कैस्टर ओलीन और कैस्टर ओलेओस्टीयरिन — विशिष्टि

(पहला पुनरीक्षण)

Castor Oleine and Castor Oleostearine — Specification

(First Revision)

ICS 71.100.40; 71.080.99

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Price Group 6

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Soaps, Detergents and Surface Active Agents Sectional Committee, CHD 25

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps, Detergents and Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Castor oil is abundantly available in India and has varied applications. However, because of the very high percentage of hydroxy acids, it is unsuitable for incorporation in fat charges for soap making, except in very restricted quantities. It is, however, possible to chemically treat this oil to reduce and/or remove the hydroxy groups so that the oil contains predominantly stearic and monoene acids and can therefore be used as raw material for soaps to substitute tallow or hardened oils.

This standard is intended to encourage more manufacturers, not only to make a standard product for captive use, but also to make it available for general use.

Castor oleine and oleostearine are triglycerides derived from castor oil, chemically treated to eliminate hydroxyl (OH) groups of ricinoleic acid. The product shall be oleine or oleostearine depending on the unsaturation in the final product.

This standard was originally published in 1983. In this revision, the scope has been broadened to allow use of the material for other non-edible industrial uses also which was earlier restricted to manufacturer of soaps only. Reference clause has been added.

This standard contains 5.1 and Table 1 which calls for agreement between the purchaser and the supplier.

The composition of the Committee responsible for the formulation of this standard is given in <u>Annex E</u>.

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

CASTOR OLEINE AND CASTOR OLEOSTEARINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for castor oleine and castor oleostearine.

2 REFERENCES

The standards listed in <u>Annex A</u> contain provisions, which through references 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 agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 286 and IS 7597 shall apply.

4 REQUIREMENTS

4.1 The material shall be reddish brown in colour having liquid or semi-solid consistency. When it is in liquid form, It shall be free from sediments, suspended particles and other foreign matter. It shall also be free from adulterants when tested as prescribed in IS 548 (Part 2).

4.2 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

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

5.2 Marking

5.2.1 The containers shall be marked with the following information:

a) Name of the material;

- b) Net mass of the material;
- c) Manufacturer's name and its recognized trade-mark, if any;
- d) Batch number or lot number in code or otherwise; and
- e) Month and year of manufacture.

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 there under, and the products may be marked with the Standard Mark.

5.2.3 In addition the containers shall also be marked with the following information either printed on the label affixed to the containers or lithographed or stenciled thereon with indelible ink in a type size of not less than 50 mm.

'FOR INDUSTRIAL (NON-EDIBLE) USE ONLY'

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 548 (Part 1/Sec 1).

7 TESTS

7.1 Test shall be carried out as prescribed in IS 548 (Part 1/Sec 2), IS 548 (Part 2) and Annex B to <u>Annex D</u> of this standard. References to the relevant test methods and Annexes of the standard are given in col (5) of <u>Table 1</u>.

7.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

Sl No.	Characteristics	Requirements for		Method of Test	
		Castor Oleine	Castor Oleostearine		
(1)	(2)	(3)	(4)	(5)	
i)	Moisture and volatile matter, percent by mass, <i>Max</i>	0.25	0.25	IS 548 (Part 1/Sec 2)	
ii)	Colour in $\frac{1}{2}$ in cell on the Lovibond scale, expressed as (Y + 5 R), <i>Max</i>	35	35	IS 548 (Part 1/Sec 2)	
iii)	Acid value, Max	25	25	IS 548 (Part 1/Sec 2	
iv)	Hydroxyl value, Max	20	20	IS 548 (Part 1/Sec 2	
v)	Iodine value (Wijs)	41 to 90*	10 to 40*	IS 548 (Part 1/Sec 2	
vi)	Saponification value	180 to 190	180 to 190	IS 548 (Part 1/Sec 2	
vii)	Unsaponifiable matter, percent by mass, Max	1	1	IS 548 (Part 1/Sec 2	
viii)	Keto acids content, percent by mass, <i>Max</i>	5	5	Annex B	
ix)	Polymer content, percent by mass, Max	2	2	<u>Annex C</u>	
x)	Nickel content, parts per million, <i>Max</i>	10	50	Annex D	

Table 1 Requirements for Castor Oleine and Castor Oleostearine

(*Clauses* 4.2 and 7.1)

^{*}Actual value shall be as agreed to between the manufacturer and the supplier.

