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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 09

Last date for Comments
22 December 2023

FOREWORD

(Formal Clauses will be added later)

n-Butyl acetate is an organic compound with the formula $\text{CH}_3\text{CO}_2(\text{CH}_2)_3\text{CH}_3$. It is the ester derived from *n*-butanol and acetic acid. It is found in many types of fruit, where it imparts characteristic flavors. It is used as an industrial solvent.

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, two grades have been formulated based on the purity requirement, with Grade 1 as 99 percent and Grade 2 as 98 percent. The requirement of colour and *n*-butanol have been incorporated. Also, the test methods for ester content (as *n*-butyl acetate), acidity and residue on evaporation have been updated and modified.

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 : 2023	Reagent grade water — Specification (<i>fourth 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>)

3 GRADES

There shall be two grades of the material, namely:

- a) Grade 1 — 99.0 percent ester content (as *n*-butyl acetate)
- b) Grade 2 — 98.0 percent ester content (as *n*-butyl acetate)

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 5 and col 6 of Table 1.

4.2.1 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.

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

Sl No.	Characteristic	Requirement		Method of Test, Ref	
		Grade 1	Grade 2	Annex	IS/ISO
(1)	(2)	(3)	(4)	(5)	(6)
i.	Ester content (as <i>n</i> -butyl acetate), percent by mass, <i>Min</i>	99.0	98.0	Annex A	—
ii.	Relative density at 27 °C /27 °C	0.872 to 0.875	0.872 to 0.875	—	Annex A of IS 229
iii.	<i>n</i> -Butanol, percent by mass, <i>Max</i>	0.50	—	Method B of Annex A	—
iv.	Acidity (as acetic acid), percent by mass, <i>Max</i>	0.02	0.02	Annex B	—
v.	Residue on evaporation, percent by mass, <i>Max</i>	0.005	0.005	Annex C	—
vi.	Distillation range, °C	124-129	120-128	—	IS 5298
vii.	Water content, percent by mass, <i>Max</i>	0.1	0.1	—	IS 2362
viii.	Colour, Pt-Co, <i>Max</i>	10	10	—	IS 8768/ IS 1448 (Part

¹⁾In case of disputes, IS 1448 (Part 178) shall be the referee method for determination of colour.

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 and 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 ester content, water content and acidity 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 g 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 General

This test method covers the determination of the *n*-butyl acetate content and impurities such as *n*-butanol by gas chromatography. Assay (as *n*-butyl acetate) is derived by subtracting sum of the various impurities like acidity, moisture, *n*-butanol etc.

A-3.2 Outline of the Test Method

A representative sample is introduced into a gas chromatograph equipped with 6 percent cyanopropylphenyl, 94 percent dimethylpoly-siloxane bonded phase fused silica capillary column. Suitable carrier gas transports the vaporized sample through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The product and other components are identified by comparing their retention times to the ones identified by analysing standards under identical conditions. The concentration of all components are determined in mass percent area by area normalization of the peak areas.

A-3.3 Apparatus

A-3.3.1 Gas Chromatograph

Any gas chromatograph equipped with a flame ionization detector (FID), a split injector (for example, split ratio – 2 : 1) and a suitable electronic integrator/software, capable of operating at the conditions listed below, may be used:

Column	:	Fused silica capillary column coated with 6 percent cyanopropylphenyl, 94 percent dimethylpoly-siloxane with length 30 m; internal diameter 0.53 mm or 0.32 mm or 0.25 mm and film thickness 1.8 or 3.0 μm or equivalent
Injector		
Temperature	:	240°C
Carrier gas	:	Nitrogen/Helium (3 ml/min or suitable as per column internal diameter)
Split ratio	:	1.25 or suitable
Detector		
Type	:	Flame ionization
Temperature	:	240 °C
Sample size	:	1 μl
Oven Program		
Initial temperature	:	60 °C
Initial hold time	:	0 min
Programme rate	:	12 °C/min
Final temperature	:	240 °C
Final hold time	:	0 min
Total run time	:	15 min

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-3.4 Reagents

A-3.4.1 *n*-Butyl Acetate

A-3.4.2 *n*-Butanol, pure

A-3.5 Data Acquisition System

Any suitable data integrator or PC based gas chromatograph software, which can handle features like external / internal standard calculations, etc.

A-3.6 Identification and Calibration

A-3.6.1 *Identification*

Determine the retention time of each component by injecting small amount of highly pure material either individually or synthetic blend mixture.

A-3.6.2 *Calibration*

A-3.6.2.1 Accurately prepare calibration standard mixture of known concentration for each component of *n*-butanol in *n*-butyl acetate.

A-3.6.2.2 Inject with the help of a clean and dry glass micro syringe, 1 µl of standard in the column taking care that no air bubble is trapped in the syringe. Inject each standard at least twice or till the repeatable results are obtained. Carry out the calibration by external standard method. Area of each component is to be measured with suitable data acquisition system.

A-3.6.2.3 The calibration factor is calculated by the following formula:

$$\text{Response factor of } n - \text{Butanol} = \frac{\text{Concentration of standard of } (n - \text{butanol}) \text{ obtained}}{\text{Area of standard of } (n - \text{butanol})}$$

NOTE — Suitable software for auto calibration with respect to mass of calibration standard may also be used.

A-3.7 Procedure

Inject 1 µl of sample by using manual or automatic liquid syringe, without any air bubble trapped in the syringe. Determine the mass concentration of all components by area normalization method.

