

**SEMASPEC Test Method for  
Determination of Ionic/Organic  
Extractables of Internal Surfaces -  
IC/GC/FTIR for Gas Distribution  
System Components**

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# SEMASPEC Test Method for Determination of Ionic/Organic Extractables of Internal Surfaces - IC/GC/FTIR for Gas Distribution

## System Components

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**Abstract:** This SEMASPEC establishes a method for testing components used in ultra-high purity gas distribution systems for ionic and organic surface residues. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation.

This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

**Keywords:** Contamination, Contaminants, Gaseous Contaminants, Defect Sources, Facilities, Gas Distribution Systems, Specifications, Components, Component Testing

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## SEMASPEC #90120399B-STD

### SEMASPEC Test Method for the Determination of Ionic/Organic Extractables of Internal Surfaces – IC/GC/FTIR for Gas Distribution System Components

#### 1. Introduction

Semiconductor cleanrooms are serviced by high-purity gas distribution systems. This document presents a test method that may be applied for the evaluation of one or more components considered for use in such systems.

##### 1.1 *Purpose*

1.1.1 The purpose of this document is to define a method for testing electropolished stainless steel components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for this installation.

1.1.2 This document establishes a method for testing components used in ultra-high purity gas distribution systems for ionic and organic surface residues.

1.2 *Scope*—This procedure applies to in-line components handling electronics grade gases.

##### 1.3 *Limitations*

1.3.1 This procedure is limited by the sensitivity of the detection instruments and by the available levels of purity in extracting solvents. While the ion and gas chromatographic methods are quantitative, the FTIR method can be used as either a qualitative or a quantitative tool. In addition, the GC and FTIR methods are used to detect hydrocarbons and halogenated substances that remain as residues on component internal surfaces. This eliminates those materials with high vapor pressures, which are analyzed per the total hydrocarbons test, from this procedure (see SEMASPEC #90120396B-STD).

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

The measurement of extractables as measured by this method may not directly correspond to contamination found when in gas service.

1.3.2 This method is written with the assumption that the operator understands the use of the apparatus at a level equivalent to twelve months of experience.

#### 2. Reference Documents

2.1 Linde Division, Union Carbide. "Techniques for Measuring Trace Gas Impurities in High Purity Gases." Linde. 1984.

2.2 ASTM E1151–87<sup>1</sup> Standard Practice for Ion Chromatography Terms and Relations, 1987.

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<sup>1</sup> American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

### 3. Terminology

- 3.1 *FTIR*—fourier transform infrared spectroscopy.
- 3.2 *GC*—gas chromatography.
- 3.3 *IC*—ion chromatography.
- 3.4 *IPA*—isopropanol (2-propanol).
- 3.5 *MS*—mass spectrometry
- 3.6 *ppbv*—parts per billion by volume (such as nl/l).
- 3.7 *ppbw*—parts per billion by weight (such as ng/g).
- 3.8 *ppmv*—parts per million by volume (such as  $\mu\text{l/l}$ ).
- 3.9 *ppmw*—parts per million by weight (such as  $\mu\text{g/g}$ ).
- 3.10 *psi*—pounds per square inch.
- 3.11 *psia*—pounds per square inch absolute.
- 3.12 *psid*—pounds per square inch differential.
- 3.13 *psig*—pounds per square inch gauge.
- 3.14 *scfm*—standard cubic feet per minute.
- 3.15 *slpm*—standard liter per minute. The gas volumetric flow rate measured in liters per minute at 0.0 °C (32 °F) and 101.3 kPa (1 atm).
- 3.16 *spool piece*—a null component, consisting of a straight piece of electropolished tubing and appropriate fittings, used in place of the test component to establish the baseline.
- 3.17 *standard conditions*—101.3 kPa, 0.0 °C (14.73 psia, 32 °F).
- 3.18 *test component*—any device being tested, such as a valve, regulator, or filter.
- 3.19 *test fluid blank*—a volume of test solvent adequate for analysis. This is used to determine the background impurity concentrations in the test fluid. This fluid is drawn at the same time as the fluid that is used to fill the spool piece and test component. It must be held in a container which does not contaminate the fluid blank.

## 4. Test Protocol

### 4.1 Test Conditions

#### 4.1.1 Precautions

4.1.1.1 This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations before using this method.

4.1.1.2 It is required that the user have a working knowledge of the respective instrumentation and proper handling of test components for trace analysis. Good laboratory practices must also be followed.

4.1.1.3 Use safety precautions such as proper ventilation and disposal when handling solvents.

4.1.1.4 Gloves are to be worn at all times. For ionic extractions, gloves are to be rinsed in the test fluid before use; for organic extractions, unrinsed gloves will be used. Contact of the gloves with the test fluid invalidates the test.

4.1.2 *Temperature*—The test component and the spool piece are to be tested at a constant temperature ( $\pm 4$  °C) in the range of 18 °C (64 °F) to 26 °C (78 °F). Solvents used must be at the same temperature.

### 4.2 Apparatus

#### 4.2.1 Materials

4.2.1.1 *Test fluid*—The purity of fluid used for the extractions will determine the detection limit of the test. Therefore, extremely high purity fluids are required. At least 18.0 M $\Omega$  water [corrected for 18 °C (64 °F)] must be used for the determination of extractable cations. The test fluid for anion analysis should be anion eluent prepared in at least 18.0 M $\Omega$  water. [18.0 M $\Omega$  water may be used for anion analysis if no water interference (dip) is present with the particular column used.] Total oxidizable carbon must be less than 50 ppbw. The water must be filtered through a 0.2  $\mu$ m (or finer) filter. High purity (distilled in glass or pesticide grade) methylene chloride (MeCl<sub>2</sub>) is to be used for the organic extractables determination.

