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भारतीय मानक

रबड़ उद्योग के लिए वल्कनीकृत वनस्पति तेल
(फैक्टिस विशिष्ट) —

(पहला पुनरीक्षण)

Indian Standard

VULCANIZED VEGETABLE OILS (*FACTICE*)
FOR RUBBER INDUSTRY — SPECIFICATION

(*First Revision*)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 2

FOREWORD

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

This standard was first published in 1982 and is being revised with some changes to suit the requirements of the users, producers and also, considering the raw material position as existing in India. The petroleum ether extract test has been included to take care of castor oil based factices which dissolve completely in acetone and will not give the value of percentages of additives such as mineral oil.

Vulcanized vegetable oils, also known as FACTICE are produced using vegetable oils vulcanized with sulphur, sulphur chloride or any other suitable sulphur containing organic compound. It is used in the rubber products, both natural and synthetic as a processing aid.

Vulcanized vegetable oils (FACTICES) are graded according to their colour, namely, brown, golden and white. Brown and golded FACTICES based on castor oil are also resistant, and hence form an integral part of this specification. Considering the raw material availability and restrictions imposed on edible oils for use in industry, this specification will be based on the raw materials for factice, available in the country, namely, castor oil. White factices are generally made from mustard oil, rape seed oil, groundnut oil and similar oils. They are high cost factices meant for crassors and similar applications.

High ash content prescribed in Type 3 Grade 2 factices is due to white mineral fillers present in them for easy granulation and powdering. They are supplied in powder or granular forms.

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

VULCANIZED VEGETABLE OILS (*FACTICE*) FOR RUBBER INDUSTRY — SPECIFICATION

(*First Revision*)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and tests for vulcanized vegetable oils (*factice*) for use in rubber industry.

1.1.1 This standard covers vulcanized vegetable oils (*Factices*) for oil resistance and speciality applications also.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1977	Specification for water for general laboratory use (<i>first revision</i>)
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1

3 TYPES AND GRADES

3.1 Types

There shall be following three types of vulcanized vegetable oils depending on their colour:

Type 1 Brown vulcanized vegetable oils,

Type 2 Golden vulcanized vegetable oils, and

Type 3 White vulcanized vegetable oils.

3.2 Grades

Depending on physico-chemical requirements, brown (Type 1) and white vulcanized vegetable oils (Type 3) shall be of two grades and golden vulcanized vegetable oils (Type 2) shall be of one grade as indicated in Table 1.

4 REQUIREMENTS

4.1 Description

Type 1 vulcanized vegetable oils are usually available in lumps or granules form, and Types 2 and 3 vulcanized vegetable oils are available in granular or powder form. Each type shall have essentially good dispersibility with rubber. The material shall be free from foreign matter like wood, dust etc.

4.2 The material shall comply with the requirements given in Table 1 when tested according to procedure specified under col 8 of the Table.

Table 1 Requirements for Vulcanized Vegetable Oils
(*Clauses 3.2 and 4.2*)

Sl No.	Characteristic	Type 1		Type 2	Type 3		Method of Test, Ref to
		Grade 1	Grade 2		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Appearance	Brown coloured lump or granular	Brown coloured lump or granular	Golden yellow powder or granules	White powder	White powder	Visual
ii)	Relative density at 27/27°C	1.05 ± 0.05	1.05 ± 0.05	1.05 ± 0.05	1.05 ± 0.05	1.10 ± 0.05	4 of IS 7086 (Part 1) : 1973
iii)	Acetone extract or petroleum ether extract, percent by mass, <i>Max</i>	20	40	15	10	15	Annex A
iv)	Free sulphur, percent by mass, <i>Max</i>	1	1.5	1.0	0.5	0.5	Annex B
v)	Ash content, percent by mass, <i>Max</i>	0.5	0.5	1.0	2.0	20	Annex C
vi)	Unsaponifiable matter, percent by mass, <i>Max</i>	1.0	1.0	1.0	1.0	5	Annex D
vii)	Chlorine, percent by mass, <i>Max</i>	0.02	0.02	0.02	3	8	Annex E

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in polyethylene-lined hessian bags.

5.2 Marking

5.2.1 Each package shall be marked with:

- name of the material, type and grade,
- indication of the source of the manufacture,
- net mass of the material,
- month and year of manufacture, and
- batch or code number.

5.2.2 Each package may also be marked with the Standard Mark.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in 15 of IS 7086 (Part 1) : 1973.

6.2 Number of Tests

Test for all characteristics shall be conducted on composite sample.

6.3 Criteria for Conformity

The lot shall be considered as conforming to the specification if the composite sample satisfies each of the requirements.

7 TEST METHODS

7.1 Tests shall be conducted according to the methods prescribed in Annexes A to E and the relevant clause of IS 7086 (Part 1) : 1973 as given in col 8 of Table 1.

7.2 Unless specified otherwise 'pure chemicals' and distilled water (see IS 1070 : 1977) shall be employed in the tests.

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

ANNEX A

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

DETERMINATION OF ACETONE/PETROLEUM ETHER EXTRACT

A-1 REAGENTS

A-1.1 Acetone

Distilled over anhydrous potassium carbonate, boiling between 56 and 57°C.

A-1.2 Petroleum Ether

Having boiling point from 40 to 60°C.

A-2 PROCEDURE

A-2.1 Weigh approximately 2 g of sample in a filter paper. Fold the paper in a way that it fits in the extracting cup, and suspend the cup in a weighed extraction flask containing 50 to 75 ml of acetone/petroleum ether. Prior to the weighing of the extraction flask, it shall have been dried for two hours at 70 ± 5°C and cooled in a desiccator to the temperature of the balance.

