

# SEMI C51-0699

## SPECIFICATION FOR XYLENES

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 C1.21 in its entirety. Originally published in 1978.

### 1 Purpose

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

2.2 This material is a mixture of ortho, meta, and para isomers and ethylbenzene. Xylene (o-, m-, and p- isomers and ethylbenzene) 99.0% min and Ethylbenzene 18% max.

### 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 | 0.86 g/mL   |
| Boiling Range   | 136 - 144°C |

### 7 Requirements

7.1 The requirements for xylenes for Grade 1 are listed in Table 1.

### 8 Grade 1 Procedures

8.1 *Assay* — Assay the sample by gas chromatography (see SEMI C1, Section 3.1, Guidelines for Assay by Wide Bore 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:              | 175°C                                    |
| Detector Temperature:              | 250°C                                    |
| Sample Size:                       | 0.2 µL splitless                         |
| Carrier Gas:                       | Helium at 3 mL/min                       |
| Detector:                          | Thermal Conductivity or Flame Ionization |
| Approximate Retention Times (min): |                                          |
| Toulene                            | 14.4                                     |
| Ethylbenzene                       | 17.0                                     |
| p-xylene                           | 16.9                                     |
| m-xylene                           | 17.2                                     |
| o-xylene                           | 17.8                                     |

8.1.1 Measure the areas under all peaks by any convenient means and calculate the concentration of all components in area percent (see SEMI C.1, Section 3.11, Guidelines for Assay by Wide Bore Column Gas Chromatography).

8.2 *Color* — Dilute 2.0 mL of platinum-cobalt stock solution (APHA No. 500) to 100 mL with water. Compare this standard (APHA No. 10) 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 0.01 N sodium hydroxide until a slight pink color persists after shaking for one-half minute. Add 38 mL (33 g) of the sample, mix well, and titrate with 0.01 N sodium hydroxide until the pink color is reproduced. Not more than 1.0 mL of the sodium hydroxide solution should be required.

8.4 *Residue After Evaporation* — Evaporate 232 mL (200 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 23 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 Chloride** — To a 100 mL separatory funnel, add 12 mL (10 g) of sample and 40 mL of water. Shake well for 30 seconds and allow the two layers to separate. Discard the sample (upper) layer. To a 20 mL portion of the water layer add 1 mL of nitric acid and 1 mL of silver nitrate reagent solution. Any turbidity produced should be no greater than that of a standard containing 0.015 mg of chloride ion (Cl) in an equal volume of solution and the amounts of reagents used.

**8.7 Phosphate** — To 12 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<sub>4</sub>) is treated as the sample.

**8.8 Arsenic and Antimony (as As)** — Evaporate 116 mL (100 g) of sample in a 250 mL beaker to a small volume in a hood. Add 50 mL of water and again evaporate to a small volume. Repeat the evaporation three times with water additions. 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 0.001 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.