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Draft Indian Standard
ETHYL ACRYLATE — SPECIFICATION
(First Revision of IS 14708)
(ICS 71.080.70)

Organic Chemicals, Alcohols and Allied
Products Sectional Committee, PCD 9

Last date for comments
1 February 2024

FOREWORD

(Formal clauses to be added later)

Ethyl acrylate is produced by the esterification of acrylic acid or by acrylonitrile. It has versatile use in the manufacturing of leather auxiliaries, paper coating etc. It also finds its uses as a textile sizing agent and kerosene substitute water-based thickener.

Ethyl acrylate is a flammable liquid. It's vapours can form explosive mixtures with air and can cause irritation. It should be kept away from heat sources and chemicals that can cause its polymerization violently evolving considerable heat.

The standard was originally published in 1999. The standard was first published by taking considerable assistance from the following standards:

- ASTM D 1364-1980 Test method for water in volatile solvents
- ASTM D 1613-1985 Test method for acidity in volatile solvents
- ASTM D 3125-1983 Test method for MEHQ in colourless monomeric acrylate esters and acrylic acids
- ASTM D 3362-1984 Test method for purity of acrylate esters by gas chromatography
- ASTM D 3548-1993 Specification for ethyl acrylate

In this (*first*) revision, requirement of colour has been modified to 10 Pt-Co from 20 Pt-Co and moisture content to 0.05 percent from 0.2 percent, as the limit present was high as compared to the material available in market. Also, the requirement of inhibitor has been modified to 10-20 ppm, as inhibitor is a must to avoid polymerization and thus minimum amount of inhibitor is incorporated. Further, instrumental methods for determination of moisture content, colour and inhibitor content have been incorporated.

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.

1 SCOPE

This standard prescribes the requirements, the methods of sampling and test for ethyl acrylate.

2 REFERENCES

The standards listed below are necessary adjuncts to this standard. The standards contain provisions which through reference 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 revisions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent water grade — Specification (<i>fourth revision</i>)
IS 1448	Methods of Test for Petroleum and its Products:
(Part 178) : 2020 / ISO 6271 : 2015	Clear Liquids - Estimation of Colour by the platinum Cobalt Colour Scale
(Part 182) : 2020 / ISO 12937 : 2000	Petroleum Products - Determination of Water - Coulometric Karl Fischer Titration Method
IS 2362 : 1993	Determination of water by Karl Fischer method (<i>second revision</i>)
IS 4905 : 2015 / ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 8768 : 2000	Method of measurement of colour in liquid chemical products Platinum cobalt scale (<i>Second revision</i>)

3 REQUIREMENTS

3.1 The material shall also comply with the requirements given in Table 1, when tested according to the methods referred in col (4) and col (5) of Table 1.

3.2 Quality of Reagents

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

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the supplier and the purchaser.

4.2 Marking

4.2.1 The material shall be legibly marked with the following information:

- a) Name of the material;
- b) Name of the manufacturer and its trade mark, if any;
- c) Month and year of manufacture;
- d) Batch number, in code or otherwise;
- e) Net mass of the material; and
- f) Any other statutory requirement.

Table 1 Requirements for Ethyl Acrylates
(Clause 3.1 and D-5.1)

SI No.	Characteristic	Requirement	Method of test , Ref to	
			Annex (4)	Indian Standards (5)
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, <i>Min</i>	99.5	A	—
ii)	Acidity (as acrylic acid), percent by mass, <i>Max</i>	00.01	B	—
iii)	Colour, Pt-Co scale, <i>Max</i>	10.00	—	IS 8768/IS 1448 (Part 178)
iv)	Water, percent by mass, <i>Max</i>	00.05	—	IS 2362/IS 1448 (Part 182)
v)	Inhibitors (as monomethyl ether hydroquinone), ppm <i>Max</i>	10-20	C	—

4.2.2 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 thereunder, and the products may be marked with the standard mark.

5 SAMPLING

The method of drawing representative samples shall be as prescribed in Annex D.

ANNEX A
[Table 1, Sl No. (i)]
DETERMINATION OF ASSAY

A-1 GENERAL

This test method covers the determination of the purity of ethyl acrylate by gas chromatography and, in addition, provides a means for measuring certain impurities such as alcohols and other esters. Water and acidity are measured by other methods and the results are used to normalize the chromatographic values.

