PU). There are different kinds of polyols , among them polyether polyols are predominantly used in the manufacturing of polyurethanes and this standard refers information relating only to polyether polyols. Generally, polyether polyols are made by the reaction of epoxides with compounds having active hydrogen atom (initiator or starting compound) . The number of active hydrogen atoms in the initiator determines the functionality of the resultant polyol. Di, tri and poly hydroxy compounds/ amines and mannich bases are regularly used as initiators. The properties of polyether polyol can be greatly varied by changing the type of initiators, epoxides, catalysts and the reaction conditions. Due to this versatility, the polyurethanes made from polyether polyols are used in wide varieties of application like seating, mattresses, thermal insulation of refrigerators and freezers, elastomeric shoe soles, coatings, sealants, and adhesives. Tri-functional polyether polyols are suitable for the production of flexible slabstock (continuous or discontinuous process) and moulded PU foam, which are used in furniture, mattresses and automotive seating application. High functional with medium to high hydroxyl values polyether polyols are used in the production of rigid PU foams that are used as thermal insulation materials. Styrene and acrylonitrile grafted polyether polyols are used in the production of high load bearing flexible PU foam.

Hydroxyl value of polyether polyol is important for quality control in production of polyurethane. It is defined as the number of mg of potassium hydroxide required to neutralize the acetic acid taken up on acetylation of 1 g of a chemical substance that contains free hydroxyl groups.

In the preparation of the test method of hydroxyl value the assistance has been derived from the following ASTM test method:

a) ASTM D 4274 Standard Test Methods for Testing Polyurethane Raw Materials: Determination of Hydroxyl Numbers of Polyols.

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Petrochemicals, the committee has decided to formulate Indian Standard on Polyether Polyol.

The composition of the committee, responsible for the formulation of this standard is listed in Annex B.

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 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. 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

POLYETHER POLYOLS — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and tests for polyether polyols used in polyurethane industry.

1.2 This standard does not cover bio-based or natural oil based polyols.

1.3 This standard does not cover formulated polyols, polyols with blowing agents and polyols with flame retardant and other additives.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No./Other Publications	*Title*
IS 2362 : 1993	Determination of water by karl fischer method — Test method (*second revision*)
IS 13360 (Part 11/ Sec 10) : 2022/ ISO 2555 : 2018	Plastics — Methods of testing: Part 11 Special properties, Section 10 Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity using a singular cylinder type rotational viscometer method
ISO 14900 : 2017	Plastics — Polyols for use in the production of polyurethane — Determination of hydroxyl number

3 GRADES

Polyether polyols shall be of the following four grades:

a) *Grade 1* — Polyether polyol used for the manufacturing of cellular materials like rigid polyurethane foams;

b) *Grade 2* — Polyether polyols without solids (styrene and/or acrylonitrile, SAN particles) used for the manufacturing of cellular materials like flexible slabstock and microcellular foams;

c) *Grade 3* — Polyether polyols co-polymerized/grafted with styrene and acrylonitrile (SAN) are used in the manufacture of flexible and microcellular and footwear applications; and

d) *Grade 4* — Polyether polyols for speciality and coating, adhesives, sealants, elastomers (CASE) applications.

4 REQUIREMENT

4.1 The material shall also comply with the requirements as given in Table 1, when tested according to the methods prescribed in col (7) of the Table 1.

4.1.1 Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070), shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do notcontain impurities which affect the results of analysis.

Table 1 Requirement for Polyether Polyol
(*Clause* 4.2, 6.2 *and* 6.3.1)

Sl No.	Characteristic	Requirement				Method of Test, Ref to
		Grade 1	Grade 2	Grade 3	Grade 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Water Content, percent by weight, *Max*	00.25	0. 20	0.20	0.10	IS 2362[1)]

Sl No.	Characteristic	Requirement				Method of Test, Ref to
		Grade 1	Grade 2	Grade 3	Grade 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
ii)	Hydroxyl Value, mg KOH/g, *Max*	1 200	260	50	800	Annex A/ISO 14900
iii)	Dynamic Viscosity at 25 ºC, mPa·s, *Max*	60 000	10 000	10 000	10 000	IS 13360 (Part 11/Sec 10)

[1)] The auto titrator and standardized ready to use karl fischer reagent may be used.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable drums or bulk tankers conforming to local transportation guidelines.

