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Indian Standard

SPECIFICATION FOR ETHYL ACETATE FOR COSMETIC INDUSTRY

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Indian Standard

SPECIFICATION FOR ETHYL ACETATE FOR COSMETIC INDUSTRY

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Indian Standard

SPECIFICATION FOR ETHYL ACETATE FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 September 1982, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Ethyl acetate is used in the manufacture of cosmetic preparations like nail lacquers. It is also used as an industrial solvent mainly in the paint and lacquer industry. Although a separate standard 1S: 229-1972* has been published to cover non-cosmetic use of the material it was considered necessary to cover the specific requirements of the cosmetic industry through formulation of this standard.

0.3 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⁺. The 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

1.1 This standard prescribes the requirements and methods of sampling and testing for ethyl acetate for cosmetic industry.

2. REQUIREMENTS

2.1 Description — The material shall be clear, colourless liquid with a characteristic odour and free from visible impurities. It shall consist essentially of the ethyl ester of acetic acid, $CH_8COOC_2H_5$.

2.2 Odour — The material shall have the characteristic odour of ethyl acetate and shall leave no residual odour after evaporation from filter paper.

2.3 The material shall also comply with the requirements given in Table 1, when tested as given in col 4 & 5 of the table.

^{*}Specification for ethyl acetate.

⁺Rules for rounding off numerical values (revised).

Sl No.	CHARAGTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN	
			Other Indian Standard	Appen- dix
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 27°/27°C	0.893 to 0.898		A-1
ii)	Residue on evaporation, per- cent by mass, <i>Max</i>	0.01		A-2
i) A	cidity as acetic acid, per- cent by mass, <i>Max</i>	0.01		A-3
iv)	Ester content as ethyl ace- tate, percent by mass, Max	99	_	A-4
v)	Distillation range:			
	 a) Initial boiling point at 101.3 KN/m² Hg, Min b) Dry point at 101.3 KN/m² Hg, Max 	76°C } 78·5°C }	IS : 229-1972*	A-5
vi)	Water content, percent by mass, Max	0.1	IS : 2362-1973†	_

TABLE 1 REQUIREMENTS FOR ETHYL ACETATE FOR COSMETIC INDUSTRY

(Clause 2.2)

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in tightly-closed containers made of galvanized iron or of any other suitable material, subject to the provisions of laws in force in the country from time to time.

3.1.1 All containers in which the material is stored shall be clean, dry and leak-proof. It shall be kept in a cool place.

3.2 Marking - Each container shall be marked with the following:

- a) Name of the material;
- b) Name of the manufacturer, initials or his trade-mark, if any;
- c) Tare, gross and net mass;
- d) Month and year of manufacture; and
- e) Batch or code number; and
- f) Any other information as required by the statutory authorities.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI 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 ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Preparation of Test Samples — Representative samples of the material shall be drawn as prescribed in Appendix B.

4.2 Number of Tests — Tests for the determination of relative density and ester content shall be conducted on individual samples. Tests for the remaining characteristics shall be carried out on the composite sample.

4.3 Criteria for Conformity — For the characteristics tested on individual samples, all test results shall individually satisfy the corresponding requirements and for the other characteristics the results on the composite sample shall meet the prescribed requirements if the lot is to be accepted under this specification.

5. TEST METHODS

5.1 Tests shall be carried out by the methods specified in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in tests.

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

*Specification for water for general laboratory use (second revision).

APPENDIX A

(*Table* 1, *clause* 2.2)

METHODS OF TEST FOR ETHYL ACETATE

A-1. DETERMINATION OF RELATIVE DENSITY

A-1.1 Apparatus

A-1.1.1 Relative Density Bottle — 50 ml capacity, of Regnault type.

A-1.2 Procedure — Clean, dry and weigh accurately the relative density bottle and the stopper. Fill the bottle with water and immerse it up to the neck in a constant temperature bath at 27.0 ± 0.5 °C and keep immersed at this temperature for 20 minutes. Wipe off the water with a bit of filter paper till the level reaches the graduation mark and weigh again. Empty the bottle, clean and dry. Repeat the operation with the material at 27°C.