A-2 APPARATUS

A-2.1 Gas Chromatograph

A-2.1.1 Any suitable gas liquid chromatographic instrument with a flame ionization detector may be used with following accessories and typical operating conditions:

Column length	6 m (20 ft)
Internal Diameter	6.4 mm (1/4 in)
Syringe	2 μ l
Sample size	1-2 μ l
Recorder	A recording potentiometer with a full scale deflection of 1 mv, full scale response time of 2 set or less
Carrier gas	Helium of purity 99.95 mol percent or nitrogen gas
Column material	Copper/stainless steel/ aluminium
Liquid phase	Polyalkylene glycol (PAG) or equivalent phase capable of achieving desired separation of various components of interest
Solid Support	Chromosorb P-acid washed 45-60 mesh size
Solvent	Methylene chloride, reagent grade

A-2.2 Instrument Conditions

Temperature, °C	
Column, Isothermal	100
Injection Port	200

Detector Block	260
Carrier Gas	Helium
Flow rate, ml/min	6.0
Typical retention time, min	17.4

NOTE — The above gas chromatographic conditions are suggestive. However, any GC having different columns (packed/capillary having different length/diameter/film thickness) and different carrier gas (He, H₂ or N₂), with different calibration technique (internal standard, external standard, area normalization) may be used provided standardization/ calibrations are done after setting up chromatographic conditions for required resolution

A-3 STANDARDS FOR CALIBRATIONS AND IDENTIFICATION

Standard samples of all components present are needed for identification by retention time and for calibration of quantitative measurement.

A-4 PROCEDURE

Introduce a representative specimen into the chromatograph using sufficient specimen to ensure a minimum 10 percent recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting of instrument. Using the same conditions as for component identification and standardization, record the peaks of all components at attenuation setting that provide maximum peak heights.

A-5 CALCULATION

A-5.1 Measure the area of all peaks and multiply each area by the appropriate detector response factors to correct the difference in response to the components. Calculate the weight percent composition by dividing the individual corrected component areas by the total corrected area. Make corrections to account for water and acidity present.

A-5.2 Calculate the percent by mass of each component as follows:

$$\text{Mass, percent} = \frac{A \times (100 - C)}{B}$$

where,

A = corrected peak response,

B = sum of corrected peak responses, and

C = sum of water and acidity.

A-6 PRECISION AND BIAS

A-6.1 Repeatability

Two results, should be considered suspect if they differ by more than 0.06 percent.

A-6.2 Reproducibility

Two results should be considered suspect if they differ by more than 0.27 percent.

ANNEX B [Table 1, SI No. (ii)] DETERMINATION OF ACIDITY

B-1 GENERAL

This test method covers the determination of total acidity as acrylic acid, in concentrations below 0.05 percent in ethyl acrylate. The specimen is mixed with either an equal volume of water or an equal volume of alcohol and titrated with aqueous sodium hydroxide solution to the phenolphthalein end point.

B-2 APPARATUS

B-2.1 Burette, 10 ml, graduated in 0.05 ml.

B-2.2 Erlenmeyer Flask, 250 ml.

B-3 REAGENTS

B-3.1 Alcohols, refined ethyl or iso-propyl.

B-3.2 Phenolphthalein Indicator Solution, 10 g/l

Dissolve 1 g of phenolphthalein in ethyl or isopropyl alcohol and dilute to 100 ml with the alcohol.

NOTE — Bromophenol blue indicator may also be used.

B-4 PROCEDURE

Measure into a 250 ml conical flask 50 ml of water, if the sample is water soluble, or 50 ml of alcohol, if the sample is not completely water soluble. Add 0.5 ml of phenolphthalein indicator solution. Titrate with water/ alcohol with 0.05 N sodium hydroxide (NaOH) solution to the first pink colour. Pipette 50 ml of the sample into the flask. Titrate with the 0.05 N sodium hydroxide (NaOH) solution to the same first pink colour originally obtained.

B-5 CALCULATION

$$\text{Acidity as acrylic acid, percent by mass} = \frac{V \times N \times 0.144}{D}$$

where

v = sodium hydroxide (NaOH) solution required for titration of the sample, in ml;

N = normality of sodium hydroxide (NaOH); and

D = specific gravity of the specimen at the test temperature.

B-6 PRECISION AND BIAS

Repeatability 0.000 8 percent

Reproducibility 0.001 4 percent

NOTE — Auto Titrator may also be used for determination of acidity.

ANNEX C

[Table 1, Sl No. (v)]

DETERMINATION OF INHIBITORS

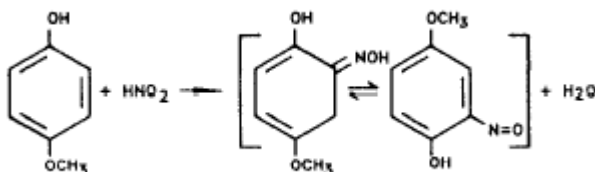
C-1 GENERAL

Two methods namely, Method A for determination of inhibitor content by spectrophotometer and Method B for determination of inhibitor content by gas chromatography have been prescribed. In case of dispute, Method B shall be the referee method.

C-2 METHOD A

C-2.1 General

This test method covers the determination of monomethyl ether of hydroquinone (MEHQ) in ethyl acrylate. It is applicable to the determination of MEHQ in the concentration range from 0 to 1 200 parts per million. MEHQ reacts with nitrous acid (sodium nitrite in acidic media) to form the nitroso derivative which equilibrates between two structures as given below.



The yellow colour of the nitroso compound is measured spectrophotometrically at a wavelength of 420 nm.

C-2.2 Apparatus

C-2.2.1 *Spectrophotometer*, with borosilicate glass cells for determining absorbance at 420 nm.

C-2.2.2 *Volumetric Flasks*, 50 and 100 ml capacity.

C-2.2.3 *Measuring Pipette*, 5 and 10 ml capacity.

C-2.3 Reagents

C-2.3.1 *Acetic Acid*, glacial.

C-2.3.2 *Monomethyl Ether of Hydroquinone (MEHQ) (4-Methoxy Phenol)*

C-2.3.3 *Sodium Nitrite Solution*

Dissolve 2 g of sodium nitrite in water and dilute to 100 ml.

C-2.4 Calibration

C-2.4.1 Weigh 0.10 g of MEHQ to the nearest 0.1 mg into a 100 ml volumetric flask containing approximately 50 ml of glacial acetic acid. Mix well until solution is complete then dilute to the mark with glacial acetic acid. Prepare a series of standards by pipetting 1, 2, 4, 6 and 10 ml portions of the MEHQ solution into respective 50 ml volumetric flasks. Dilute each flask to the mark with glacial acetic acid and mix well. A 10 ml aliquot of each of these standards contains 200, 400, 800, 1 200 and 2 000 µg of MEHQ respectively.

C-2.4.2 Determine the absorbance each of these standards by pipetting 10 ml aliquots into 50 ml volumetric flasks containing 20 ml of glacial acetic acid. To each flask add 1 ml of 2 percent sodium nitrite (NaNO₂), solution and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min. With cells appropriate to the instrument, determine the absorbance at 420 nm using acetic acid as the blank. Construct a calibration curve on rectangular coordinate graph paper by plotting the absorbances of the standards at 420 nm against the micrograms of MEHQ.

C-2.5 Procedure

C-2.5.1 Perform analysis in duplicate and carry a blank through the analysis using 49 ml of glacial acetic acid in place of the specimen solution. Weigh the appropriate amount of specimen into a 50 ml volumetric flask containing 20 ml of glacial acetic acid.

C-2.5.2 Add 1 ml of 2 percent sodium nitrite (NaNO₂), solution to the specimen and dilute to the mark with glacial acetic acid Mix well and allow to stand for 10 min.

C-2.5.3 Using the procedure followed for the calibration, determine the absorbance of the solution at 420 nm with the blank solution in the reference position. From the calibration curve, determine the micrograms of MEHQ corresponding to the absorbance obtained.

C-2.6 Calculation

Calculate the concentration of MEHQ in ppm as follows:

$$\text{MEHQ, ppm} = \frac{M}{S}$$

where

M = amount of MEHQ from calibration curve, in µg; and

S = weight of specimen used in the test, in g.

C-2.7 PRECISION

C-2.7.1 Repeatability

Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 4.0 percent relative.

C-2.7.2 Reproducibility

Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 15 percent relative.

C-3 METHOD B

C-3.1 Apparatus

C-3.1.1 Gas Chromatograph

C-3.1.1.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split/spitless injector and a suitable electronic integrator/software, capable of operating at the conditions listed below, may be used:

- Column : Stationary Phase of (5 percent phenyl)-methylpolysiloxane with length 30 m; internal diameter 0.32 mm and film thickness 1.0 μ m or equivalent
- Sample size : 1 μ l
- Flow
 - Carrier gas : Helium at constant flow of 1 ml/min or Nitrogen gas
 - Air flow rate : 450 ml/min
 - Hydrogen flow rate : 40 ml/min
 - Make-up gas : Nitrogen or Helium at constant flow rate of 30 ml/min
- Split ratio : 100:1

- Run time : 39 min

C-3.1.1.2 Temperature Programme of Oven, Detector and Injector:

<i>Injector Temperature, °C</i>	<i>Detector Temperature, °C</i>	<i>Temperature, °C</i>	<i>Oven Hold Time, min</i>	<i>Ramp Rate, °C/min</i>
250	300	50	5	—
		110	—	4

NOTE — The above gas chromatographic conditions are suggestive. However, any GC having different columns (packed/capillary having different length/diameter/film thickness) and different carrier gas (He, H₂ or N₂), with different calibration technique (internal standard, external standard, area normalization) may be used provided standardization/ calibrations are done after setting up chromatographic conditions for required resolution.

C-3.1.2 Analytical Balance

C-3.1.3 Vial with screw cap, 30 ml.

C-3.1.4 Autosampler, 2 ml

NOTE — Syringe may also be used for injecting the sample.

C-3.2 Reagents

C-3.2.1 Ethyl Acrylate, purity of 99.5 percent, minimum

C-3.2.2 Ethyl Acetate, purity of 99.5 percent, minimum

C-3.2.3 Monomethyl ether of hydroquinone (MEHQ, 4-methoxyphenol), purity of 99 percent, minimum

C-3.3 Procedure

Inject 1 µl aliquot of the sample manually by syringe or by automatic sampler into the column and obtain the chromatograph. Determine the mass concentration of all components by area normalization method.

C-3.4 Calculation

$$\text{MEHQ content, percent by mass} = \frac{R_{\text{MEHQ}} \times A_{\text{MEHQ}}}{\sum(R_i \times A_i)}$$

where

R_{MEHQ} = relative response factor of MEHQ;

R_i = relative response factor of each component;

A_{MEHQ} = peak area of MEHQ;

A_i = peak area of each component; and

$\sum(R_i \times A_i)$ = sum of product of each peak area times its relative response factor

C-3.5 Report

Report inhibitors (as monomethyl ether hydroquinone) content in ppm.

ANNEX D
(Clause 5)
SAMPLING OF ETHYL ACRYLATE

D-1 GENERAL

D-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

D-1.2 Sampling instrument shall be clean and dry.

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

D-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

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

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

D-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture and other important particulars of the consignment.

D-1.8 Samples shall be stored in a cool and dry place.

D-2 SCALE OF SAMPLING

D-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

D-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately.

D-2.3 The number of container (n) to be selected for drawing the samples shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be selected for Sampling
(Clause D-2.3)

SI No.	Lot Size	No. of Containers to be Selected
(1)	(N) (2)	(n) (3)
i)	Up to 25	2
ii)	26 to 50	3
iii)	51 to 100	5

iv)	101 to 300	7
v)	301 and above	10

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

D-2.4 These containers shall be selected at random from the lot and to ensure the randomness of selection, procedure given in IS 4905 may be followed.

D-3 TEST SAMPLE AND REFEREE SAMPLE

D-3.1 From each of the container selected as in **D-2.3**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

D-3.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

D-3.3 From the samples (*see D-3.2*) representing different containers selected in **D-2.3**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out testing for the characteristics specified. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

D-3.4 The referee samples consisting of a composite sample shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

D-4 TESTS

D-4.1 Tests for all the characteristics shall be conducted on the composite sample.

D-5 CRITERIA FOR CONFORMITY

D-5.1 For declaring the conformity of a lot to the requirements of all other characteristics (*see D-4.1*) tested on the composite sample, the test results for the characteristics shall satisfy the relevant requirements given in Table 1.