5.2 Marking

5.2.1 Each container shall be securely closed and shall bear and indelibly the following information:

a) Name of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Net weight of material;
d) Month and year of the manufacture;
e) Batch or code number; and
f) Any other statutory requirements.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in **4** of IS 5299.

6.2 Tests for the determination of all other characteristics given in Table 1 shall be conducted.

6.3 Criteria for Conformity

6.3.1 *For Composite Sample*

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

ANNEX A
[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF HYDROXYL VALUE (OH VALUE) OF POLYOLS

A-1 GENERAL

This procedure is used to determine the hydroxyl number (OH Value) in polyether over the range of 20 mg KOH/g to 1800 mg KOH/g. Two methods namely, Method A Determination of hydroxyl value by phthalic anhydride solution with 1-methyl imidazole as catalyst medium using pressure bottle as reaction container and Method B Determination of hydroxyl value by phthalic anhydride solution, reflux condenser used during the reaction are prescribed.

NOTE — Instead of phthalic anhydride, acetylation mixture may also be used.

A-2 METHOD A

A-2.1 Apparatus

A-2.1.1 *Auto Titrator*

NOTE — Manual Titrator complying to the procedure of the mentioned test method may also be used.

A-2.1.2 *Analytical Balance*, accurate to 1.0 mg.

A-2.1.3 *Glass Screw Thread Erlenmeyer Flask*, 150 ml.

A-2.1.4 *p*H *Electrode with 3 M Potassium Chloride* (*KCl*) *Electrolyte.*

A-2.1.5 *Hot Plate*, capable to maintain (110 ± 2) °C.

A-2.1.6 *Syringe,* 2 ml.

A-2.1.7 *Magnetic Stirrer and Bar*

A-2.2 Reagents

A-2.2.1 *Phthalic Anhydride Solution,* using 1-methylimidazole.

Add 270 g of phthalic anhydride to 1.5 litres of pyridine. After the anhydride is completely dissolved, add 45 ml of 1-methylimidazole into a 2.5 litres amber coloured glass bottle. Mix it for atleast 2 h. Use the solution at least 24 h after preparing it.

A-2.3 Procedure

A-2.3.1 *Blank Titration*

Switch on the titrator, click manual control, and recirculate 500 ml of sodium hydroxide (NaOH) solution before titration. Insert a magnetic bar in a 150 ml erlenmeyer flask. Add 20 ml of phthalic anhydride solution into the flask with dispenser. Add 90 ml of distilled water.

Select the blank method procedure from the instrument, if it is programmed already else select the option manually. Click the start button. Titrate with 1 N sodium hydroxide (NaOH) to the equivalent point. View the results in the report window. Constantly monitor and record the titrant value each time a blank series is run. Repeat blank tests when the titrant's value varies considerably from previous test.

NOTES

1 Recirculate 80 ml of phthalic solution through dispenser before use.
2 Electrode calibration is not mandatory since the titration occur at equivalent point.

A-2.3.2 *Sample Titration*

Weigh a clean and dry erlenmeyer flask on analytical balance. Weigh an amount of sample in a 150 ml flask according to the following formula:

$$\text{Sample weight, g} = \frac{374}{theoretical\ OH\ number}$$ (used for OH values graeter than 300)

NOTE — For OH value less than 300, weigh 1.7 g.

