

**BUREAU OF INDIAN STANDARDS**  
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*Draft Indian Standard*  
**NORMAL BUTYL ACETATE — SPECIFICATION**  
(Third revision of IS 230)  
(ICS 71.080.70)

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

SI 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	99.0, <i>Min</i>	98.0, <i>Min</i>	Annex A	—
	Percent by mass is to be replaced by				
	Ester content (as <i>n</i> -butyl acetate), percent by area	99.00, <i>Min</i>	98.00, <i>Min</i>	Annex A	—

Alternative method

**ANNEX A**  
[Table 1, SI No. (ii)]

## DETERMINATION OF ESTER CONTENT (PURITY) AND BUTANOL BY GAS CHROMATOGRAPHIC METHOD

### A-3.1 General

This test method covers the determination of the Butyl Acetate content and other impurities such as Butanol & other suspected impurities by gas chromatography. Assay as Butyl Acetate is derived by subtracting sum of the various impurities like Acidity, Moisture, Butanol etc.

Or

GC purity % by area as n-butyl acetate is derived by directly calculated by GC.

### A-3.2 Summary of Test Method

A representative sample is introduced into a gas chromatograph equipped with 6% Cyanopropylphenyl, 94% Dimethylpoly-Siloxane bonded phase fused silica capillary column. Suitable carrier gas transports the vapourized 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:** capable of operating at the conditions listed below. A heated flash vaporizing injector designed to provide a linear sample split injection (for example, 2: 1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. Pressure control devices and gauges shall be designed to attain the linear velocity required in the column used. A flame ionization detector with associated gas controls and electronics, designed for optimum response with open tubular columns, is required.

**A-3.3.2 Sample Introduction** - Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 0.2 to 1.0 µl injections are suitable. It should be noted that inadequate splitter design, poor injection technique, and overloading the column could result in poor resolution. Avoid overloading, particularly of the main product peak and eliminate this condition during analysis.

**A-3.3.3 Column** - This test method utilizes a fused silica open tubular Capillary Column as below,

Column : Fused silica Capillary Column Coated with 6% Cyanopropylphenyl, 94 %  
Dimethylpoly-Siloxane (HP- 624, BP-624, DB-624, RTX-624 or

Equivalent Column is suitable)

Column length : 30 m  
Internal diameter : suitable from 0.53 / 0.32 / 0.25 mm  
Film thickness : suitable from 1.8 / 3.0  $\mu\text{m}$

#### **A-3.3.4 Typical instrument conditions**

##### ***Injector***

Temperature : 240°C  
Carrier gas : Nitrogen/Helium (3 ml/min or suitable as per column ID)  
Split ratio : 1.25 or suitable  
Sample size : 1  $\mu\text{l}$

##### ***Detector***

Type : Flame ionization  
Temperature : 240°C

##### ***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.

#### **A-3.3.5 Data acquisition system**

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

#### **A-3.4 Reagents Materials**

- i. Butyl Acetate AR grade
- ii. Butanol pure

also other suspected impurities as higher acetates etc.

### **A-3.5 Identification and Calibration**

#### **A-3.5.1 Identification**

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

#### **A-3.5.2 Calibration**

Accurately prepare calibration standard mixture of known concentration for each component like Butanol and other suspect impurities in AR grade Butyl acetate.

Inject with the help of a clean and dry glass micro syringe, 1 micro liter 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.

The calibration factor is calculated by the following formula:

Concentration of standard Butanol obtained.

R.F. of Butanol = -----

Area of standard Butanol

Same way Response factor of all other impurities can be derived.

Or use suitable software for auto calibration with respect to mass of calibration standard.

#### **A-3.5.3 Analysis Procedure**

Inject 1 micro liter of sample without any air bubble trapped in the syringe.

Determine the mass concentration of all components by area normalization method.

#### **A-3.5.4 Calculations**

Calculate concentrations of impurities by correcting with respective response factor e.g.

Conc. of Butanol in ppm = Area of Butanol x RF

Calculate the assay of Butyl acetate on dry basis as below.

Percent Butyl acetate = (100 – C)

where.

C = Sum of moisture, acidity as acetic acid, Butanol and other impurities.

Or

GC purity of n-Butyl Acetate % by area = n-Butyl acetate by GC

**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, H<sub>2</sub> or N<sub>2</sub>), 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.