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भारतीय मानक मसौदा

ग्लिसराइल मोनोस्टेरेट, खाद्य ग्रेड — विशिष्टि

(*आइ एस* 9953 *का पहला पुनरीक्षण*)

Draft Indian Standard

GLYCERYL MONOSTEARATE, FOOD GRADE— SPECIFICATION

(*First Revision of IS* 9953)

ICS No. 67.220.20

Food Additives Sectional Committee, FAD 08 Last Date of Comments: **20 September 2023**

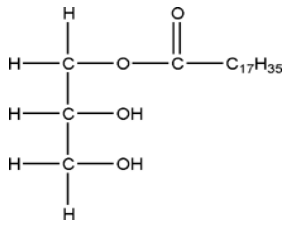
FOREWORD

(*Formal clauses would be added later*)

With the increased production of processed foods, food additives are added generally in small quantities to improve the appearance and properties of the processed foods like flavour, texture or storage properties, etc. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives.

Glycerol monostearate is a glycerol ester of stearic acid, usually manufactured by the glycerolysis of edible fats and oils. It may also be prepared by esterification of fatty acids with glycerol, with or without molecular distillation of the product. Glycerol monostearate is widely used as thickening, emulsifying, anti-caking, and preservative agent. Glycerol monostearate is largely used in baking preparations to add "body" to the food. It is responsible for giving ice cream and whipped cream its smooth texture. It is sometimes used as an anti-staling agent in bread. Use of glycerol monostearate is permitted as a food additive in the *Food Safety and Standards (Food Products Standards and Food Additives) Regulations*, 2011.

Glycerol monostearate is also known as monostearin. It's chemical formula, structural formula and molecular mass are given below:

Molecular Formula	Structural formula	Molecular mass
$C_{21}H_{42}O_4$		358. 6

This standard was first published in 1981.

In this revision, the following major changes have been made:

a) The requirement for heavy metals has been removed as the limit of lead (contaminant in food additives) is already covered through the standard;
b) The identification tests have been incorporated;
c) The limits of soap, mercury, and cadmium have been incorporated; and
d) The updated test method for determination of monoglyceride and free glycerol have been incorporated.

In the formulation of this standard, due consideration has been given to the provisions of the *Food Safety and Standards Act*, 2006 and the *Rules* and *Regulations* framed thereunder and the *Legal Metrology (Packaged Commodities) Rules*, 2011. However, this standard is subject to the restrictions imposed under these, wherever applicable.

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*)' This number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1 SCOPE

This standard specifies requirements and methods of sampling and test for glycerol monostearate, food grade.

2 REFERENCES

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

IS No.	*Title*
IS 1699: 1994	Methods of sampling and test for food colours *(second revision)*
IS 2491: 2013	Food hygiene – General Principles – Code of Practice *(third revision)*
IS 3508: 1996	Method of sampling and test for Ghee
IS 4236: 1999	Glycerol monostearate for cosmetic industry - Specification *(third revision)*

3 REQUIREMENTS

3.1 Description

It shall be white to creamish white in colour, in the wax like solid form, powder or granules. It shall have slight characteristic fatty odour and taste and shall be free from rancidity.

3.2 Identification

3.2.1 *Solubility*

It shall be insoluble in water and soluble in ethanol, chloroform and benzene

3.2.2 *Infrared absorption*

The infrared spectrum of the sample of the product shall be characteristic of a partial fatty acid ester of a polyol.

3.2.3 *Tests for Fatty Acids*

Treat 1 g of the sample by reflux using a 0.5 M potassium hydroxide solution for 1 hour. Add 15 ml of water and acidify with hydrochloric acid diluted to 30 percent (*v/v*) (R) (approximately 4-5 ml). Oily drops or a white/yellowish white precipitate will form. Extract the fatty acids released using 5 ml hexane, separating the solvent. Repeat the extraction with 5 ml of hexane and reunite the two extracts. Detect the fatty acids in the hexane extract by gas phase chromatography using a semi-polar column, e.g., Carbowax 20M ® measuring 25 m × 0.32

mm × 0.25 mm phase thickness. Run solution of stearic acid in hexane and match the peaks for identification.

3.2.4 *Detection of Glycerol*

Place 5 ml of the aqueous phase in a test tube. Add an excess amount of powdered calcium hydroxide and place the test tube in boiling water for five minutes, stirring from time to time. Cool and filter. Place one drop of the filtrate in a test tube and add approximately 50 mg of potassium hydrogen sulphate. At the end of the test tube, place a piece of filter paper soaked in the reagent obtained by mixing extemporaneously equal volumes of a sodium nitrosopentacyanoferrate solution (R) and piperidine (F'). Heat the paper using a small flame. A blue colouring of the reactive paper indicates the presence of acrolein. The colour turns red by adding 1 M sodium hydroxide solution.

