

SEMI E180-1220

TEST METHOD FOR MEASURING SURFACE METAL CONTAMINATION THROUGH ICP-MS OF CRITICAL CHAMBER COMPONENTS USED IN SEMICONDUCTOR WAFER PROCESSING

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1 Purpose

1.1 This Document describes a method for a quantitative analysis for surface trace-metal concentration of critical chamber components (CCCs) by using inductively coupled plasma-mass spectrometry (ICP-MS). This Standard is intended to promote communication between the users and the processing-equipment suppliers. It can be also used to facilitate better communication regarding surface metal-contamination expectation and its test method between the processing equipment suppliers and the CCC manufacturers.

1.2 This Document describes the procedure for trace-metal measurement, including the surface trace-metal collection from a CCC, as it influences the reliability of measurement data and reproducibility of each test facility.

1.3 The use of this Document is to ensure consistency in the reporting of results provided by each processing-equipment supplier or CCC manufacturer.

2 Scope

2.1 This Document applies to the measurement of surface trace-metal concentration of CCCs (e.g., showerheads, pedestals) by ICP-MS.

2.2 This Document applies to an unused CCC or its parts.

NOTE 1: This Document may be applied for a refurbished CCC if such application is agreed upon by the user and the supplier.

2.3 This Document covers local extraction and full-immersion trace-metal collection techniques.

2.4 The objective metals in this measurement are aluminum (Al), sodium (Na), potassium (K), calcium (Ca), lithium (Li), iron (Fe), copper (Cu), nickel (Ni), chromium (Cr), cobalt (Co), titanium (Ti), magnesium (Mg), and zinc (Zn).

NOTE 2: Either additional or fewer metals can be agreed upon between the user and the supplier.

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 It is not intended to be used to compare results of measurements on a CCC designed to be used in a model of processing equipment by one supplier to another CCC designed to be used in a different model of processing equipment by another supplier.

3.2 The accuracy of the method is limited by the detection limits of the ICP-MS instruments and by the sample preparation procedure.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI C10 — Guide for Determination of Method Detection Limits

SEMI C16 — Guide for Precision and Data Reporting Practices

SEMI F63 — Guide for Ultrapure Water Used in Semiconductor Processing

4.2 *ASTM Standards*¹

ASTM D1193 — Standard Specification for Reagent Water

4.3 *ISO Standards*²

ISO 14644-1 — Cleanrooms and Associated Controlled Environments – Part 1: Classification of Air Cleanliness by Particle Concentration

ISO/IEC 17025:2017 — General Requirements for the Competence of Testing and Calibration Laboratories

4.4 *International Roadmap for Devices and Systems*³

International Roadmap for Devices and Systems (IRDS™) 2017 Edition

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

5 Terminology

5.1 *Abbreviations and Acronyms*

5.1.1 *CCC* — critical chamber component

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

5.1.3 *ppb* — part(s) per billion (ng/mL)

5.1.4 *ppt* — part(s) per trillion (pg/mL)

5.1.5 *UPW* — ultrapure water

5.2 *Definitions*

5.2.1 *critical chamber component* — any component of the processing equipment that is in line of sight to the wafer in the process environment during processing and that:

1. is in physical contact with wafer (e.g., pedestal, mechanical clamp);
2. feeds gases, reagents, or liquids to the wafer (e.g., shower heads, gas inlets, nozzles);
3. sputters materials on or implants materials to the wafer; or
4. is shielding or chamber parts that have risk of transferring contaminants (e.g., particles, metals, molecules) to the wafer.

5.2.2 *pipette* (v.) — to measure or transfer a quantity of a liquid with a pipette.

5.2.3 *process chamber* — a component of the processing equipment capable of processing wafers (i.e., adding manufacturing value) and is usually a part of a process module.

5.2.4 *processing equipment* — equipment that has process chamber(s), control mechanisms, and their controllers, and is capable of processing wafers (i.e., adding manufacturing value). Processing equipment is directed in its processing by equipment recipes, which are under the control of the factory user.

5.2.5 *supplier* — party that provides equipment to, and directly communicates with, the user. A supplier may be a manufacturer, an equipment distributor, or an equipment representative. See also the definition for user. [SEMI S2]

NOTE 3: The use of ‘equipment’ in this definition as well as the definition for the ‘user’ is equivalent to ‘processing equipment’ in this Standard.

5.2.6 *user* — party that acquires equipment for the purpose of using it to manufacture semiconductors. See also the definition for supplier. [SEMI S2]

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² International Organization for Standardization, ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; Telephone: +41.22.749.01.11, <http://www.iso.org>

³ International Roadmap for Devices and Systems, <https://irds.ieee.org/>

6 Summary of Test Method

6.1 The test solution containing surface trace-metals is collected from the component using either a local-extraction method or full-immersion method. The trace metals in the test solution are measured by ICP-MS. The preparation technique (e.g., dilution by water) for the test solution is agreed upon between the supplier and the analytical laboratory. The trace-metal concentration is determined using a working curve, which is obtained by using a set of standard solutions.

7 Qualification of Analytical Laboratory

7.1 The analytical laboratory that performs the trace-metal contaminants should have a measurement quality-management system in accordance with ISO/IEC 17025:2017 or equivalent.

7.1.1 One way to show management competency is accreditation.

8 Apparatus

8.1 Measurement Instrument

8.1.1 An ICP-MS shall be used to measure the trace-metal concentration of samples obtained using local-extraction or full-immersion methods.

8.1.2 An ICP-MS consists of a sample introduction system, an ion source, a sampling interface, an ion lens, a mass spectrometer, and a detector.

8.1.3 *Instrument Condition* — An ICP-MS has the capability to detect multiple elements with an instrument detection limit in the low parts per trillion (ppt) to subparts per billion (ppb) range. Appropriate isotopes should be selected for each element of interest for accurate quantification and minimum interferences.

8.2 Additional Equipment Used for Full-Immersion Method

8.2.1 Orbital shaker

9 Reagents and Materials

9.1 Common Materials Used for Localized-Extraction Method and Full-Immersion Method

9.1.1 Lab Wares

9.1.1.1 Polypropylene volumetric flask

9.1.1.2 Micropipette

9.1.1.3 Sample vials (to contain extraction solutions)

9.1.2 Reagents

9.1.2.1 *Ultrapure Water (UPW)* — Resistivity $\geq 18.0 \text{ M}\Omega/\text{cm}$ at 25°C per ASTM D1193 and SEMI F63

9.1.2.2 *Inorganic Reagent* — Use high-purity reagents with low trace-metal impurities.

9.1.2.2.1 The required level of allowable trace-metal impurities varies depending on the target level of cleanliness, material of the CCC, etc. In order to achieve high sensitivity of detection and to avoid contamination of the CCC under test by the extraction solution, a high-purity level of reagents is necessary. For example, 100 ppt or lower of trace-metal level should be deemed sufficient in most cases.

NOTE 4: According to IRDS 2017 Table YE3, the maximum allowable contamination level of chemical that contacts with the surface of the wafer in production is less than 1 ppt.

9.1.2.3 *Standard Solutions* — Choose an appropriate, traceable, trace-metal standard that is available for the analytical laboratory and is suitable for the expected level of contamination for the CCC.

9.2 Additional Materials Used in Full-Immersion Method

9.2.1 Precleaned, polymeric, extraction container