ANNEX A

(<u>Clause 2</u>)

LIST OF REFERRED STANDARDS

IS No.	Title	IS No.	Title
IS 286 : 2018	Methods of sampling and test	(Sec 2) : 2021	Physical and chemical tests
	for soaps (third revision)	(Part 2): 1976	Purity test (third revision)
IS 548	Methods of sampling and test for oils and fats:	IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
(Part 1)	Sampling, physical and chemical tests,	IS 7597 : 2001	Surface active agents — Glossary of terms (<i>first</i>
(Sec 1) : 2021	Sampling		revision)

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ANNEX B

[Clause 7.1 Table 1, S1 No. (viii)]

DETERMINATION OF KETO ACIDS CONTENT

B-1 REAGENTS

B-1.1 Ethyl Alcohol — 95 percent (v/v)

B-1.2 Bromophenol Blue Indicator — 0.1 percent solution

B-1.3 Standard Alcoholic Sodium Hydroxide Solution — 0.5 N

B-1.4 Dilute Hydrochloric Acid — 0.5 N

B-1.5 Hydroxylamine Hydrochloride Solution — 0.5 N

B-1.5.1 *Preparation of* 0.5 N *Hydroxylamine Hydrochloride Solution*

Dissolve 39.9 g of recrystallized hydroxylamine hydrochloride in 100 ml of distilled water; warm to a temperature of 65 °C on a steam bath to yield a clear solution. Add this solution slowly to 1 000 ml of 95 percent alcohol and mix thoroughly. Then add 50 ml of 0.1 percent solution of bromophenol blue indicator in 50 percent alcohol and sufficient 0.5 N alcoholic sodium hydroxide solution to a greenish shade; this usually requires about 20 ml to 25 ml of alkali. The proper degree of neutralization is obtained when the addition of 35 ml of the solution shows a distinct greenish shade which changes to a distinct yellow upon the addition of one drop of dilute hydrochloric acid. A stable solution of hydroxylamine hydrochloride is thus obtained which is approximately 0.5 N, an exact adjustment is not required.

B-2 PROCEDURE

Weigh accurately requisite amount of sample

(*see* Note below) into a 100 ml saponification flask and add 35 ml of hydroxylamine hydrochloride solution measured from a graduated cylinder. Permit the flask to stand at room temperature for 15 min and titrate the liberated hydrochloric acid with freshly standardized alcoholic sodium hydroxide solution. The titration is continued until the original greenish shade of the hydroxylamine solution is obtained. A second flask containing 35 ml of hydroxylamine hydrochloride solution may be used as a blank to assure a more accurate colour match.

 NOTE — Mass of the sample to be taken for analysis shall be as follows:

- a) 2.5 g for keto content up to 10 percent;
- b) 1.0 g for keto content from 11 percent to 25 percent; and
- c) 0.5 g for keto content above 25 percent.

B-3 CALCULATION

Calculate the percentage of keto acid present in the sample as follows:

Percentage of keto acids =
$$\frac{V \times m}{20 M_1}$$

where

- *V* = volume, in ml, of 0.5 N alcoholic sodium hydroxide solution used for neutralization;
- m = molecular mass of keto acids which can be taken as 298; and
- M_1 = mass, in g, of the sample taken for analysis.

ANNEX C

[*Clause* 7.1 *Table* 1, *S1 No.* (ix)]

DETERMINATION OF POLYMER CONTENT

C-1 REAGENTS

C-1.1 Solid Potassium Hydroxide

C-1.2 Glycerine — dynamite or C. P. grade

C-1.3 Concentrated Sulphuric Acid

C-1.4 Methyl Alcohol

C-2 PROCEDURE

C-2.1 Preparation of Fatty Acids

With the aid of heat, dissolve 40 g of solid potassium hydroxide in 200 g of glycerine. Bring the temperature of the mixture to 150 °C and add 100 g of oil or melted fat sample and reheat to 140 °C to 150 °C. Continue stirring until saponification is complete. Do not heat to above 150 °C. Cool slightly and add about 400 ml to 500 ml water, stir the mass well and heat until the soap is dissolved. Add carefully while stirring 100 ml of sulphuric acid (50 percent by mass). Boil until the fatty acids are completely melted and clear. Remove the aqueous layer by siphon, add fresh water and boil again till the fatty acids are clear and melted. Siphon the aqueous layer and repeat washings till the aqueous layer is neutral to methyl orange indicator. Pass the fatty acids through filter paper to dry them.

C-2.2 Preparation of Methyl Esters

Reflux the dried fatty acids as obtained in <u>C-2.1</u> for 2 h with about 4 times their mass of methyl alcohol containing 1 percent of concentrated sulphuric acid. Distil the unreacted methanol and wash the esters free of mineral acid.

C-2.3 Distillation

Weigh accurately about 50 g of the methyl esters as obtained in <u>C-2.2</u> in a 100 ml distillation flask. Distil the methyl esters at 210 °C under a vacuum of 1.5 mm of mercury. Weigh the residue left in the distillation flask.