A-2.2 Extract the sample continuously for 8 hours, heating at a rate such that time

required to fill and empty the siphon cup is between 2.5 and 3.5 minutes.

A-2.3 Evaporate the solvent over a steam bath, using a gentle current or filtered air to prevent boiling. Remove the flask from the steam bath just prior to the disappearance of the last traces of solvent to prevent loss of extract. Continue the passage of air through the flask for 10 minutes to remove the remaining solvent and dry the flask for two hours in a 70 ± 5°C air oven.

A-2.4 Cool in a desiccator to the room temperature and weigh.

A-3 CALCULATION

$$\text{Acetone extract/petroleum ether extract, percent by mass} = \frac{A}{B} \times 100$$

where

A = mass in g of the extract, and
B = mass in g of sample used,

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

DETERMINATION OF FREE SULPHUR

B-1 REAGENTS**B-1.1 Sodium Sulphite Solution** — 10 percent.**B-1.2 Formaline** — 40 percent formaldehyde solution.**B-1.3 Cadmium Chloride Solution** — 3 percent.**B-1.4 Strontium Chloride Solution** — 10 percent solution.**B-1.5 Iodine Solution** — 0.1 N.**B-2 PROCEDURE**

Extract 5 g of the material with acetone/petroleum ether as in Annex A. Boil the acetone extract/petroleum ether extract under reflux for two hours with 30 ml of 10 percent

sodium sulphite solution with the addition of 5 ml of ethanol. After cooling add 10 ml of formaline to remove excess sulphite, then add 10 ml of strontium chloride solution as well as 5 ml cadmium chloride solution to precipitate free fatty acids. After standing for 30 minutes filter the whole mass through cotton wool and react the filtrate with 15 ml of 20 percent acetic acid. Titrate the thiosulphate so formed with 0.1 N iodine solution using starch as indicator.

B-3 CALCULATION

$$\text{Free sulphur, percent by mass} = \frac{0.0032 V}{M} \times 100$$

where

 V = ml of 0.1 N iodine solution; and M = mass in g of the sample taken.

ANNEX C
[Table 1, Sl No. (v)]

DETERMINATION OF ASH CONTENT

C-1 APPARATUS**C-1.1 Silica or Porcelain Crucible****C-1.2 Muffle Furnace****C-2 PROCEDURE**

Take a known mass of the material (previously dried at 105°C) in a silica crucible. Ignite the same in a muffle furnace at 550°C ± 25°C for 16 h. Cool the content of the crucible in a

desiccator after ignition. Weigh the crucible and its contents.

C-3 CALCULATION

$$\text{Ash content, percent by mass,} = \frac{C - A}{B} \times 100$$

where

 A = mass in g of the empty crucible, B = mass in g of the specimen taken, and C = mass in g of the crucible and the ash.

ANNEX D
[Clause 4.2, Table 1, Sl No. (vi)]

DETERMINATION OF UNSAPONIFIABLE MATTER

D-1 REAGENTS**D-1.1 Alcoholic Potash Solution**

Prepare a 2 N alcoholic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows:

Dissolve 1.5 g of silver nitrate in 3 ml of water, and add it to one litre of alcohol. Dissolve 3 g of potassium hydroxide in the

smallest amount of hot water, cool, add it to the silver nitrate solution and shake thoroughly. Allow the solution to stand for 24 hours, filter and distil.

D-1.2 Petroleum Ether

Having boiling point from 40 to 60°C.

D-2 PROCEDURE

D-2.1 Boil acetone extract of 5 g of material under reflux with 30 ml of 2 N alcoholic

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potassium hydroxide for two hours. Add 5 ml of water and boil for a further 20 minutes.

D-2.2 Rinse the soap solution so formed with 50 percent alcohol in a separating funnel and shake at least 3 times with 50 ml petroleum ether. Break up the emulsion by the addition of a small amount of alcohol. Wash the combined petroleum ether extract once with alcohol which is slightly alkaline to phenolphthalein, and then with 25 ml of 50 percent alcohol until this is no longer red to phenolphthalein when diluted with 2-3 volumes of water. After

distilling off the petroleum ether, the residue is heated on a water bath under vacuum to constant mass.

D-3 CALCULATION

$$\text{Unsaponified matter, percent by mass} = \frac{A}{B} \times 100$$

where

A = mass in g of the residue, and

B = mass in g of the sample taken.

ANNEX E

[*Clause 4.2, Table 1, Sl No. (vii)*]

DETERMINATION OF CHLORINE

E-1 REAGENTS

E-1.1 Silver Nitrate Solution

E-1.2 Ferric Nitrate

E-1.3 Potassium Hydroxide

E-1.4 Sodium Peroxide

E-1.5 Standard Thiocyanate Solution

E-2 PROCEDURE

Heat 0.2 to 0.3 g of the material with a mixture of 10 g of powdered potassium hydroxide and 5 g of sodium peroxide in a nickel crucible.

Heat the mixture gradually. Raise the temperature till the mass is fused and melted. Cool the melt and extract with distilled water. Precipitate the chloride as silver chloride with an excess of standard silver nitrate. Determine the excess silver nitrate by titration with thiocyanate solution using ferric nitrate as the indicator. A little nitrobenzene may be used to depress the solubility of silver chloride.

E-3 CALCULATION

Calculate the amount of chlorine from the formula:

1 ml of normal silver nitrate solution = 0.035 46 g of chlorine.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.