

SEMI C62-0309 (Reapproved 0618)

SPECIFICATION FOR POROGEN PRECURSORS USED IN LOW K CVD PROCESSES

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1 Purpose

1.1 The purpose of this Document is to provide specifications for porogen precursors used in Low K CVD processes for which a need has been identified.

2 Scope

2.1 The scope of this Document lists the proposed impurity limits for porogen precursors (e.g., alpha-terpinene, limonene, bicycloheptadiene, and cyclooctane) used in the semiconductor industry.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 None.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 None.

6 Physical Properties (for information only)

	<i>Alpha-Terpinene</i>	<i>Limonene</i>	<i>Bicycloheptadiene</i>	<i>Cyclooctane</i>
Chemical Formula	C ₁₀ H ₁₆	C ₁₀ H ₁₆	C ₇ H ₈	C ₈ H ₁₆
CAS#	99-86-5	138-86-3	121-46-0	292-64-8
Appearance	Clear liquid	Clear liquid	Clear Liquid	Clear Liquid
Density	0.845 gm/mL	0.840 gm/mL	0.906 gm/mL	0.834 gm/mL
Mol. Wt	136 g/mol	136.23 g/mol	92.14 g/mol	112.21 g/mol
Boiling Point	172°C–175°C	175°C–176°C	88°C–90°C	151°C–153°C

7 Requirements

7.1 The requirements for porogen precursors for Grade 1 are listed in Table 1.

8 Grade 1 Procedures

8.1 *Assay* — The following method has given satisfactory results in determining assay at the value specified in these chemicals. Alternative methods may be used as long as appropriate method validation as per SEMI C1 can be demonstrated.

8.1.1 Since most impurities in each of these porogens have the same empirical formula as the main component, the concentration of each component is directly proportional to the peak areas and no external calibration is necessary.

8.1.2 Program a gas chromatograph fitted with a 30 m long $\times$ 250 μ m ID $\times$ 0.5 μ m 5% phenyl—95% polydimethylsiloxane film column, a split injection port, and a flame ionization detector (FID) according to the parameters in the table.

Parameter	Setting	Parameter	Setting
Oven		Inlet	
Initial Temp [C]	60	Temperature [C]	200
Initial Time [min]	2	Mode	Split
Ramp1 [C/min]	4	Split Ratio	50-1
Temp1 [C]	100	Detector (FID)	
Time1 [min]	2	Temperature [C]	260
Ramp2 [C/min]	15	Hydrogen Flow [mL/min]	40
Temp2 [C]	250	Air Flow [mL/min]	450
Time2 [min]	2	Mode	Const Makeup
Column		Makeup Flow [mL/min]	45
Flow	1.3 mL/min		

8.1.3 Identify the retention time of eucalyptol (CAS# 470-82-6) or any other oxygen-containing impurity using these conditions. Oxygen compounds are NOT to be included in the assay.

8.1.4 Inject 0.1 μ L of neat liquid.

8.1.5 For alpha-terpinene, sum the area of all peaks eluting between 10 and 15 minutes (monoterpenes with formula $C_{10}H_{16}$, similar vapor pressure). Subtract the area of eucalyptol which elutes in this time range. Divide the area of the monoterpenes by the total area of all compounds detected and multiply by 100% to calculate the ATRP assay.

8.1.6 For other porogens, establish the retention time range which includes all the isomers of the main component and sum all these peaks. Subtract the area of any oxygen-containing impurity which may elute in this time range. Divide the area of the porogens by the total area of all compounds detected and multiply by 100% to calculate the porogen assay.

8.2 *Water* — The following method has given satisfactory results in determining water at the value specified in these chemicals. Alternative methods may be used as long as appropriate method validation as per SEMI C1 can be demonstrated.

8.2.1 Prepare and operate a Karl Fischer coulometric titrator according to the manufacturer's instructions. Insure that there is minimal background drift due to moisture permeating into the titration cell.

8.2.2 Using a dry syringe, draw 1 mL of sample and inject into the titration cell.

8.2.3 Calculate the sample weight from the density and volume of liquid delivered to the cell.

8.2.4 Divide the μ g of water found by the sample weight to calculate the water content in ppmw.

8.3 *Chloride* — The following method has given satisfactory results in determining chloride at the value specified in these chemicals. Alternative methods may be used as long as appropriate method validation as per SEMI C1 can be demonstrated.

8.3.1 Accurately weigh and transfer approximately 50 g of chemical into a separatory funnel. Add 10 mL of distilled deionized water to the separatory funnel and shake thoroughly to extract the hydrolysable chloride from the sample. Vigorous manual shaking for 5 minutes is sufficient. An automatic shaker may also be used. If an automatic shaker is used, shake at a minimum speed of 120 RPM for a minimum of 15 minutes.

8.3.2 Analyze the aqueous layer by ion chromatography (see SEMI C1) using conditions suitable to separate the chloride peak from other anionic peaks in the sample.