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Draft Indian Standard

NORMAL BUTYL ACETATE — SPECIFICATION

(Third revision of IS 230)

(ICS 71.080.70)

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

Last date for Comments
26 January 2023

FOREWORD

(Formal Clauses will be added later)

This standard was first issued in 1957 and subsequently revised in 1964 and 1972. The first revision was taken up to delete grade 2 (85 percent purity product) and retain grade 1 with slight modification in the requirements based on the latest development, as grade 2 had no application in market. In the second revision, the requirement for distillation range was modified and limit for water content was incorporated.

In this (third) revision, three grades have been incorporated based on the purity requirement. The requirement of colour and *n*-butanol have been incorporated. Also, the test methods have been updated and modified. This standard has been revised by taking considerable assistance from the ASTM D 4615 Standard Specification for *n*-butyl acetate and other referred ASTM test methods in ASTM D 4615.

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

1.1 This standard prescribes the requirements and the methods of sampling and test for normal butyl acetate (*n*-butyl acetate) suitable for industrial purposes.

2 REFERENCE

The following standards contain provisions which through reference in the 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 agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

| <i>IS No.</i> | <i>Title</i> |
|--|---|
| IS 229 : 2021 | Ethyl Acetate — Specification (<i>fourth revision</i>). |
| IS 1070 : 1992 | Reagent grade water — Specification (<i>third revision</i>) |
| IS 1448 (Part 178) : 2020/ ISO 6271 : 2015 | Methods of Test for Petroleum and its Products: Part 178 Clear Liquids — Estimation of Colour by the platinum Cobalt Colour Scale |
| IS 2362 : 1993 | Determination of water by karl fischer method — Test Method (<i>second revision</i>) |
| IS 4905 : 2015 / ISO 24153 : 2009 | Random sampling and randomization procedures (<i>first revision</i>) |
| IS 5298 : 2013 | Method for determination of distillation range and distillation yield (<i>second revision</i>) |
| IS 8768 : 2000 | Method of measurement of colour in liquid chemical products platinum-cobalt scale (<i>second revision</i>) |
| IS 15464 : 2022 | Anhydrous ethanol for use as blending component in motor gasoline — Specification (<i>first revision</i>) |

3 GRADES

There shall be four grades of the material, namely:

- a) Grade 1 — 99.5 percent purity
- b) Grade 2 — 98 percent purity
- c) Grade 3 — 90-92 percent purity

4 REQUIREMENTS

4.1 Description

The material shall be a clear, colourless liquid, free from visible impurities and with a characteristic ester odor. It shall consist essentially of the acetic ester of *n*-butanol ($\text{CH}_3\text{COOCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$).

4.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 of Table 1.

Table 1 Requirements for *n*-Butyl Acetate
(Clause 4.2)

| Sl No. | Characteristic | Requirement | | | Method of Test, Ref | |
|--------|----------------|-------------|---------|---------|---------------------|--------|
| | | Grade 1 | Grade 2 | Grade 3 | Annex | IS/ISO |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |

| | | | | | | |
|-------|---|------------------|------------------|-------------|---------|-------------------------------------|
| i. | Relative density at 27 /27°C ¹⁾ | 0.872 to 0.875 | 0.872 to 0.875 | 0.866-0.868 | — | Annex A of IS 229 / A-3 of IS 15464 |
| ii. | Ester content (as <i>n</i> -butyl acetate), percent by mass | 99.5, <i>Min</i> | 98.0, <i>Min</i> | 90-92 | Annex A | — |
| iii. | <i>n</i> -Butanol, percent by mass, <i>Max</i> | 0.5 | — | — | Annex A | — |
| iv. | Acidity (as acetic acid), percent by mass, <i>Max</i> | 0.01 | 0.01 | 0.01 | Annex B | — |
| v. | Residue on evaporation, percent by mass, <i>Max</i> | 0.005 | 0.005 | 0.005 | Annex C | — |
| vi. | Distillation range, °C | 124-129 | 120-128 | 118-128 | — | IS 5298 |
| vii. | Water content , percent by mass, <i>Max</i> | 0.1 | 0.1 | 0.2 | — | IS 2362 |
| viii. | Colour, Pt-Co, <i>Max</i> | 10 | 10 | 10 | — | IS 8768/ ISO 6271 |

4.2.1 Specific Requirement

When the material is urethane grade material, it shall be tested as per the requirement of grade 1 in addition to water content as 0.05, percent by mass, *Max* or as agreed between the purchaser or supplier.

4.2.2 Quality of Reagents

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

NOTE — ‘Pure Chemicals’ shall mean chemicals that do not contain impurities which affect the result of analysis.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in well-closed containers made of galvanized iron or of any other suitable material, subject to the provisions of law in force in the country for the time being or as agreed to between the purchaser and the supplier.

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

5.2 Marking

5.2.1 Each container shall bear legibly and indelibly the following information:

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

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

6 SAMPLING

6.1 Preparation of Test Sample

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

6.2 Number of Test

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.