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

Table 1 Requirements for Glyceryl Monostearate, Food Grade
(*Clause 3.3*)

Sl No.	Characteristic	Requirement	Annex of this Standard/ Other Indian Standards
(1)	(2)	(3)	(4)
i)	Acid value, *Max*	6.0	IS 4236
ii)	Monostearate content, percent by mass, *Min*	40.0	Annex A
iii)	Free glycerol, percent by mass, *Max*	7.0	Annex A
iv)	Soap, percent by mass, *Max*	6.0	Annex B
v)	Melting point (°C)	54 – 60	IS 4236
vi)	Iodine value, *Max*	5.0	IS 4236
vii)	Residue on ignition, percent by mass, *Max*	1.0	IS 4236
viii)	Iron (as Fe), mg/kg, *Max*	20.0	IS 4236
ix)	Moisture, percent by mass, *Max*	2.0	IS 4236
x)	Saponification value	140 – 155	IS 3508
xi)	Lead (as Pb), mg/kg, *Max*	2.0	IS 1699
xii)	Arsenic (as As), mg/kg, *Max*	3.0	IS 1699
xiii)	Mercury (as Hg), mg/kg, Max	1.0	IS 1699
xiv)	Cadmium (as Cd), mg/kg, Max	1.0	IS 1699

4 PACKING AND STORAGE

4.1 The material shall be properly packed in polythene lined laminated waterproof gunny bags or laminated woven polythene or polypropylene bags. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage — The material shall be stored in a cool and dry place.

5 MARKING

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

a) Name and type of the material, including the words 'Food Grade';
b) Name and address of the manufacturer;
c) Net contents;
d) Monostearate content;
e) Batch or code number;
f) Month and year of manufacture; and
g) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules,* 2011 and *Food Safety and Standards (Packaging and Labelling) Regulation*, 2011 and the Rules framed thereunder.

5.2 *BIS Certification Marking*

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

6 SAMPLING

Representative samples of the material shall be drawn according to the method prescribed in IS 1699.

7 TESTS

7.1 Tests shall be carried out by the methods specified in col **(4)** of Table 1.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) 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.* (ii) and (iii)]
DETERMINATION OF 1-MONOGLYCERIDE AND FREE GLYCEROL

A-1 REAGENTS

A-1.1 Acetic Periodic Acid – Dissolve 5.4 g of periodic acid in 100 ml of distilled water and then add 1900 ml of glacial acetic acid and mix thoroughly. Store the solution in a dark glass-stoppered bottle or store in the dark in a clear glass-stoppered bottle.

A-1.2 Potassium Iodide – A 16.5 percent (*w*/*v*) solution of potassium iodide (KI) in water (approximately N). Store in a light-resistant container.

A-1.3 Starch – Triturate 1 g of arrowroot starch with 10 ml of cold water, and pour slowly, with constant stirring, into 200 ml of boiling water. Boil the mixture until a thin, translucent fluid is obtained. (Longer boiling than necessary renders the solution less sensitive.) Allow to settle, and use only the clear, supernatant liquid. Prepare this solution fresh.

A-2 PREPARATION OF SAMPLES

A-2.1 Solid Samples in Flake Form – Mix without melting and take a portion for analysis.

A-2.2 Solid Samples not in Flake Form

Melt at not more than 10° above melting point, mix thoroughly and take a portion for analysis. Do not attempt to test samples which contain so much free glycerol that it separates when the sample solidifies.

A-2.3 Semi-solid and Liquid Samples

Liquify by heating at not more than 10°C above melting point, mix thoroughly, and take a portion for analysis. Do not attempt to test samples which contain so much free glycerol that it separates from the sample when cooled to room temperature.

NOTE – The sample must not be subjected to a temperature in excess of that required to melt it, as this may reduce the monoglyceride content if any soap is present.

A-3 PROCEDURE FOR 1-MONOGLYCERIDE

A-3.1 Weigh to the nearest mg duplicate samples of 1 g into a 100 ml glass-stoppered volumetric flask. Dissolve in 50 ml of chloroform. Add 25 ml of water and shake vigorously for 30-60 sec. Transfer the aqueous layer to a glass-stoppered 100 ml volumetric flask, using a glass siphon. If an emulsion forms due to the presence of soap in the sample, add 3 or 4 ml of glacial acetic acid to break the emulsion. Extract 3 more times using 25, 25 and 20 ml of distilled water. Add chloroform to the flask until the level of the chloroform coincides with the 100 ml mark. Using the glass siphon, transfer as much as possible of the aqueous layer above the chloroform layer to the flask containing the aqueous extracts. The aqueous extracts in the volumetric flask are saved for the determination of free glycerol.