A-1.3 Calculation

Relative density at
$$27^{\circ}/27^{\circ}C = \frac{A-B}{C-B}$$

where

A = mass in g of the relative density bottle with the materialat 27°C;

B = mass in g of the relative density bottle; and

C = mass in g of the relative density bottle with water at 27°C.

A-2. DETERMINATION OF RESIDUE ON EVAPORATION

A-2.1 Procedure — Evaporate 100 ml of the material to dryness in a weighed platinum or silica or borosilicate glass basin on a boiling waterbath. Dry the residue in an oven at a temperature of $100 \pm 2^{\circ}$ C to constant mass. Cool in a desiccator and weigh.

A-2.2 Calculation

Residue on evaporation, percent by mass = $\frac{M_1}{d}$

where

 $M_1 = \text{mass in g of the residue; and}$ d = relative density of the sample.

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A-3. DETERMINATION OF ACIDITY

A-3.1 Reagents

A-3.1.1 Rectified Spirit - 95 percent (see IS: 323-1959*).

A-3.1.2 Standard Sodium Hydroxide Solution - 0.1 N.

A-3.1.3 Phenolphthalein Indicator — Dissolve 0.5 g of the phenolphthalein in 100-ml of 95 percent rectified spirit. Make the solution faintly pink by adding dilute sodium hydroxide solution.

A-3.2 Procedure — Take 50 ml of rectified spirit, add 0.5 ml of phenolphthalein indicator and neutralize with sodium hydroxide solution. Add 50 ml of the sample. Titrate the mixture immediately with the standard sodium hydroxide solution until the first pink colour persists for at least 10 seconds.

A-3.3 Calculation

Acidity (as acetic acid), percent by mass = $\frac{6 V N}{50 d}$

where

V = volume in ml of standard sodium hydroxide solution;

 \mathcal{N} = normality of standard sodium hydroxide solution; and

d = relative density of ethyl acetate at the test temperature.

A-4. DETERMINATION OF ESTER CONTENT

A-4.1 Reagents

A-4.1.1 Standard Alcoholic Sodium Hydroxide Solution - 0.5 N.

A-4.1.2 Standard Hydrochloric Acid - 0.5 N.

A-4.1.3 Phenolphthalein Indicator — same as in A-3.1.3.

A-4.2 Procedure — Weigh accurately 1.0 to 1.5 g of the material in a small tared tube and transfer the tube with its contents to a flask containing 50 ml of standard alcoholic sodium hydroxide solution. Fit a water-cooled reflux condenser to the flask and heat it for one hour over a boiling water-bath. Withdraw the flask, still carrying its condenser, and immerse it in cold water. When cool, rinse the inside of the condenser with distilled water and collect the flask and wash the ground glass joint with distilled water and collect the washings in the flask in which the reaction has been carried out. Add 0.5 ml of phenolphthalein indicator and titrate the mixture immediately with the standard hydrochloric acid solution until the pink colour is just discharged.

^{*}Specification for rectified spirit (revised).

A-4.2.1 Carry out a blank determination, using all the reagents excepting the material, under similar conditions and at the same time.

A-4.3 Calculation

Ester content (as ethyl acetate), = $\frac{8.81 (V_2 - V_1) N}{M}$

where

- V_2 = volume in ml of standard hydrochloric acid required in the blank determination;
- V_1 = volume in ml of standard hydrochloric acid required in the test with the material;

 \mathcal{N} = normality of standard hydrochloric acid; and

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

APPENDIX B

(Clause 4.1)

SAMPLING OF ETHYL ACETATE FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected area with good ventilation.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for sample from adventitious contamination.

B-1.4 To draw a representative sample the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking, stirring, rolling or by any other suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass or metal containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

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B-1.7 Each sample container shall be sealed air-tight with a suitable stopper, after filling and marked with the manufacturer's name or trademark, grade of the material, the month and year of manufacture of the material, the batch number (if available) and other details of sampling, such as the date of sampling, sampler's name, etc.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SAMPLING INSTRUMENT

B-2.1 It is made of thick glass or metal on which the material has no action and is 20 to 40 mm in diameter and 400 to 800 mm in length (see Fig. 1).

