SEMI F48-0600 (Reapproved 0709)
TEST METHOD FOR DETERMINING TRACE METALS IN POLYMER MATERIALS

This standard was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on May 13, 2009. It was available at www.semi.org in June 2009 and on CD-ROM in July 2009. Originally published June 2000.

1 Purpose

1.1 This method provides a procedure for determining the nonvolatile trace inorganic impurities in bulk polymeric materials.

2 Scope

2.1 Following digestion by dry ashing (DDA) or digestion in closed vessel (DCV) preparation techniques, samples previously obtained and cleaned according to SEMI F40 are analyzed for trace inorganics using inductively coupled plasma-mass spectrometry (ICP-MS), graphite furnace atomic absorption spectroscopy (GFAAS), and/or inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

2.2 Materials for analysis include, but are not limited to:

- Raw polymer materials (resins), such as pellets of perfluoroalkoxy (PFA), polyvinylidene fluoride (PVDF), ethylenechlorotrifluoroethylene (ECTFE), polyetheretherketone (PEEK), polypropylene (PP), polyethylene (PE), acetal resin, polyvinyl chloride (PVC), Perfluoromethylether-based Perfluoro-alkoxy (MFA) and powders of polytetrafluoro-ethylene (PTFE).
- Polymer components of tubing, piping, fittings, valves, regulators, filter housings, filter cartridges, O-rings and gaskets used in ultrapure water (UPW) and liquid chemical distribution systems (LCDS). See ¶ 3.8 for further information.
- Ion-exchange resins.
- Polymer products used in the manufacturing of semiconductor devices, such as wafer carriers and wands, as well as accessories internal to wet equipment (e.g., drums in spin rinse dryers, tanks in quick dump rinsers). See ¶ 3.8 for further information.

2.3 The DDA sections of this document refer to an ashing technique, whereby the sample is placed into a platinum or quartz crucible and thermally decomposed. Thermal decomposition in muffle furnace or microwave muffle furnace may also be used. Additionally, oxygen plasma may be used separately or in conjunction with these techniques.

2.4 The DCV sections of this document refer to closed vessel microwave acid decomposition at elevated temperature and pressure. Alternatively closed vessel thermal conduction heating may also be applied.

2.5 ICP-MS, GFAAS, and ICP-AES are all appropriate methods for inorganic analysis. ICP-MS is the preferred method because it is more sensitive and efficient. Alternate procedures may be used if they meet the same analytical performance criteria. Each laboratory is responsible for verifying the validity of each method within its own operation.

2.6 This method is applicable for the elements found in Table 1.

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Table 1 List of Applicable Elements (See #1)

Aluminum	Magnesium
Barium	Manganese
Calcium	Nickel
Chromium	Potassium
Cobalt	Sodium
Copper	Strontium
Iron	Tin
Lead	Titanium
Lithium	Zinc
Molybdenum	Zirconium

#1 See Limitations, ¶ 3.3.

2.7 This method may be used for other materials, or other nonvolatile elements, if the end-user wishes and performance is demonstrated for the analyte of interest, in the matrices of interest, at the concentration levels of interest.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 The accuracy of the method is limited by the detection limits of the instruments and by the sample preparation procedure.

3.2 This procedure anticipates analysis levels in the ppm (mass/mass) range. Impurities less than 0.1 ppm may not be detected by this method.

3.3 When extending the method to other elements recovery should be evaluated during validation. Poor recovery rates are often found for volatile elements such as boron (B), arsenic (As), antimony (Sb), mercury (Hg), gold (Au), and tungsten (W) because of the relatively high temperature sample preparation method and poor stability of some elements in aqueous solution. Elements forming volatile halogenides can be affected due to the in-situ production of hydrogen halogenides when halogenated polymers are ashed.

3.4 This is a bulk analysis technique. For leachable testing or surface analysis refer to the Related Documents (§ 16) of this method.

3.5 Due to the rapid advances in digestion technology, consult the manufacturer's recommended instructions for guidance when conducting analyses using the DCV sections of this document.

3.6 DCV techniques can generate gaseous digestion reaction products, very reactive, or volatile materials at high pressures. Spontaneous venting which can occur during sample heating may cause venting of the vessels with potential loss of sample and analytes. Sample sizes greater than 0.25 g may accentuate this event.

3.7 In the use of the DCV technique, TiO_2, alumina, and other oxides may not be totally dissolved. Sequestering of target analyte elements may occur.

3.8 Although this method allows the sampling of small pieces of polymer that are mechanically removed from a larger item, obtaining such samples in a clean manner may be difficult. Multiple sampling, separation and preparation techniques might be necessary to establish confidence in the results.

3.9 This document is not intended to supersede international, national or local codes, regulations, and laws. Each should be consulted to ensure that the method meets regulatory requirements in each location.