C-3 CALCULATION

Calculate the polymer content in the sample as follows:

Polymer content, percent by mass = $\frac{M_1 \times 100}{M_2}$

where

- M_1 = mass, in g, of the residue left in the distillation flask; and
- M_2 = mass, in g, of methyl esters taken for distillation.

ANNEX D

[*Clause* 7.1 *Table* 1, *Sl No.* (x)]

DETERMINATION OF NICKEL

D-1 OUTLINE OF THE METHOD

The method is based on the isolation of metal from the soap and reaction between nickel in the oxidised form with dimethyl glyoxime forming a red colour, the intensity of which is proportional to the amount of nickel present in the sample.

D-2 APPARATUS — UV spectrophotometer

D-3 REAGENTS

D-3.1 Sodium Hydroxide — solid

D-3.2 Concentrated Hydrochloric Acid

D-3.3 Dimethyl Glyoxime Solution — 0.1 percent (v/v) in 95 percent ethyl alcohol

D-3.4 Saturated Bromine Water

D-3.5 Standard Nickel Sulphate Solution — containing 1 000 µg of nickel (Ni) per ml

D-3.6 Liquor Ammonia — relative density 0.9

D-4 PROCEDURE

D-4.1 Isolation of Metals from Castor Oleine and Castor Oleostearine

To 40 g of sample in a 500 ml beaker add 40 ml of caustic soda solution containing 8 g of sodium

hydroxide; mix well and heat on water bath until the saponification is complete. Add 200 ml hot water, heat until all the soap goes into solution. Add to this soap solution 40 ml of concentrated hydrochloric acid, stir and keep on steam bath until fatty acid layer separates. Add 20 g of paraffin wax, stir at intervals and allow to settle until phases are clear. Cool to room temperature.

Remove wax cake with rod, rinse with water and add rinsings to aqueous phase. Evaporate aqueous phase to about 60 ml by gentle boiling. Add 100 ml of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 ml. Cool and transfer to volumetric flask. Make up the volume to 100 ml. Take aliquot portions for metal estimation.

D-4.2 Determination of Nickel Content

Take 50 ml aliquot of the aqueous solution from the test solution in a 250 ml beaker. Evaporate the solution to about 15 ml by heating. Transfer the solution to a 50 ml glass stoppered volumetric flask using a small quantity of water for rinsing the solution from the beaker into the volumetric flask. Add to the flask 3 ml of saturated bromine water and allow to stand for one minute. Add ammonia liquor dropwise until excess bromine is destroyed as indicated by the disappearance of brown colour. Then add 5 ml of ammonia liquor in excess. If a precipitation occurs, filter the solution and wash the precipitate with water; combine the filtrate and the

washings and concentrate to a volume of a few ml and transfer to a 50 ml volumetric flask. Add 10 ml of dimethyl glyoxime solution, followed by 15 ml to 20 ml of 95 percent ethyl alcohol. Mix thoroughly and make up the volume and again mix thoroughly. Allow the solution to stand for 5 min to permit full development of colour and take the absorption/ transmittance reading at 445 nm.

Prepare and conduct blank determination simultaneously and similar in all respects. The transmittance of the blank should be 98 percent ± 1 percent. Determine the nickel content of the sample by reference to a concentration-transmittance graph prepared as follows.

D-4.3 Preparation of Concentration Transmittance Graph

Weigh accurately 2.261 7 g of nickel sulphate (99 percent $NiSO_{4.}6H_{2}O$) and dissolve in water in a

500 ml volumetric flask. Add 30 ml of concentrated hydrochloric acid and bring to volume. This solution contains 1 000 μ g of nickel per ml. Make appropriate dilutions of this solution and process this solution as in sample above. The dilutions should cover a range 0 μ g to 100 μ g. Finally plot a curve relating transmittance to micrograms of nickel.

D-4.4 Calculation

Nickel content, parts per million =
$$\frac{M_1 - M_2}{m}$$

where

- M_1 = micrograms of nickel present in sample;
- M_2 = micrograms of nickel present in blank; and
- m =mass, in grams, of the sample taken for the test.

ANNEX E

(Foreword)

COMMITTEE COMPOSITION

Soaps, Detergents and Surface Active Agents Sectional Committee, CHD 25

Organization	Representative(s)
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Central Pollution Control Board, New Delhi	Shri Dinabandhu Gouda Shri Vishal Gandhi (<i>Alternate</i>)
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Member Secretary Shri Virendra Singh Scientist 'D'/Joint Director (Chemical), BIS

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