A-3.8 Calculation

A-3.8.1 Calculate concentrations of impurities by correcting with respective response factor:

$$\text{Concentration of } n\text{-butanol, percent by mass} = \text{Area of } n\text{-butanol} \times \text{Response Factor}$$

A-3.8.2 Calculate the assay of *n*-butyl acetate on dry basis as below.

$$n\text{-Butyl acetate, percent by mass} = (100 - C)$$

where

C = Sum of moisture, acidity (as acetic acid) and *n*-butanol, percent by mass.

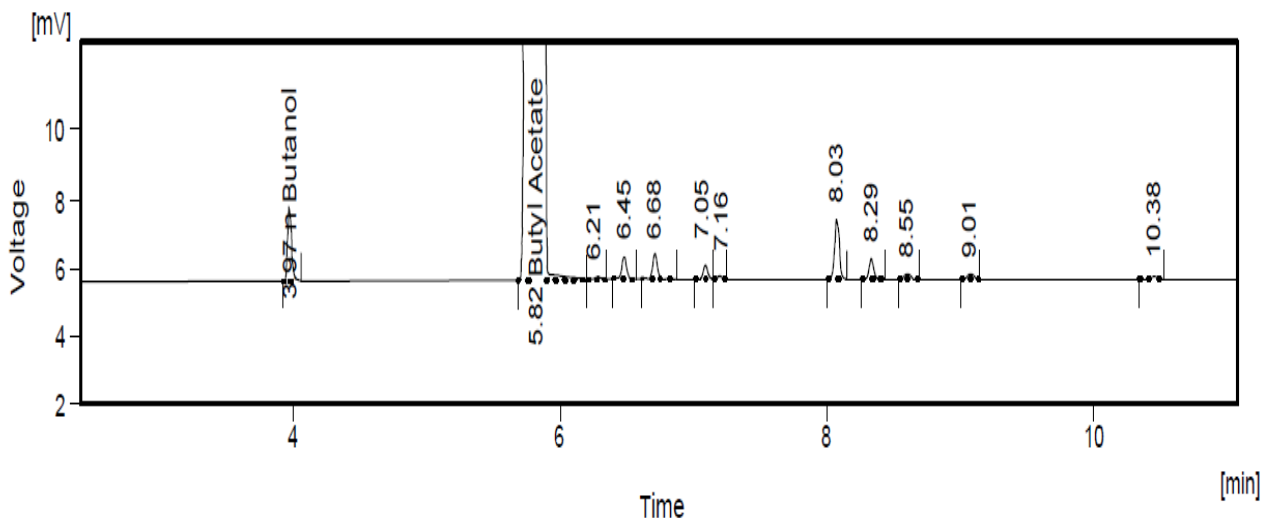


FIG. 1 TYPICAL CHROMATOGRAPH

ANNEX B

[Table 1, Sl No. (iii)]

DETERMINATION OF ACIDITY (as ACETIC ACID)

B-1 REAGENTS

B-1.1 Rectified Spirit, 95 percent (*see* IS 323).

B-1.2 Standard Sodium Hydroxide Solution, 0.025 or 0.050 N

B-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.

B-2 PROCEDURE

B-2.1 Take 50 ml of rectified spirit. To it 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 s.

B-3 CALCULATION

$$\text{Acidity (as acetic acid), percent by mass} = \frac{6.005 \times V \times N}{50 \times d}$$

where

V = Volume of standard sodium hydroxide solution, in ml;

N = Normality of standard sodium hydroxide solution, and

d = Relative density of *n*-butyl acetate at the test temperature.

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, 100 ml or 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).

C-2.3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M}{D \times V} \times 100$$

where

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

D = density of the sample, in 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 airtight 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.

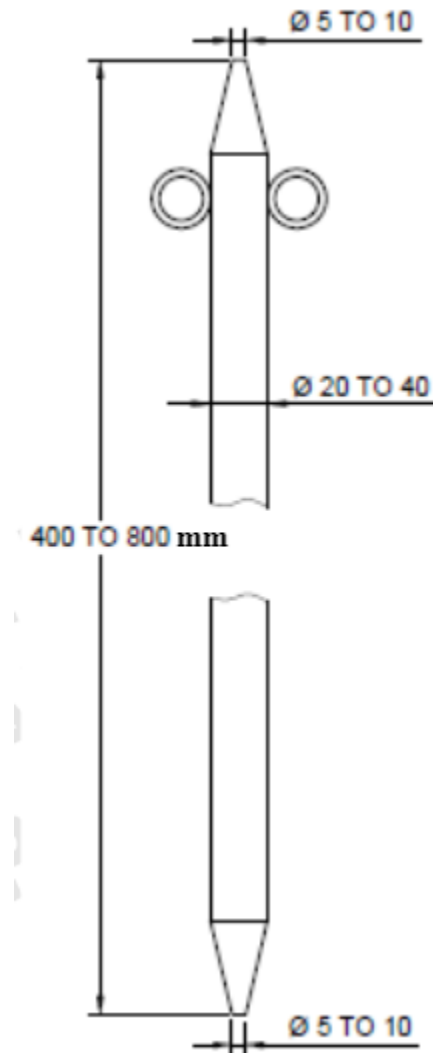
D-1.8 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the information indicating the source of manufacture, grade of the material, the month and year of manufacture of the material, the batch number and other details of sampling, such as the date 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 mm to 40 mm in diameter and 400 mm to 800 mm in length (*see* Fig. 2).

The upper and lower ends are conical and reach 5 mm 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 milliliter
FIG. 2 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 **D-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-1.8**.

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.