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भारतीय मानक

प्रोपाइलीन ग्लाइकोल, खाद्य ग्रेड — विशिष्टि Indian Standard

PROPYLENE GLYCOL, FOOD GRADE — SPECIFICATION

UDC 664'009'6: 547'422'23

BIS 1993

BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

AMENDMENT NO. 1 FEBRUARY 2006 TO IS 13702: 1993 PROPYLENE GLYCOL, FOOD GRADE — SPECIFICATION

(Page 1, clause 2) — Substitute '1699: 1995 Methods of sampling and test for food colours (second revision)' for '1699: 1974 Methods of sampling and test for food colours (first revision)'.

[Page 1, Table 1, Sl No. (iii), col 5] — Substitute 'Cl 15 of IS 1699: 1995' for 'Cl 9 of IS 1699: 1974'.

[Page 1, Table 1, Sl No. (vi), col 3] — Substitute '0.07' for '0.007'.

(Page 2, clause **5.1**, line 5) — Substitute '4 of IS 1699 : 1995' for '3 of IS 1699 : 1974'.

(FAD 8)

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As certain impurities in these substances could be harmful, it is necessary to have a strict quality control of these food additives. A series of standards, is therefore, being prepared by this Bureau to cover purity and identification of these substances. These standards would help in checking purity which require to be checked at the stage of manufacture for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies.

Propylene glycol is a clear, colourless, practically odourless, viscous liquid having a slight characteristic taste. It is used as a diluent or filler material in liquid food colour preparations. It is also used as a solvent, wetting agent and humectant.

Propylene glycol is also known as 1, 2-propanediol, 1, 2-dihydroxypropane and methyl glycol. Its molecular weight is 76.1 and empirical formula $C_3H_8O_2$.

In the preparation of this standard considerable assistance has been derived from Food Chemical Codex, National Academy of Science, Pub. National Research Council, Washington D.C. USA 1981.

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

PROPYLENE GLYCOL, FOOD GRADE — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for propylene glycol, food grade.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title	
1070 : 1992	Reagent grade water (third revision)	
1699 : 1974	Methods of sampling and test for food colours (first revision)	
2362:1973	Determination of water by the Karl Fischer method (first revision)	

3 REQUIREMENTS

3.1 Description

Propylene glycol shall be a clear, colourless, practically odourless, viscous liquid having a slight characteristic taste. It shall absorb moisture when exposed to air. It shall be miscible with water, acetone and chloroform in all proportions. It shall be soluble in ether and will dissolve many essential oils, but is immiscible with fixed oils.

3.2 Identification

3.2.1 Mix 500 mg propylene glycol with 3.6 grams of triphenylchloromethane and 5 ml of pyridine and heat under a reflux condenser on a steam bath for one hour. Cool, dissolve the

mixture in 100 ml of warm acetone and stir well with 100 mg of activated charcoal. Filter, evaporate the filtrate to about 50 ml and allow to stand overnight in a refrigerator. Filter off the crystals, recrystallize until free from pyridine (three times) and dry in a current of air. The crystals so obtained melt at about 176°C.

3.2.2 Heat gently 1 ml of propylene glycol with 500 mg of potassium bisulphate. A fruity odour is evolved, and when the solution is heated to dryness, no sharp acrid odour of acrolein is perceptible.

3.3 Specific Gravity

The specific gravity of the material at 25°/25°C shall be between 1.035 and 1.037.

3.4 Distillation Range

A 100 ml sample shall distil completely between 185°C and 189°C.

3.5 The material shall also conform to the requirements given in Table 1.

4 PACKING, STORAGE AND MARKING

4.1 Packing and Storage

The material shall be filled in well closed containers so as to preclude air contamination of the contents with metal or other impurities.

4.2 Marking

Each container shall be marked legibly to give the following information:

a) Name of the material including the words 'food grade';

Table 1 Requirements for Propylene Glycol, Food Grade (Clause 3.5)

SI	Characteristic	Requirements	Method of Test, Ref to	
No.			Annex of this Standard	Other Indian Standards
(1)	(2)	(3)	(4)	(5)
i)	Purity (as C ₂ H ₈ O ₂), percent by mass, Min	. 99.5	A	_
ii)	Moisture, percent by mass, Max	0.2		IS 2362: 1973
iii)	Arsenic (as As), mg/kg, Max	3		Cl 9 of IS 16 99 : 1974
iv)	Heavy metals (as Pb), mg/kg, Max	10	В	
v)	Acidity	To pass test	C	
vi)	Sulphated ash (on dry basis), percent by mass, Max	0.007	D	
vii)	Presence of other polyhydroxy compounds	A bsent	Α	
viii)	Ethylene glycol	Absent	A	

IS 13702: 1993

- b) Name and address of the manufacturer:
- c) Minimum net mass of contents;
- d) Batch or code number;
- e) Date of manufacture; and
- f) Any other details required under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977 and Prevention of Food Adulteration Rules, 1955.
- 4.2.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 The representative samples of the material shall be drawn and conformity of the material to

the requirements of this specification shall be determined according to the procedure prescribed in 3 of IS 1699: 1974.