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4 Referenced Standards and Documents

4.1 *SEMI Standard*

SEMI F40 — Practice for Preparing Liquid Chemical Distribution Components for Chemical Testing

4.2 *ASTM Standard*[1]

ASTM D4375 — Standard Practice for Basic Definitions, Notation, and Symbology for Statistics in Committee D19 on Water

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

5 Terminology

5.1 *Abbreviations and Acronyms*

5.1.1 *AAS/GFAAS* — atomic absorption spectroscopy/graphite furnace atomic absorption spectroscopy

5.1.2 *amu* — atomic mass unit

5.1.3 *DCV* — digestion in closed vessel

5.1.4 *DDA* — digestion by dry ashing

5.1.5 *GFAAS* — graphite furnace atomic absorption spectroscopy

5.1.6 *ICP-AES* — inductively coupled plasma-atomic emission spectroscopy

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

5.1.8 *ppb* — parts per billion by mass (ng/g)

5.1.9 *ppm* — parts per million by mass (µg/g)

5.1.10 *UPW* — ultrapure water (see § 9.4)

6 Summary of Test Method

6.1 Samples previously prepared using SEMI F40 are ashed or digested under pressure within a digestion device, and trace inorganics in the residue are dissolved into acid and UPW. The sample is then analyzed by ICP-MS, GFAAS, and/or ICP-AES to determine the inorganic content of the material. This method applies only to nonvolatile metals (i.e., alkali metals, alkaline earths, and transition metals).

6.2 Data from different tests can be compared to determine the inorganic content in different materials and in the same material from different manufacturers.

7 Significance and Use

7.1 Determining the metallic contamination concentration in bulk polymer materials used in either distribution systems for process fluids or products in direct contact with the wafer is important criterion for deciding the suitability of a material. For example, ultrapure water contaminated by distribution system components may adversely affect microelectronic and other processes.

7.2 This method measures the total amount of impurities in the bulk of the material. These impurities will not necessarily leach into a process fluid stream.

8 Apparatus

8.1 *Muffle Furnace* — With temperature control ranging up to a minimum of 700°C and equipped with a means to regulate air circulation.

8.2 Microwave Muffle Furnace

1 American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585; Fax: 610.832.9555; http://www.astm.org

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8.3 *Crucibles* — Made of either platinum or quartz and with a 30 mL capacity.

8.4 ICP-MS

8.5 GFAAS

8.6 *ICP-AES* — Either simultaneous or sequential reading type.

8.7 Chemical Fume Hood

8.8 Propane Torch or Appropriate Heating Source with a minimum temperature of 650°C.

8.9 Device for digestions under a pressure of at least 30 bar (435 psi), with temperature control. This can be a laboratory microwave-based system or a system based on other heating sources.

8.9.1 In the case of microwave digestion devices: Laboratory microwave digestion systems should be used that possess appropriate temperature control of chemical reactions. Closed microwave systems must have controlled pressure relief.

8.9.2 Digestion vessels of appropriate internal volume should be used and construction should be of appropriate chemically inert materials. If the vessel is pressurized, it should be capable of withstanding a minimum pressure of 30 atm (30 bar or 435 psi), with controlled pressure relief of reagents and digestion products.

NOTE 1: Only microwave manufacturer's approved vessels for that device should be used.

8.9.3 In case of a laboratory microwave digestion device: Oscillating turntable to insure homogeneous distribution of microwave radiation to all vessels.

8.9.4 Filter paper, qualitative or equivalent.

8.9.5 Filter funnel, polypropylene, polyethylene or equivalent.

8.10 Volumetric flasks, 20 mL or 50 mL capacity or equivalent.

8.11 Analytical balance, of appropriate capacity, with a ±0.0001 g or appropriate precision for the weighing of the sample. Optionally, the vessel with sample and reagents may be weighed, with an appropriate precision balance, before and after microwave processing to evaluate the seal integrity in some vessel types.

9 Materials

9.1 Argon Gas 99.99% pure or better.

9.2 *Standards and Reference Materials*

9.2.1 For preparation of multi-element standard solutions, use NIST,[2] NIST-traceable, or other appropriate international standards as stock solutions.

9.2.2 From these stock solutions, multi-element working standard solutions must be prepared daily by pipeting the appropriate volumes of the trace metal standards and diluting to the desired concentrations.

NOTE 2: Prepare these working standards using the same amount of acid as used for the sample.

9.2.3 For validation purposes, use appropriate international reference materials that match the sample matrix as close as possible.

9.3 All reagents should be of appropriate purity or high purity (acids for example, should be sub-boiling distilled where possible) to minimize the blank levels due to elemental contamination. If the purity of a reagent is questionable, analyze the reagent to determine the level of impurities. The reagent blank must be less than the minimum detection limit in order to be used.

9.3.1 Ultrapure Hydrochloric Acid less than 1 ppb for each trace metal.

9.3.2 Ultrapure Nitric Acid less than 1 ppb for each trace metal.

2 National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899-3460, USA. Telephone: 301.975.6478; http://www.nist.gov

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