

SEMI C105-0923

GUIDE FOR TRACE IRON ANALYSIS IN HIGH PURITY 2-PROPANOL (IPA)

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NOTICE: The section symbol (§) is used to reference a numbered section, paragraph, or header and all subordinate paragraphs, headers, and embedded material (i.e., EXCEPTIONs, lists) therein. The paragraph symbol (§) is used to reference a particular numbered paragraph and embedded material therein. When duplicated (i.e., §§ and §§§) the symbols are used to reference more than one section or paragraph, respectively.

NOTICE: Paragraphs entitled ‘NOTE:’ are not an official part of this Standard or Safety Guideline and are not intended to modify or supersede the official Standard or Safety Guideline. These have been supplied by the global technical committee to enhance the usage of the Standard or Safety Guideline.

1 Purpose

1.1 The purpose of this Guide is to recommend procedures for trace iron analysis in high purity 2-propanol (IPA) needed by the semiconductor industry.

2 Scope

2.1 The scope of this Guide is the measurement of iron concentration in IPA, used in the semiconductor industry, with higher purity than Grade 4 IPA described in SEMI C41.

NOTE 1: Higher purity than Grade 4 is typically needed for semiconductor devices where the iron concentration of IPA must be less than 100 ppt.

2.2 The quantitative range of this method is 1 to 100 ppt.

2.3 Instruments (bottles, etc.) for sampling IPA to be analyzed are described.

2.4 A procedure for quantitative analysis of trace iron levels in IPA sampled in bottles is described.

2.5 Ultrapure water and nitric acid to be used are described.

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 Referenced Standards and Documents

3.1 *SEMI Standards and Safety Guidelines*

SEMI C41 — Specification and Guide for 2- Propanol

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

4 Terminology

4.1 *Abbreviations and Acronyms*

4.1.1 *ICP-MS* — inductively coupled plasma mass spectrometry

4.1.2 *IPA* — isopropyl alcohol, 2-propanol (IUPAC)

4.1.3 *PFA* — perfluoroalkoxy alkane

5 Physical Properties of IPA (for information only)

5.1 Density (at 25°C): 0.78 g/mL

5.2 Boiling Point: 82.3°C

NOTE 2: Related Information 1 shows the criteria for IPA described in Table 1 of SEMI C41.

NOTE 3: The scope of this Standard (refer to § 2) is IPA with higher purity than Grade 4.

6 Procedures

6.1 Sampling

6.1.1 This Standard does not provide operations or procedures for sampling the IPA. It is preferable to perform sampling in a clean environment to avoid contamination from the external environment. The following are concerns and cautions regarding sampling.

- Iron contamination from sampling lines.
- Iron contamination from the external environment.
- Iron adsorption to sampling lines (surface in contact with IPA).

6.1.2 It is important to take great care not to contaminate the sample during both sampling and testing operations. Pay attention to the material, shape, and cleaning of the instruments used. In addition, careful attention should be paid to prevent contamination from operators by wearing clean gloves, etc.

6.1.3 *Sampling Lines* — Sampling lines for IPA to be analyzed are assembled and prepared. Sampling tubes, fittings, and valves of high cleanliness should be used. It is preferable to use products made of fluoroplastic such as perfluoroalkoxy alkane (PFA) or polytetrafluoroethylene (PTFE). However, as there is a risk of a small amount of iron being absorbed from the IPA being sampled by the inner surface of the PFA tube at the initial stage of IPA flow, sampling should be performed after sufficient purging. Regarding the cleanliness of the sampling line, it must be confirmed that the background of iron concentration in the IPA flow, which has iron impurity of less than 100 ppt at the inlet, become stable within a certain range below the measured concentration. As needed, lines are cleaned by ultrapure water and IPA purging. For example, a background of 10 ppt or less is checked as an iron concentration of one tenth or less than the measurement of 100 ppt max. In addition, it is preferable to construct the sampling lines in a clean environment to avoid contamination from the external environment.

6.1.4 *Sampling Bottles* — Use clean bottles made of PFA or similar material (such as high density polyethylene [HDPE]). The cleanliness of the bottles is checked by iron extraction tests.

NOTE 4: These test procedures do not include measurement of the iron concentration in the IPA before it is placed in the bottle to be tested. There is a risk, therefore, of finding an unacceptable iron concentration after the storage period and not knowing whether that was due to iron contamination of the IPA prior to it being placed in the bottle or contamination by the bottle.

6.1.4.1 Recommended extraction test:

1. Fill the bottle with high purity IPA.
2. Add nitric acid as described in ¶ 6.2.4.
3. Store at room temperature for 24 to 72 hours.
4. Measure the iron concentration in the IPA.

6.1.4.2 Alternative extraction test:

1. Fill the bottle with a 0.5 to 1 wt % aqueous nitric acid solution, prepared with high purity nitric acid and ultrapure water.
2. Store at room temperature for 24 to 72 hours.
3. Measure the iron concentration in the solution.

6.1.4.3 The bottle may be used if the increase in iron concentration during storage is less than or equal to one tenth of the standard value for the measurement. For example, if the difference between the iron concentration in the IPA after storage and the iron concentration in the IPA before it was added to the bottle is less than 10 ppt, the bottle may be used for a test for compliance with a 100 ppt requirement.

6.1.4.4 If the iron concentration measured in the extraction test is more than one tenth of the limit for the IPA to be tested, clean the bottle with ultrapure water or high purity acid.