***भारतीय मानक***

***Indian Standard***

 **Doc: PCD 09 (21797) F**

 **IS 230 : 202X**

 **सामान्य ब्यूटाइल एसीटेट — *विशिष्टि***

*(* तीसरा पुनरीक्षण )

**NORMAL BUTYL ACETATE — SPECIFICATION**

*( Third Revision )*

ICS 71.080.70

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भारतीय मानक ब्यूरो

BUREAU OF INDIAN STANDARDS

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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

**FOREWORD**

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

*n*-Butyl acetate is an organic compound with the formula CH3CO2(CH2)3CH3. 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 originally published 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.

The composition of the Committee, responsible for the formulation of this standard is given at Annex E.

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.

*Indian Standard*

**NORMAL BUTYL ACETATE — SPECIFICATION**

*(Third Revision)*

**1 SCOPE**

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

**2 REFERENCES**

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:

|  |  |
| --- | --- |
| *IS No.* | *Title* |
| IS 229 : 2021 | Ethyl acetate — Specification (*fourth revision*) |
| IS 1070 : 2023 | Reagent grade water — Specification (*fourth revision*) |
| 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 (*second revision*) |
| IS 4905 : 2015 / ISO 24153 : 2009  | Random sampling and randomization procedures (*first revision*) |
| IS 5298 : 2013  | Method for determination of distillation range and distillation yield (*second revision*) |
| IS 8768 : 2000 | Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*) |

**3 GRADES**

There shall be two grades of the material, namely:

1. Grade 1 — 99.0 percent ester content (as *n-*butyl acetate)
2. 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 (CH3COOCH2CH2CH2CH3).

**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.** | **Characteristics** |  **Requirement** |  **Method of Test, Ref to** |
| Grade 1 |  Grade 2  |  | Annex IS/ISO |
|  |  |
| (1) | (2) | (3) | (4) |  | (5) |  (6) |
|  | Ester content (as *n-*butyl acetate), percent by area, *Min* | 99.0 | 98.0 |  | A | — |
|  | Relative density at 27 °C/27 °C | 0.872 to 0.875 | 0.872 to 0.875 |  | — | Annex A of IS 229  |
|  | *n*-Butanol, percent by mass, *Max* | 0.50 | — |  | Method B of Annex A | — |
|  | Acidity (as acetic acid), percent by mass, *Max* | 0.02 | 0.02 |  | B | — |
|  | Residue on evaporation, percent by mass, *Max* | 0.005 | 0.005 |  | C | — |
|  | Distillation range, °C  | 124 to 129 | 120 to 128 |  | — | IS 5298 |
|  | Water content, percent by mass, *Max* | 0.1 | 0.1 |  | — | IS 2362 |
|  | Colour, Pt-Co, *Max* | 10 | 10 |  | — | IS 8768/ IS 1448 (Part 178)1) |
|   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 of 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.* (i)]

**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.l** 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 1 h 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 except the material, under similar conditions and at the same time.

**A-2.3 Calculation**

Ester content (as *n*-butyl acetate), percent by mass = $\frac{11.62 x \left(V\_{2}-V\_{1}\right) × N}{M}$

where

*V2* = volume, in ml, of standard hydrochloric acid required in the blank determination;

*V1* = volume, in ml, of standard hydrochloric acid required in the test with the material;

*N* = normality of the standard hydrochloric acid; and

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

**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.

**A-3.2 Outline of the Test Method**

A representative sample is introduced into a gas chromatograph equipped with 6 percent cyanopropylphenyl, 94 percent dimethylpolysiloxane 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 dimethylpolysiloxane with length 30 m; internal diameter 0.53 mm or 0.32 mm or 0.25 mm and film thickness 1.8 µm 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 |
| Injection volume | : | 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*, know purity

**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:

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

**A-3.6.2.4** Similarly, calculate the response factor for *n*-butyl acetate.

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:

Concentration of *n*-butanol, ppm = Area of *n*-butanol x Response factor

**A-3.8.2** Similarly, calculate the concentration for n-butyl acetate.

**A-3.9 Report**

The reporting of purity is to be done as percent by area and impurities as percent by mass.



FIG. 1 TYPICAL CHROMATOGRAPH

**ANNEX B**

[*Table* 1, *Sl No.* (iv)]

**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 N 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**

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

where

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

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.* (v)]

**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 (*M1*). 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 (*M2*).

**C-2.2 Calculation**

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

where

*M* = mass,in g, of the residue (*M2 – M1*);

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

*V* = volume, in ml, of sample.

**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 milliliters

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.

**ANNEX E**

(*Foreword*)

**COMMITTEE COMPOSITION**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

|  |  |
| --- | --- |
| *Organization* | *Representative(s)* |
| National Chemical Laboratory (NCL), Pune | DR C. V. RODE (***Chairman***) |
| All India Distillers Association (AIDA), New Delhi | SHRI SUKHRAJ SONISHRI A.K. SINGHAL (*Alternate*)SHRI RAJESH DHINGRA (*Alternate*) |
| BASF India Limited, Mumbai | SHRI DATTATRAY ANNASO GURAVSHRI HEMAL (*Alternate*) |
| CSIR-Central Drug Research Institute (CDRI), Lucknow | DR SANJEEV KANOJIYA |
| Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi | SHRI UDAY CHAND |
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| Deepak Phenolics Limited, Vadodara  | SHRI DHARMESH SIDDHAPURIASHRI MEHUL KUMAR PATEL (*Alternate*) |
| Dow Chemical International Private Limited, Mumbai | SHRI V. MOHANDOSSSHRI GOVIND GUPTA (*Alternate*) |
| Godavari Biorefineries, Mumbai  | SHRI SHANUL LAXMANRAO PAGARSHRI APPASAHEB J. WANI (*Alternate*) |
| Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad | DR R. M. PATELSHRI C. S. PATEL (*Alternate*) |
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| India Glycols Limited, Uttarakhand | DR R. K. SHARMA SHRI ALOK SINGHAL (*Alternate*) |
| Indian Chemical Council (ICC), Mumbai | SHRI J SEVAK SHRI DHRUMIL SONI (*Alternate*) |
| Indian Oil Corporation Limited, Panipat | DR Y. S. JHALA |
| Jubilant Agri and Consumer Products Limited, Gurugram | DR KANAK BARAN DASS |
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| United Phosphorus Limited (UPL), Mumbai | SHRI M D VACHHANI |
| In Personal Capacity (*37 Nandanvan Society, Near GNFC Township, Narmadanagar, Bharuch, Gujarat – 392015*) | DR MAYUR J. KAPADIA |
| BIS Director General | SHRIMATI MEENAL PASSI, SCIENTIST ‘F’/SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS DEPARTMENT) [Representing Director General (*Ex-Officio*)] |
| *Member Secretary*MS ADITI CHOUDHARYSCIENTIST ‘B’/ASSISTANT DIRECTOR (Petroleum, Coal and Related Products Department), BIS |