

SEMI C32-0699

SPECIFICATION FOR METHYL ETHYL KETONE

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 C1.11 in its entirety. Originally published in 1978.

1 Purpose

1.1 The purpose of this document is to standardize requirements for methyl ethyl ketone 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 methyl ethyl ketone 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 methyl ethyl ketone used in the semiconductor industry.

3 Limitations

3.1 None.

4 Referenced Documents

SEMI C1 — Specifications for Reagents

5 Terminology

5.1 None.

6 Physical Property (for information only)

Density at 25°C	0.80 g/mL
Boiling Point	79.6°C

7 Requirements

7.1 The requirements for methyl ethyl ketone for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

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):	
Methyl Ethyl Ketone	9.0
2-Butanol	10.5

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 25 mL of water in a glass-stoppered flask, add 10 mL of sample and 0.1 mL of phenolphthalein indicator solution. Add 0.10 N sodium hydroxide until a slight, pink color persists after shaking for one-half minute. Add 21 mL (17 g) of the sample, mix well, and titrate with 0.01 N sodium hydroxide until the pink color is reproduced. Not more than 3.0 mL of the sodium hydroxide solution should be required.

8.4 **Residue After Evaporation** — Evaporate 63 mL (50 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 pyridine-dichloromethane mixture (1 + 1) 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 (20 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.

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

8.6 *Phosphate* — To 12.5 mL (10 g) of sample, add 10 mL of sodium carbonate reagent solution and evaporate to dryness on a steam bath in a hood. Carefully 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 *Heavy Metals (as Pb)* — Evaporate 25 mL (20 g) of sample to dryness on a steam bath in a hood. Dissolve the residue in 3 mL of dilute hydrochloric acid (1 + 1) and dilute with water to 15 mL. If necessary, filter through a small filter, and wash the evaporating dish and the filter with 10 mL of water. Dilute to 25 mL with water. For the standard, dilute a solution containing 0.02 mg of lead ion (Pb) and 3 mL of dilute hydrochloric acid (1 + 1) to 25 mL with water. Adjust the pH of both solutions to between 3 and 4 with dilute ammonium hydroxide (10% NH_3) or with acetic acid, and dilute with water to 40 mL. To each solution, add 10 mL of freshly prepared hydrogen sulfide water and compare. The sample solution should be no darker than the standard.

8.8 *Arsenic and Antimony (as As)* — Evaporate 250 mL (200 g) of sample in a 400 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 and 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).

8.9 *Trace Element Contents* — By a suitable emission spectrographic procedure, determine for each of the specified trace elements that its content is not greater than the stated specification limit (see SEMI C1, Section 3.5, Guidelines for Determination of Trace Elements by Emission Spectrography).

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.

Table 1 Impurity Limits and Other Requirements for Methyl Ethyl Ketone

Previous SEMI Reference #	C1.11-95
	Grade 1
	(Specification)
Assay ($\text{CH}_3\text{COCH}_2\text{CH}_3$)	99.5% min
Color (APHA)	15 max
Acidity	0.6 $\mu\text{eq/g}$ max
Residue after Evaporation	20 ppm max
Water (H_2O)	0.2% max