

SEMI F114-0220

TEST METHOD FOR THE DETERMINATION OF ORGANIC CONTAMINANTS PRESENT ON WETTED SURFACES OF ULTRA HIGH PURITY CHEMICAL DELIVERY SYSTEMS AND COMPONENTS

This Standard was technically approved by the Gases Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on December 12, 2019. Available at www.semiviews.org and www.semi.org in February 2020.

1 Purpose

1.1 This Document defines a test method for determining organic compounds on the wetted surfaces of ultra high purity (UHP) chemical delivery systems and components.

2 Scope

2.1 This Test Method is applicable to UHP chemical delivery systems and components. Examples of test samples include valves, regulators, filters, and mass flow controllers, tubing, weld fittings, and face seal fittings.

2.2 This Test Method could also be used to determine the contamination contributed from an assembly or fabrication area.

2.3 This Document details the method to extract and measure organic contaminants present on the wetted surfaces.

2.4 This Test Method could be used to evaluate the effectiveness of various flushing or purging processes.

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 This Test Method is limited to metallic components of which the majority are comprised of SEMI F20 or similar stainless steels. Other metals, such as nickel-based alloys, used for construction of UHP components can also be evaluated using this method. This method does not apply to polymer based components or systems.

3.2 This Test Method is intended for use by operators who understand the use of the test apparatus and equipment at a level equivalent to twelve months of experience.

3.3 This Test Method can be used for UHP components with wetted elastomeric materials. However, the analysis results can be affected by elastomeric materials. In order to avoid such interference, it requires the generation or obtaining of the spectra or fingerprint of the elastomeric material separately, then the deduction of the elastomeric material from the analysis results.

3.4 This Test Method is also limited to components compatible with the test fluid. Certain polymeric materials can dissolve or degrade in the test fluid.

3.5 This Test Method will not provide any recommendations for flushing or purging steps.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI E49.8 — Guide for High Purity and Ultrahigh Purity Gas Distribution Systems in Semiconductor Manufacturing Equipment

SEMI F20 — Specification for 316L Stainless Steel Bar, Forgings, Extruded Shapes, Plate, and Tubing for Components Used in General Purpose, High Purity and Ultra-High Purity Semiconductor Manufacturing Applications

4.2 ISO Standard¹

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

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

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *CAD* — computer aided design

5.1.2 *FTIR* — Fourier transform infrared spectroscopy

5.1.3 *GC-MS* — gas chromatography-mass spectrometry

5.1.4 *MFC* — mass flow controller

5.1.5 *NVR* — non-volatile residue

5.1.6 *UHP* — ultra high purity

5.2 Definitions

5.2.1 *contaminant* — a undesirable substance on the wetted surfaces of UHP chemical delivery systems and components that can be transferred to the process fluid.

5.2.2 *leachate* — the organic solution obtained from the leaching process which includes the test fluid and the compounds extracted from the wetted surfaces of UHP chemical delivery systems and components.

5.2.3 *test component* — an individual piece or a complete assembly of individual pieces being tested, including industrial products that are manufactured as independent units, capable of being joined with other pieces or components. Example of these components include valves, fittings, regulators, tubing, mass flow controllers, and gas delivery tubing and fittings.

6 Apparatus

6.1 *Gas Chromatography-Mass Spectrometry (GC-MS)* — An analytical instrument that detects volatile and semi-volatile organic contaminants in the gas phase. A liquid is injected and heated to vapor phase. The sample is then passed through a column containing an adsorbent. A carrier gas is used as the mobile phase. The retention times of the various peaks help to identify the organic species. The area under the respective peak yields the quantity of the species in the mobile phase. The GC-MS used in this Test Method must be calibrated using standard laboratory practices and manufacturer's recommendations.

6.2 *Fourier Transform Infrared Spectroscopy (FTIR)* — An analytical instrument that qualitatively or quantitatively identifies non-volatile residue (NVR) contaminants based on characteristic frequencies of adsorption of infrared radiation. The FTIR used in this Test Method must be calibrated using standard laboratory practices and manufacturer's recommendations.

6.3 *Scale* — An instrument that qualitatively measure the weight of NVRs. Sensitivity of the scale is critical to the accuracy of the measurement. A minimum sensitivity of 0.001 mg of the scale is recommended.