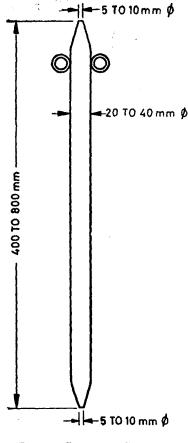


FIG. 1 SAMPLING TUBE

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The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples, the apparatus is first closed at the top with thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

B-2.1.1 For small containers, the size of the sampling tube may be altered suitably.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment, all the containers of the same grade, size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different grades or of different batches of manufacture or of different sizes of containers, the containers belonging to the same grade, batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose from a lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2	SCALE OF SAMPLING
LOT SIZE	No. of Containers to be Selected
(<i>N</i>)	(n)
(1)	(2)
U p to 15	3
16 ,, 50	4
51 ,, 100	5
101 ,, 150	6
151 ,, 300	7
301 and above	8

B-3.3 These containers shall be selected as random from the lot and in order to ensure randomness of selection a random number table (*see* IS: 4905-1968*) shall be used. In case such a table is not available, the following procedure may be used:

Starting from any container in the lot count them as 1, 2,.... up to r and so on in one order, where r is the integral part of \mathcal{N}/n (\mathcal{N} being the lot size and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to give a sample for tests.

*Methods for random sampling.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected according to **B-3.3** a representative portion of the material approximately 800 ml in volume (*see* Note) shall be drawn with the help of sampling tube. Out of these portions equal quantity of material shall be taken and mixed thoroughly to form a composite sample not less than 1 500 ml in volume. The composite sample shall be divided into three equal parts one for the purchaser, another for the supplier and the third to be used as a referee sample. These composite samples shall be transferred to containers of 600 ml capacity and shall be sealed and marked with full identification particulars given in **B-1.7**.

Note — This amount may be taken out in one or more operations of sampling tube by filling it partially or completely as occasion demands.

B-4.2 The remaining portions representing each of the individual containers shall be transferred to separate sample containers and these are termed individual samples. These sample containers, after filling, shall be sealed and marked with full identification particulars.

B-4.3 The referee sample prepared under **B-4.1** shall bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

INDIAN STANDARDS

ON

COSMETIC RAW MATERIALS

IS:

263-1977	Boric acid (third revision)
918-1968	Calcium carbonate, precipitated, for cosmetic industry (first revision)
1462-1977	Talc for cosmetic industry (second revision)
1463-1977	Kaolin for cosmetic industry (second revision)
1767-1980	Dicalcium phosphate for dentifrice (second revision)
2519-1977	Calcium stearate for cosmetic industry (first revision)
2 520-19 77	Zinc stearate for cosmetic industry (first revision)
2521-1977	Magnesium stearate for cosmetic industry (first revision)
2528-1977	Magnesium carbonate for cosmetic industry (first revision)
2 529-1977	Magnesium oxide for cosmetic industry (first revision)
2850-1977	Zinc oxide for cosmetic industry (first revision)
2851-1978	Titanium dioxide for cosmetic industry (first revision)
3 986-197 3	Sodium lauryl sulphate for cosmetic industry (first revision)
3987-1977	Sorbitol solution (70 percent) (first revision)
4028- 1977	Beeswax, bleached, for cosmetic industry (first revision)
42 36-1977	Glyceryl monostearate for cosmetic industry (first revision)
4652-1980	Ethyl p-hydroxybenzoate for cosmetic industry (first revision)
4653-1977	Methyl p-hydroxybenzoate for cosmetic industry (first revision)
4887-1980	Petroleum jelly for cosmetic industry (first revision)
5340-1981	Lanolin, anhydrous, for cosmetic industry (first revision)
5356-1977	iso-propyl myristate for cosmetic industry (first revision)
63 33- 1977	Propyl p-hydroxybenzoate for cosmetic industry (first revision)
633 4- 1980	Butyl p-hydroxybenzoate for cosmetic industry (first revision)
7101-1973	Coconut diethanolamide
7299-1974	Mineral oil for cosmetic industry
9601-1980	Sodium silicate for cosmetic industry
9681-1980	Stearic acid for cosmetic industry
9830-1981	Water soluble sodium carboxy methyl cellulose for cosmetic industry
9831-1981	Sodium hydroxide for cosmetic industry