Pipet out 50 ml of acetic periodic acid (*see* **A-1.1**) into each of a series of 500 ml glass-stoppered Erlenmeyer flasks. Prepare 3 for blanks, adding 50 ml of chloroform to two and 50 ml of water to the third. The titrations of the water and chloroform blanks are used as a check (within 0.5 ml) on the chloroform. Pipet out 50 ml of chloroform sample solution into one the flasks containing 50 ml of acetic periodic acid (*see* **A-1.1**) and shake gently to effect thorough mixing. Allow to stand for at least 30 min but not longer than 1.5 h. To each flask add 20 ml of potassium iodide (*see* **A-1.2**). Mix by gentle shaking, allow to stand at least 1 min but not more than 5 min before titrating. Do not allow to stand in strong sunlight. Add 100 ml of distilled water and titrate with 0.1 N sodium thiosulfate. Use a variable speed magnetic stirrer to keep the solution thoroughly mixed. Continue the titration to the disappearance of the brown iodine colour from the aqueous layer. Add 2 ml of starch (*see* **A-1.3**) and continue the titration to the disappearance of iodine from the chloroform layer and the disappearance of the blue iodostarch colour from the aqueous layer.

A-3.2 Calculation

1-monoglyceride (as 1-monoglyceride content as pure monostearate),

$$\text{percent by mass} = \frac{(B-S) \times N \times 17.927}{M}$$

where,

B = the sodium thiosulfate consumed in the titration of blank containing 50 ml of chloroform;

S = the sodium thiosulfate consumed in the titration of sample;

N = the exact normality of 0.1 N sodium thiosulfate; and

M = the mass of sample, represented by aliquot pipetted for test in g.

NOTE – The 1-monoglyceride content may be calculated in terms of a monoester other than the monostearate by dividing the molecular weight of the monoglyceride by 20 and substituting this value for 17.927 in the formula above.

A-4 PROCEDURE FOR FREE GLYCEROL

A-4.1 Add distilled water to the combined aqueous extracts from the monoglyceride determination until the volume is 100 ml and mix thoroughly. Pipet out 50 ml of acetic periodic acid (*see* **A-1.1**) into each of a series of 500 ml glass-stoppered Erlenmeyer flasks. Pipet out 50 ml of aqueous sample solution into one of the flasks containing 50 ml of acetic periodic acid (*see* **A-1.1**) and shake gently to effect thorough mixing. Allow to stand for at least 30 min but not longer than 1.5 h. To each flask add 20 ml of potassium iodide (*see* **A-1.2**). Mix by gentle shaking, allow to stand at least 1 min but not more than 5 min before titrating. Do not allow to stand in strong sunlight. Add 100 ml of distilled water and titrate with 0.1 N sodium thiosulfate. Use a variable speed magnetic stirrer to keep the solution thoroughly mixed. Continue the titration to the disappearance of the brown iodine colour from the aqueous layer. Add 2 ml of starch (*see* **A-1.3**) and continue the titration to the disappearance of iodine from the chloroform layer and the disappearance of the blue iodostarch colour from the aqueous layer.

A-4.2 Calculation

Free glycerol, percent by mass = $\frac{(B-S) \times N \times 2.30}{M}$

where,

B = the sodium thiosulfate consumed in the titration of blank containing 50 ml of chloroform;

S = the sodium thiosulfate consumed in the titration of sample;

N = the exact normality of 0.1 N sodium thiosulfate; and

M = the mass of sample, represented by aliquot pipetted for test in g.

ANNEX B
[*Table* I*, Sl. No.* (iv)]
DETERMINATION OF SOAP

C-1 PROCEDURE

Add 10.00 g of the sample to a mixture of 60 ml of acetone and 0.15 ml of bromophenol blue solution (0.5%), previously neutralized with 0.1 N hydrochloric acid or 0.1 N sodium hydroxide. Warm gently on a water bath until solution is complete, and titrate with 0.1 N hydrochloric acid until the blue colour is discharged. Allow to stand for 20 min, warm until any solidified matter has re-dissolved and, if the blue colour reappears, continue the titration. Each ml of 0.1 N hydrochloric acid is equivalent to 0.0304 g of $C_{18}H_{33}O_2Na$.