6 TESTS

6.1 Tests shall be carried out by the methods specified in 3.2, 3.3, 3.4 and col. 4 and 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070: 1992) shall be employed in tests.

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

ANNEX A

[Table 1, Sl No. (i), (vii) and (viii)]

DETERMINATION OF PURITY

A-1 APPARATUS

A-1.1 Gas Chromatograph

- a) Column: Stainless steel 1 m×1/4 inch packed with 4% carbowax compound 20 M on 40/60 mesh chromosorb T or equivalent material.
- b) Temperature:
 Column: 120° to 200° programmed at the rate of 5° per minute.
- c) Carrier Gas: Helium flowing at 75 ml per minute.
- d) Detector: Thermal conductivity type.

A-2 PROCEDURE

Inject a 10 μ l portion of the sample into a suitable gas chromatograph the details of which are given in A-1.1. Measure the values under all peaks by any convenient means and calculate the area percentage of propylene glycol and report as weight percentage.

NOTES

- 1 Under the conditions described, the approximateretention time for propylene glycol is 5.7 minutes and for the three isomers of dipropylene glycol 8.2, 9.0 and 10.2 minutes.
- 2 Polyglycols are known to be highly toxic substances and the gas chromatography method also detects other polyglycols as well as contaminating compounds other than polyglycols,

ANNEX B

[Table 1, Sl No. (iv)]

DETERMINATION OF HEAVY METALS

B-1 REAGENTS

B-1.1 Ammonia Solution

Dilute 400 ml of ammonium hydroxide (28 percent) to 1 000 ml with water.

B-1.2 Hydrochloric acid — 10 percent.

B-1.3 Lead Nitrate Stock Solution

Dissolve 159.8 mg of lead nitrate in 100 ml of water containing 1 ml of nitric acid. Dilute with water to 1000 ml and mix. Prepare and

store the solution in lead-free glass containers.

B-1.4 Standard Lead Solution

Dilute 10 ml of lead nitrate stock solution accurately measured, with water to 100 ml. Each ml of the solution so prepared contains the equivalent of 10 μ g of lead ion (Pb). Prepare the solution on the day of use.

B-1.5 Nitric Acid — 10 percent (v/v).

B-1.6 Sulphuric Acid — 94.5 to 95.5 percent (ν/ν) .

B-1.7 Acetic Acid — 6 percent (m/v).

B-1.8 Hydrogen Sulphide

A saturated solution of hydrogen sulphide made by passing H₂S in cold water.

B-2 PROCEDURE

B-2.1 Solution A

Take 2 ml of the standard lead solution in a 50-ml Nessler tube and add 23 ml of water. Adjust the pH to between 3.0 to 4.0 by addition of acetic acid or ammonia solution. Dilute with water to 40 ml and mix.

B-2.2 Solution B

Make a solution of 2 grams of substance in 25 ml of water and put in a 50-ml Nessler tube that matches the one used for solution A and adjust the pH to between 30 and 40 (pH indicator paper) by the addition of dilute acetic acid solution or ammonia solution. Dilute to 40 ml with water and mix.

B-2.3 To each tube add 10 ml of freshly prepared hydrogen sulphide, mix and allow to stand for 45 minutes and view down over a white surface. The colour of solution B shall not be darker than that of solution A.

ANNEX C

[Table 1, Sl No. (v)]

TEST FOR ACIDITY

C-1 REAGENTS

C-1.1 Phenolphthalein

Dissolve 1 gram of phenolphthalein in 100 ml of alcohol.

C-1.2 Sodium Hydroxide - 0.1 N.

C-2 PRO CEDURE

C-2.1 Add 1 ml of phenolphthalein to 50 ml of water, then add 0.1 N sodium hydroxide until the solution remains pink for 30 seconds. To this solution add 10 ml of the sample, accurately measured and titrate with 0.1 N sodium hydroxide until the original pink colour returns and remains for 30 seconds. Not more than 0.2 ml of 0.1 N sodium hydroxide is required.

ANNEX D

[Table 1, Sl No. (vi)]

DETERMINATION OF SULPHATED ASH

D-1 REAGENTS

D-1.1 Dilute Sulphuric Acid — 10 percent (m/v).

D-2 PROCEDURE

D-2.1 Transfer 50 grams sample in a tared 100-ml shallow dish. Heat until it ignites and allow it to burn without further application of heat in a place free from drafts. Cool, moisten the residue with 5 ml of sulphuric acid, and ignite in a muffle furnace at $800 \pm 25^{\circ}$ C for 15

minutes. Cool in a desiccator and weigh.

D-3 CALCULATION

Sulphated ash,

percent by mass =
$$\frac{M_1}{M} \times 100$$

where

 $M_1 = \text{mass}$, in g, of residue, and

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

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