6.3 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 result on the composite sample shall meet the prescribed requirement if the lot is to be accepted under this specification.

ANNEX A

[Table 1, Sl No. (ii)]

DETERMINATION OF ESTER CONTENT (*as n*-BUTYL ACETATE)

A-1 GENERAL

Two methods namely, Method A determination of assay by titration and Method B determination of assay by gas chromatography have been prescribed. In case of disputes, method B shall be the referee method.

A-2 METHOD A

A-2.1 Reagents

A-2.1.1 *Standard Alcoholic Sodium Hydroxide Solution, 0.5 N.*

A-2.1.2 *Standard Hydrochloric Acid, 0.5 N.*

A-2.1.3 *Phenolphthalein Indicator*

Dissolve 0.5 g of phenolphthalein in 100 ml of 95 percent rectified spirit. Make the solution faintly pink by adding dilute sodium hydroxide solution.

A-2.2 Procedure

A-2.2.1 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. Disconnect the flask and wash the ground glass joint with distilled water. 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.

A-2.2.2 Carry out a blank determination, using all the reagents excepting the material, under similar conditions and at the same time.

A-2.3 Calculation

$$\text{Ester content (as } n\text{-butyl acetate), percent by mass} = \frac{11.62 \times (V_2 - V_1) \times N}{M}$$

where

V_2 = volume of standard hydrochloric acid required .in the blank determination, in ml;

V_1 = volume of standard hydrochloric acid required in the test with the material, in ml;

N = normality of the standard hydrochloric acid; and

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

A-3 METHOD B

A-3.1 Apparatus

A-3.1.1 Gas Chromatograph

Any commercially available gas chromatograph with a flame ionization detector or thermal conductivity detector, a split/splitless injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

| | | |
|-------------------------------|---|--|
| Column | : | 3 m length of 6.4 mm outside diameter aluminium or stainless steel tubing, packed with 80/100 mesh size of polymeric or silica based porous material coated with 9.05 percent fluorsilicone polymer and 0.45 percent nonylphenoxypoly(ethyleneoxy)ethanol or equivalent. |
| Carrier gas | : | Helium or hydrogen with thermal conductivity detector and nitrogen, helium or argon with flame ionization detector. Minimum purity of carrier gas 99.95 percent. |
| Carrier gas flow rate, ml/min | : | 75 |
| Sample size, μ l | : | 5 μ l with thermal conductivity and 1 to 2 μ l with flame ionization detector |
| Temperature, $^{\circ}$ C | | |
| Column, isothermal | : | 90 |
| Injection port | : | 180 |
| Detector Block | : | 280 |

Solvents : Methylene Chloride and Acetone

NOTE — The above gas chromatographic conditions are suggestive. However, any GC having difference in detector, column packing material and type (packed/capillary having different length/diameter/film thickness etc) 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-3.2 Reagents

A-3.2.1 Standards for Calibrations and Identification

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

A-3.3 Calibration

A-3.3.1 Identification

Determine the retention time of each component by injecting small amount of highly pure material either individually or synthetic blend mixture. The below given table shows the typical retention times for esters:

| <i>Sequence of elution</i> | <i>Approx. Retention time, min</i> |
|----------------------------|------------------------------------|
| Air | 0.11 |
| <i>n</i> -Butanol | 0.42 |
| Dibutyl ethers | 0.55 |
| Peak 4 (unidentified) | 0.60 |
| Peak 5 (unidentified) | 0.68 |
| Peak 6 (unidentified) | 0.70 |
| <i>n</i> -Butyl Acetate | 1.00 |
| Peak 8 (unidentified) | 1.05 |
| Peak 9 (unidentified) | 1.18 |
| Peak 10 (unidentified) | 1.33 |
| Peak 11 (unidentified) | 1.51 |

A-3.3.2 The area under each peak is a measure of the corresponding sample component's concentration, if a detector responds equally to all components in sample. While for both flame ionization detectors and thermal conductivity detectors, the response to different components varies significantly, particularly for flame ionization detectors. Relative response factor calculated by injecting and measuring the response to known blends may be used to correct the detector response.

A-3.4 Procedure

Introduce a sample (for sample size see **A-3.1.1**) into the chromatograph using sufficient material 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-3.5 Calculation

A-3.5.1 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-3.5.2 Calculate the mass percent of each component as follows:

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

where

A = corrected peak response of the component;

B = sum of corrected peak responses; and

C = sum of water and acidity (as acetic acid).

ANNEX B

[Table 1, Sl No. (iii)]

DETERMINATION OF ACIDITY (as ACETIC ACID)

B-1 GENERAL

This test method covers the determination of total acidity as acetic acid, in concentrations below 0.05 percent in n-butyl acetate by mixing the sample with either an equal volume of water or an equal volume of alcohol and then titrating it 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 up to 100 ml with the alcohol.

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

B-4.1 If the sample is water soluble, take 50 ml of water or if the sample is not completely water-soluble, take 50 ml of alcohol into a 250 ml conical flask. To it add 0.5 ml of phenolphthalein indicator solution and titrate it with 0.05 N sodium hydroxide 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 solution until the same originally first pink colour is obtained.

