



SEMI C24-0699

SPECIFICATION FOR n-BUTYL ACETATE

This specification was technically approved by the Global Process Chemicals Committee and is the direct responsibility of the North American Process Chemicals Committee. Current edition approved by the North American Regional Standards Committee on April 23, 1999. Initially available on SEMI OnLine May 1999; to be published June 1999. This document replaces SEMI C1.5 in its entirety. Originally published in 1978.

1 Purpose

1.1 The purpose of this document is to standardize requirements for n-butyl acetate used in the semiconductor industry and testing procedures to support those standards. Test methods have been shown to give statistically valid results. This document also provides guidelines for grades of n-butyl acetate for which a need has been identified. In the case of the guidelines, the test methods may not have been statistically validated yet.

2 Scope

2.1 The scope of this document is all grades of n-butyl acetate used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

SEMI C1 — Specifications for Reagents

SEMI Suggested Guidelines for Pure Water for Semiconductor Processing

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	0.88 g/mL
Boiling Point	126.5°C

7 Requirements

7.1 The requirements for n-butyl acetate for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

NOTE 1: Each laboratory is responsible for verifying the validity of the method within its own operation.

8.1 Assay — Analyze the sample by gas chromatography (see SEMI C1, Section 3.1, Guidelines for Assay by Wide Bore Column Gas Chromatography). The parameters cited have given satisfactory results.

Column: 30 meter × 530 micron I.D. fused silica capillary, coated with 5 micron film of DB-1 or equivalent (100% methyl silicone which has been surface bonded and cross linked).

Column Temperature: 40°C isothermal for 5 minutes, then programmed to 200°C at 10°C/min.

Injector Temperature:	150°C
Detector Temperature:	250°C
Sample Size:	0.2 µL splitless
Carrier Gas:	Helium at 3 mL/min
Detector:	Thermal Conductivity
Approximate Retention Times (min):	
Butyl Acetate	8.0

8.2 Color — Dilute 3.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 15) with 100 mL of sample in Nessler tubes. View vertically over a white background. The sample must be no darker than the standard.

8.3 Acidity — To 57 mL (50 g) of sample in a 250 mL conical flask, add 0.5 mL of phenolphthalein indicator solution and titrate with 0.1 N alcoholic potassium hydroxide until a slight pink color persists for at least 15 seconds. Not more than 1.0 mL of the potassium hydroxide solution should be required.

8.4 Residue after Evaporation — Evaporate 90 mL (80 g) of sample to dryness. Dry at 105°C for 30 minutes, cool in a desiccator, and weigh (see SEMI C1, Section 3.3, Determination of Residue after Evaporation).

8.5 Water — Add 25 mL of methanol to a dry titration flask and add Karl Fischer (KF) reagent to a visually or electrometrically determined endpoint that persists for 30 seconds. Add 25 mL (22 g) of sample, taking care to protect the sample and contents of the flask from moisture. Stir vigorously and titrate with Karl Fischer reagent to the same endpoint.

$$\% \text{ Water (H}_2\text{O)} = \frac{\text{mL KF reagent} \times \text{KF factor (gH}_2\text{O/mL)} \times 100}{\text{Weight of sample (g)}}$$



8.6 Phosphate — To 11 mL (10 g) of sample, add 10 mL of sodium carbonate reagent solution and evaporate to dryness on a steam bath in a hood. Dissolve the residue in 25 mL of 0.5 N sulfuric acid. Add 1 mL of ammonium molybdate reagent solution and 1 mL of p-(methylamino)phenol sulfate reagent solution. Allow to stand at room temperature for 2 hours. Any blue color should be no greater than that produced when 0.01 mg of phosphate ion (PO_4) is treated as the sample.

8.7 Arsenic and Antimony (as As) — Evaporate 45 mL (40 g) of sample in a 150 mL beaker to a small volume in a hood. Add 50 mL of water and again evaporate to a small volume. Repeat the evaporation with water addition. Do not allow to go to dryness. Add 5 mL of nitric acid and 5 mL of sulfuric acid and evaporate to dense fumes of sulfur trioxide. Cool, cautiously add 10 mL of water, and again evaporate to dense fumes of sulfur trioxide. Cool, and cautiously wash into a generator flask with water to make a volume of 35 mL. Proceed as described in the General Method for Arsenic (and Antimony) under SEMI C1, Section 3.4.5, starting with the sentence which begins: "Swirl the flask...." Any red color in the silver diethyldithiocarbamate solution of the sample should be no greater than that of the standard containing 0.002 mg of arsenic (As).

8.8 Trace Metal Analysis — The following method has given satisfactory results in determining trace metal impurities at the value specified for each of the following trace metals: aluminum (Al), boron (B), calcium (Ca), chromium (Cr), copper (Cu), gold (Au), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), tin (Sn), titanium (Ti), and zinc (Zn). Alternate methods may be used as long as appropriate studies demonstrate recovery between 75 - 125% of a known sample spike for half of the value of each specified item.

8.8.1 Special Reagents

8.8.1.1 Solution A — Glycerol (ACS Reagent Grade) 10 g, Adipic Acid (99 + %) 1 g, EDTA Acid (ACS Reagent Grade) 0.1 g. Dilute to 1 L using water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for "acceptable" or better.

8.8.1.2 Nitric Acid, Ultra Pure — Use 70% nitric acid specified for ultra low metal ion content.

8.8.1.3 2% Nitric Acid Solution — Dilute 20 mL of ultra pure nitric acid to 1 L using water meeting the SEMI Suggested Guidelines for Pure Water for Semiconductor Processing for "Acceptable" or better.

8.8.2 Sample Preparation

8.8.2.1 In a clean environment, place 250 g of sample in a PTFE dish. Add 50 mL of Solution A. Slowly evaporate in a hot plate avoiding loss of sample by effervescence or spattering until there is no further loss of liquid. Cool. Add 1 mL of ultra pure, 70% nitric acid. While maintaining volume, carefully warm several minutes to dissolve any residue. Cool. Transfer quantitatively to a 50 mL volumetric flask using 2% nitric acid for rinsing and dilution to volume. Run a reagent blank.

8.8.3 Analysis

8.8.3.1 Using the prepared sample, analyze group I elements by flame atomic absorption spectroscopy and all other elements by plasma emission spectroscopy. Apply, if necessary, a reagent blank correction to the final determined value of the sample.

NOTE 2: Repeat analysis for tin using larger sample size if the instrument sensitivity is insufficient at the specified limit.

9 Grade 2 Procedures

9.1 This section does not apply to this chemical.

10 Grade 3 Procedures

10.1 This section does not apply to this chemical.

11 Grade 4 Procedures

11.1 This section does not apply to this chemical.

12 Grade 5 Procedures

12.1 This section does not apply to this chemical.

13 VLSI Grade Procedures

13.1 This section does not apply to this chemical.

14 Tier A Procedures

14.1 This section does not apply to this chemical.

15 Tier B Procedures

15.1 This section does not apply to this chemical.

16 Tier C Procedures

16.1 This section does not apply to this chemical.

17 Tier D Procedures

17.1 This section does not apply to this chemical.