7 Reagents and Materials

7.1 *Test Fluid* — A common extraction fluid (solvent) used for organic analysis. A recommended test fluid is methylene chloride (CH_2Cl_2). The purity of methylene chloride is critical to the overall sensitivity of the test. For good lot-to-lot consistency, 99.9% purity (GC grade) methylene chloride is recommended. Storage in glass containers is also recommended.

¹ 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>

7.2 *Caps* — Used to seal the ends of the test component and shall be made of 316L stainless steel. For tube stub ended components, 316L stainless steel compression fittings with metal ferrules shall be used. For face seal fittings, stainless steel gaskets shall be used. All the caps used in this Test Method shall be cleaned to UHP levels (see SEMI E49.8).

7.3 *Gloves* — Cleanroom compatible and test fluid resistant gloves shall be used in this Test Method.

8 Sample Preparation

8.1 Extreme care shall be exercised to prevent adding contamination to the test component. Cross contamination from the exterior of the test component to the wetted surface is of utmost concern. Identify every use of plastic materials that can be wetted during the testing. The plastic materials for sealing and their grade shall be specified so that they are compatible with the desired level of process sensitivity of the test method.

8.2 The test component shall be in the condition to be studied. Since environmental factors will play a role in the contamination of the items to be tested, the testing plan shall take into consideration the time of exposure to ambient air, etc. For example, new components would likely be tested immediately after removal from the packaging to evaluate the “as delivered” contamination level. For UHP chemical delivery systems, testing should occur after final assembly or tubing preparation. Test component condition, exposure time to atmosphere (if applicable), etc. should be clearly identified in the test report.

8.3 Clean metal plugs and caps are recommended for cavity sealing. Materials used for caps and plugs should match the article of study, and, if possible, be isolated from the test fluid. An example of isolating caps and plugs from the test fluid would be the use of unplated blind face seal gaskets. For caps and plugs that cannot be isolated from the test fluid, an additional analysis of those items will be needed to quantify the additive contaminants, then deduct from the final analysis results.

8.4 For the following components, the following preparation steps shall be used:

8.4.1 *Regulators* — Rotate the handle fully clockwise (or fully counter-clockwise for a back pressure regulator) to open the regulator. Filling may require adding fluid from both sides due to the small orifice size between the high and low pressure sides of the regulator to ensure interior surfaces are wetted with test fluid. If the sample volume is sufficient for testing, separate test samples shall be taken from both the inlet and outlet sides. If the volume is insufficient for testing, combine the inlet and outlet sample fluid volumes.

8.4.2 *Valves* — All valves shall be opened fully before starting the test. If the valve has an internal orifice $\leq 20\%$ of the nominal tube diameter, filling and extracting shall be done from each end to ensure the interior surfaces have been wetted with the test fluid. If the volume is insufficient for testing, combine the inlet and outlet sample fluid volumes.

8.4.3 *Filters* — Cap the outlet end, rotate the filter so that the inlet is facing up and fully fill the inlet with test fluid. Then cap the inlet end, rotate the filter so that the outlet is facing up and fully fill. Some slight shaking and tilting may be necessary to allow air bubbles to escape. Samples shall be taken from both ends and analyzed separately. If the fluid volume is insufficient for testing, combine the inlet and outlet sample fluid volumes.

8.4.4 *Mass Flow Controller (MFC)* — Applying power to fully open the device is not recommended due to the hazard of the use of liquids around electrically energized components. Extra time and care will be required to fill all the internal cavities. For MFCs with a downstream control valve, cap the inlet, rotate the MFC so that the outlet is facing up and fully fill the outlet with test fluid. Then cap the outlet, rotate the MFC so that the inlet is facing up and fully fill with test fluid. Some slight shaking and tilting may be necessary to allow air bubbles to escape. The MFC construction contains internal structures that will require extra time to fill. Samples shall be taken from both ends and analyzed separately.

8.4.5 Additional precautions to avoid the formation or retention of bubbles may be necessary. Using a syringe to slowly supply the test fluid and/or using a vacuum to remove air before introducing the test fluid are suggested methods.

8.4.6 Striking any component to remove bubbles is not recommended.

9 Procedure

9.1 Background

9.1.1 The environment in which the sample preparation and analysis is performed will have a direct impact on the detection limits because of background levels. A cleanroom (minimum ISO Class 5 or equivalent) environment is recommended for better sensitivity.