B-5 CALCULATION

$$\text{Acidity (as acetic acid), percent by mass} = \frac{V \times N \times 0.12}{D}$$

where

V = sodium hydroxide solution required for titration of the sample, in ml;

N = normality of sodium hydroxide solution; and

D = density of sample, g/ml.

ANNEX C

[Table 1, Sl No. (iv)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1 APPARATUS

C-1.1 Oven, 105 ± 5 °C.

C-1.2 Evaporating Dish, platinum, 125 ml.

NOTE — Aluminium or porcelain dish may also be used.

C-1.3 Analytical Balance, precision to ± 0.1 mg.

C-2 PROCEDURE

C-2.1 Dry evaporating dish in an oven at 105 ± 5 °C. Cool it in desiccator and weigh (M_1). Now take 100 ml of the sample in above conditioned evaporating dish and place it on a steam bath or hot plate in a hood to evaporate the sample to dryness. Dry the residue in an oven at a temperature of 105 ± 5 °C to constant mass. Cool in a desiccator and reweigh (M_2).

$$\text{Residue on evaporation, percent by mass} = \frac{M}{D \times V}$$

where

M = mass of the residue ($M_2 - M_1$), in g;

D = density of the sample, g/ml; and

V = volume of sample, in ml.

ANNEX D
(Clause 6.1)
SAMPLING OF *n*-BUTYL ACETATE

D-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

D-1.2 Sampling instruments 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 shaking, stirring, rolling or by any other suitable means.

D-1.5 In case of bulk storage in tank, to draw a representative sample through sample valve located in pump discharge line, after thorough re-circulation of the material in tank.

D-1.6 The samples shall be placed in suitable, clean, dry and air-tight glass or metal containers on which the material has no action.

D-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

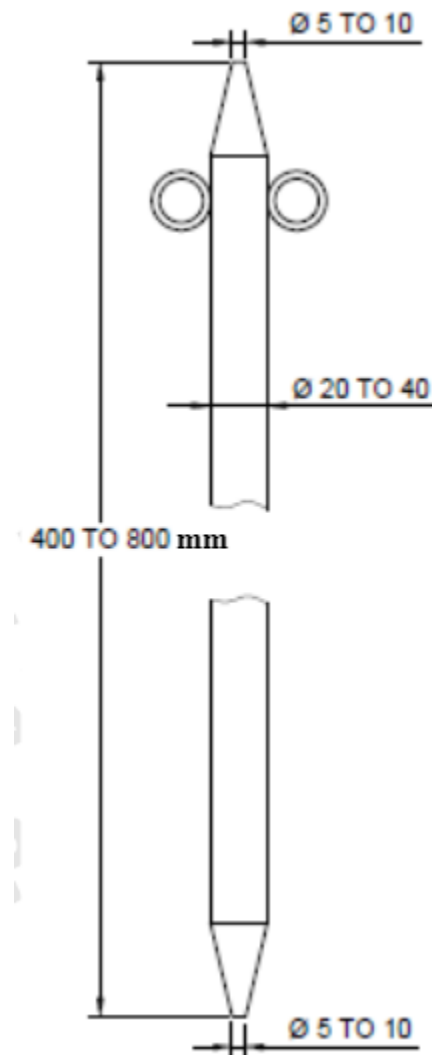
D-1.8 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the information indicating the source of manufacture, grade of the material, the month and year of manufacture of the material, the batch number (if available) and other details of sampling, such as the date of sampling, sampler's name, etc.

D-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

D-2 SAMPLING INSTRUMENT

D-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).

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.



All dimensions in millimeter
FIG. 1 SAMPLING TUBE

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

D-2.1.2 In case of sampling from bulk storage tank (through circulation pump sampling valve) suitable sampling container can be used.

D-2.1.3 In case of filled tanker (sampling through bottom valve) suitable sampling container can be used.

D-3 SCALE OF SAMPLING

D-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 or in case of bulk storage in tank, day / week composite sample collected as D-1.4 will constitute a separate lot.

D-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 Number of Containers to be Selected from
Lots of Different Sizes**
(Clause D-3.2)

| Lot Size | No. of Containers to be selected |
|-----------------|---|
| (1) | (2) |
| Up to 15 | 3 |
| 16 to 50 | 4 |
| 51 to 100 | 5 |
| 101 to 150 | 6 |
| 151 to 300 | 8 |
| 301 and above | 10 |

D-3.3 These containers shall be selected at random from the lot. In order to ensure randomness of selection, procedures given in IS 4905 may be followed.

D-4 TEST SAMPLES AND REFEREE SAMPLES

D-4.1 From each of the containers selected according to **B-3.3** a representative portion of ethyl acetate approximately 800 ml in volume (*see* NOTE) shall be drawn with the help of sampling data. 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, and 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 **D-1.7**.

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

D-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.

D-4.3 The referee sample prepared under **D-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 and is to be used in the case of any dispute between the two.