Add 20 ml of the phthalic anhydride solution into the erlenmeyer flask with dispenser. Cap the erlenmeyer flask and place it in hot plate, maintened at (110 ± 2) °C for 30 min to complete the esterification reaction. Remove the erlenmeyer flask from the heating device and cool to room temperature for 10 min. Carefully uncap the flask and rinse the cap with 50 ml of distilled water, catch this water in the bottle and add another 40 ml of distilled water washing with care the walls of the flask. Allow the solution to mix for at least 2 min and then titrate with 1 N sodium hydroxide (NaOH) to equivalent point. Wash carefully the electrode with distilled water after each titration. Between one analysis and the other one, it's advisable to leave the electrode in a 50/50 (*v/v*) solution of isopropyl alcohol/water. For longer periods, leave in proper electrolyte.

A-2.4 Calculation

$$\text{Hydroxyl Number, mg KOH/g} = \frac{(V_1 - V_2) \times N \times 56.1}{W}$$

where

N = normality of sodium hydroxide (NaOH);
V_1 = volume of sodium hydroxide solution required for titration of blank, in ml;
V_2 = volume of sodium hydroxide solution required for titration of sample, in ml; and
W = weight of sample, in g.

NOTE — Molecular weight of potassium hydroxide (KOH) is 56.1 g.mole^{-1}

$$\text{Hydroxyl content, percent by weight} = \frac{Hydroxyl\ number \times 17}{560}$$

A-3 METHOD B

A-3.1 Apparatus

A-3.1.1 *Oil Bath*, Maintained at 115 °C to 120 °C

A-3.1.2 *Flat Bottom Reflux Flask*, 250 ml.

A-3.1.3 *Air Condenser*, 1 m length to fit the reflux flask.

A-3.2 Reagents

A-3.2.1 *Pyridine-Phthalic anhydride solution*, 15 percent.

A-3.2.2 *Sodium Hydroxide Solution*, 0.5 N.

A-3.2.3 *Phenolphthalein Solution (in pyridine),* 1.0 percent.

A-3.3 Procedure

Weigh the calculated amount of sample carefully and accurately into the reflux flask. Transfer exactly 25 ml of pyridine-phthalic anhydride solution in the reflux flask and swirl the content thoroughly to ensure complete mixing of the sample and reagent. Insert air condensers to the reflux flasks and place the flasks in an oil bath maintained at 115 °C to 120 °C for 30 min**.** Ensure that there is sufficient oil in the bath to cover approximately one half of the flask.

After the heating period, remove the assembly from the bath and cool to room temperature. Wash the condenser with 50 ml of pyridine and remove the condenser. Add 0.5 ml of phenolphthalein solution and titrate with 0.5 N sodium hydroxide solution to pink end point that persists for alteast 15 s. Note the titrate value in ml as V_2.

Run a blank titration (without sample) using the above procedure and note the titrate value in ml as V_1.

NOTES

1 To arrive at the sample size, use the relation, sample size $= \frac{561}{\text{Expected Hydroxyl value}}$.

2 Always perform the experiment with a duplicate and use a standard sample of known hydroxyl number.

A-3.4 Calculation

Hydroxyl Number (mg KOH/g) =

$$\frac{(V_1 - V_2) \times N \times 56.1}{W}$$

where

N = normality of sodium hydroxide (NaOH);

V_1 = volume of sodium hydroxide solution required for titration of blank, in ml;

V_2 = volume of sodium hydroxide solution required for titration of sample, in ml; and

W = weight of sample, in g.

NOTE — Molecular weight of potassium hydroxide (KOH) is 56.1 $g.mole^{-1}$.

ANNEX B
(*Foreword*)

COMMITTEE COMPOSITION
Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
National Chemical Laboratory (NCL), Pune	DR C. V. RODE **(*Chairperson*)**
Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (*Alternate*)
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL (*Alternate*)
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Member Secretary
MS ADITI CHOUDHARY
SCIENTIST 'B'/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: PCD 09 (19088).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi