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Draft Indian Standard

***n*-BUTYL ACRYLATE — SPECIFICATION**

(First Revision of IS 14709)

(ICS 71.080.70)

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

Last date for comments
6 January 2024

FOREWORD

(Formal clauses to be added later)

n-Butyl acrylate is produced by the esterification of acrylic acid or by acrylonitrile.

n-Butyl acrylate has various industrial usage. It is used in BOPP tape adhesives and pressure sensitive adhesives. It also finds use in the manufacturing of paint emulsions, textile binders and auxiliaries. It is also used as leather binder, construction chemical and in paper coatings.

The standard was originally published in 1999 by taking considerable assistance from the following ASTM test methods:

- 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 3547-1981 Specification for *n*-butyl acrylate

In this (*first*) revision, an alternate method for determination of water has been introduced. Further, amendment no. 1 and 2 have also 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 *n*-butyl acrylate.

2 REFERENCES

The standards listed below contain provisions, which through reference in this text constitute the 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 editions 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 grade water— Specification (<i>fourth revision</i>)
IS 1448 (Part 182) : 2020 / ISO 12937 : 2000	Methods of Test for Petroleum and its Products: Part 182 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

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 to enable the lot of manufacture to be traced back from records; and
- e) Net mass of the material and
- f) Any other statutory requirement

Table 1 Requirements for *n*-Butyl Acrylate
(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>	99.0	A	—
ii)	Acidity (as acrylic acid), percent by mass, <i>Max</i>	00.01	B	—
iii)	Colour, Pt-Co scale, <i>Max</i>	20.00	—	IS 8768
iv)	Water, percent by mass, <i>Max</i>	00.15	—	IS 2362/IS 1448 (Part 182)
v)	Inhibitors (as butyl etherhydroquinone), <i>Max</i>	120	A ¹ /C	—

¹) In case of dispute, Annex A shall be the referee method for determination of inhibitors.

4.2.1 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 SAMPLING

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

ANNEX A

[Table 1, SI No. (i)]

DETERMINATION OF ASSAY

A-1 GENERAL

This test method covers the determination of the purity of *n*-butyl 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-2.2.1 Any gas chromatograph equipped with a flame ionization detector (FID), a suitable electronic integrator/software, capable of operating at the conditions listed below, may be used:

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 s or less
Carrier gas	Helium of purity 99.95 mol percent or Nitrogen gas
Column material	Copper/Stainless steel/ Aluminum
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-2.2.2 Instrument Conditions

Temperature, °C	
Column, Isothermal	140
Injection Port	200
Detector Block	260
Carrier Gas	Helium
Flow rate, ml/min	55
Typical retention time, min	19.4

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 mass 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 mass percent of each component as follows:

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

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, Sl 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 *n*-butyl 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 iso-propyl alcohol and dilute it to 100 ml with the alcohol.

NOTE — Bromophenol blue indicator may also be used.

B-3.3 Sodium Hydroxide Standard solution, 0.05 N

NOTE — Potassium hydroxide solution may also be used as an alternate to sodium hydroxide.

B-4 PROCEDURE

Into a 250 ml conical flask, take 50 ml of water, if the sample is water soluble or 50 ml of alcohol, if the sample is not completely water soluble. To it add 0.5 ml of phenolphthalein indicator solution and titrate with 0.05 N sodium hydroxide (NaOH) solution to the first pink colour. Now, pipette 50 ml of the sample into the flask and titrate it with the 0.05 N sodium hydroxide (NaOH) solution until the same originally first pink colour is 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, in g/ml.

B-6 PRECISION AND BIAS

Repeatability 0.000 8 percent

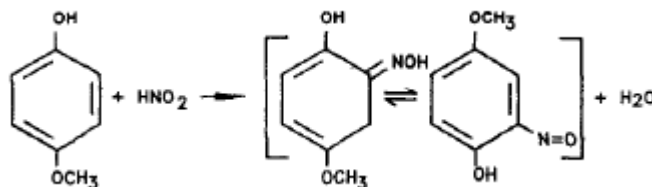
Reproducibility 0.001 4 percent

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

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

C-1 GENERAL

The test method is used to determine amount of monomethylether of hydroquinone (MEHQ) in *n*-butyl acrylate. It is applicable to the determination of MEHQ in the concentration range from 0 to 1 200 ppm. 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 then measured by spectrophotometer at a wavelength of 420 nm.

C-2 APPARATUS

C-2.1 Spectrophotometer, with borosilicate glass cells for determining absorbance at 420 nm.

C-2.2 Volumetric Flasks, 50 and 100 ml capacity.

C-2.3 Measuring Pipette, 5 and 10 ml capacity.

C-3 REAGENTS

C-3.1 Acetic Acid, glacial.

C-3.2 Monomethyl Ether of Hydroquinone (MEHQ) (4-methoxyphenol)

C-3.3 Sodium Nitrite Solution

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

C-4 CALIBRATION

C-4.1 In a 100 ml volumetric flask containing approximately 50 ml of glacial acetic acid, add 0.10 g (to the nearest 0.1 mg) of MEHQ. Mix well until solution is completely dissolved, 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-4.2 Determine the absorbance of 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.

C-4.3 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-5 PROCEDURE

C-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.

C-5.2 Weigh the appropriate amount of specimen into a 50 ml volumetric flask containing 20 ml of glacial acetic acid. Add 1 ml of 2 percent sodium nitrite (NaNO_2) solution to the specimen and dilute to the mark with glacial acetic acid. Mix well and allow to stand for 10 min.

C-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-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-7 PRECISION

C-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-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.

ANNEX D

(Clause 5)

SAMPLING OF *n*-BUTYL 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
	(N)	(n)
(1)	(2)	(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, procedures 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