SEMI C76-0811 (Reapproved 0317)
GUIDE FOR ZIRCONIUM TERT-BUTOXIDE

This Standard was technically approved by the Liquid Chemicals Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 8, 2016. Available at www.semiviews.org and www.semi.org in March 2017; originally published August 2011.

1 Purpose

1.1 The purpose of this Document is to provide a guide for zirconium tert-butoxide for which a need has been identified.

2 Scope

2.1 The scope of this Document covers high purity zirconium tert-butoxide which is used in the semiconductor industry for the deposition of zirconium oxide based layers by atomic layer deposition or chemical vapor deposition.

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

4.2 *ASTM Standard*[1]

ASTM D5127 — Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

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

5 Terminology

5.1 *Abbreviations and Acronyms*

5.1.1 *NMR* — nuclear magnetic resonance

5.1.2 *TMS* — tetramethylsilane

6 Properties

Table 1 Properties for Zirconium Tert-Butoxide and CAS Number (for information only)

Molecular Formula	$Zr[OC(CH_3)_3]_4$
Molecular Weight	383.74 g/mol
CAS Number	2081-12-1
Boiling Point	81°C at 3 mmHg
Melting Point	3°C
Solubility in Water	Reacts
Appearance	Colorless liquid

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

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7 Suggested Values

7.1 The suggested values for zirconium tert-butoxide for Tier A are listed in Table 2.

8 Grade 1 Procedures

8.1 This section does not apply to this chemical.

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 Tier A Procedures

NOTE 1: Standardized test methods are being developed for all parameters at the purity level indicated. Until standardized test methods are published, test methodology shall be determined by user and producer.

12.1 *Trace Metals Analysis* — The following method has given satisfactory results in determining trace metal impurities at the specified value for the elements listed in Table 2.

12.1.1 *Mixed Acid* — Prepared by dilution of ultrapure 49% HF, ultrapure 70% HNO_3 and ultrapure 31% H_2O_2 with ultrapure water in the following ratio: 25 mL of HF, 10 mL of HNO_3 and 10 mL of H_2O_2 to 500 mL with water.

12.1.2 *Standards* — Prepare multielement standard solutions (calibration standards and quality control check standards) by diluting with mixed acid described in ¶ 12.1.1 of appropriate weights for inductively couple plasma mass spectrometry (ICP-MS).

12.1.3 *Sample Preparation* — Dispense approximately 0.1 mL of the zirconium tert-butoxide sample into a dry and clean PFA sample bottle and record the weight to at least 3 decimal places. Under hood, add 49% HF dropwise into the sample aliquot. The sample will react vigorously with the HF; allow the reaction to proceed until it subsides before adding the next drop. Repeat until 2 mL of HF have been added. Dilute the sample with the acid mixture containing H_2O_2, HNO_3 and HF (¶ 12.1.1). The final dilution factor of the sample should be appropriate to minimize the space charge effects in the ICP-MS measurement of the sample. This dilution factor will be dependent on the type of ICP-MS used for analysis.

12.1.4 *Analysis* — Analyze the samples by ICP-MS. High-resolution ICP-MS is suggested. It is recommended to run each sample in duplicate. In addition prepare a sample preparation blank for each analysis. It is also recommended to use an internal standard or a spiked sample solution to correct for the matrix effect.

12.2 *Hydrocarbons Analysis* — The following method has given satisfactory results in determining total hydrocarbons at the specified value using H-NMR.

12.2.1 *Sample Preparation* — Under an inert atmosphere transfer 0.2 mL of zirconium tert-butoxide into an NMR tube. Add 1 mL of a solution of the internal standard (tetramethylsilane [TMS]) in deuterated benzene.

12.2.2 *Analysis* — Calculate hydrocarbons from the integrations of peaks in the hydrocarbon region with that of the internal standard. For convenience hydrocarbons may be calculated as hexane.

12.2.3 *Assay by NMR* — Ratio of the integration peaks from zirconium tert-butoxide to total integration of all peaks in the range −0.5 to 8 ppm.

13 Tier B Procedures

13.1 This section does not apply to this chemical.

14 Tier C Procedures

14.1 This section does not apply to this chemical.

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