4.2.1.2 The spool piece shall be a straight run of electropolished 316L stainless steel tubing with no restrictions. The length of the spool piece shall be approximately 200 mm. The spool piece should be the same diameter as the test component. The spool piece can be used for multiple components tested concurrently.

4.2.1.3 Caps used to seal the ends of the test component and spool piece are to be of 316L stainless steel. For stub end components, 316L stainless steel compression fittings with nylon or teflon ferrules are to be used. For face seal fittings, stainless steel gaskets must be used.

4.2.1.4 Gloves are to be made of powder free vinyl or polyethylene.

#### 4.2.2 Instrumentation

4.2.2.1 *Ion chromatograph*—The ion chromatograph (IC) is an analytical instrument that detects ionic species in deionized (DI) water. The eluent is passed through a column containing ion exchange resin. A conductivity detector is used to detect the ionic species. The retention times of the various ionic species are used to identify the species. The area under the respective peak yields the quantity of the species in the eluant. This method uses a column for mono- and polyvalent anions and a column for mono- and polyvalent cations. A suppressor column may be used to increase sensitivity.

4.2.2.2 *Gas chromatograph*—The gas chromatograph (GC) is an analytical instrument that detects organic species in the gas phase. A liquid sample is injected and heated to the 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. Since the peak of the solvent will be large, it will obscure those species that have a carbon number below that of the solvent.

4.2.2.3 *Fourier transform infrared spectrometer*—The Fourier transform infrared spectrometer (FTIR) is an analytical instrument that qualitatively or quantitatively identifies functional groups based on characteristic frequencies of absorption of infrared radiation. By identifying combinations of absorption frequencies, identification of contaminants can be made.

4.2.2.4 *Instrument calibration*—Instruments shall be calibrated using standard laboratory practices and manufacturer's recommendations.

4.2.3 Setup and Schematic - per instructions of instrument manufacturer.

#### 4.3 Test Procedure

4.3.1 Rinse the outside of the spool piece with the test fluid to be used for analysis. Rinse the caps and rinse any gaskets or ferrules to be used with the respective test fluid.

4.3.2 Remove the caps accompanying the spool piece. Install the stainless steel cap, rinsed as in Sections 4.3.1, on one end of the spool piece. Using a burette or graduated pipette, measure the amount of test fluid required to completely fill the internal volume of the spool piece less a minimal air space. Extreme care must be taken to avoid overfilling the spool piece. Overfilling invalidates the test. Cap the open end.

4.3.3 Invert the spool piece 20 times across the long axis, one inversion every 30 seconds. Allow the spool piece to rest along the horizontal axis for 18 to 24 hours at a constant temperature ( $\pm 4$  °C). (See Section 4.1.2.)

- 4.3.4 After 18 to 24 hours, invert the spool piece 20 times across the long axis, one inversion every 30 seconds.
- 4.3.4.1 Storage of samples and corresponding blanks in an external container is permitted for a maximum time period of two weeks, under refrigeration, provided suitable precautions are maintained to insure no increase or decrease in concentration of the analytes occurs.
- 4.3.5 Remove one cap and sample the fluid, using a glass syringe fitted with a stainless steel needle. Recap the spool piece immediately to limit evaporation of the solvent. If multiple samples are to be run, agitate the spool piece (4 inversions per 4.3.4) prior to each sample.
- 4.3.6 If any species are detected, the spool piece is to be cleaned with the appropriate test fluid and the analysis repeated. Those species still remaining are taken to be the background contribution of the test fluid.
- 4.3.7 Repeat Sections 4.3.1 to 4.3.5 simultaneously with the test component and the spool piece.
- 4.3.8 For the following components, observe the given guidelines.
- 4.3.8.1 *Regulators*—Wind the handle fully counter clockwise (or fully clockwise for a back pressure regulator). This will close the regulator. Fully fill the inlet side first with test fluid. Then cap the inlet end. Turn the regulator over and prepare to fill the outlet end. Prior to adding the test fluid, turn the handle in the reverse direction to open the regulator. Now fully fill the outlet end. Cap the outlet and begin the extraction. During extraction, the regulator must be resting on its side. Due to the small orifice separating the high and low pressure sides of a regulator, samples should be taken from both the inlet and outlet. If taking samples from both ends of the regulator results in a sample volume less than 1.5 mL, combine the inlet and outlet samples.
- 4.3.8.2 *Valves*—All valves should be fully open before starting the test. If the minimum orifice is <20% of the tube nominal diameter, samples should be taken from both ends. If taking samples from both ends of the valve results in a sample volume less than 1.5 mL, combine the inlet and outlet samples.
- 4.3.8.3 *Electropolished Tubing*—An entire random length (stick) of tubing must be used for this test. This is to assure that the sample is representative of the entire tube. If it is not possible to invert the sample, it may be rolled.
- 4.3.8.4 *Filters*—With the outlet end capped, fully fill the inlet side with test fluid. Then cap the inlet end. Turn the filter over and fill the outlet end. During the filling operation, slightly tilt and gently shake the filter to release air bubbles that may be trapped in the filter structure. Cap the outlet and begin the extraction. During extraction, the filter must be resting on its side. Samples should be taken from both the inlet and outlet.

- 4.3.9 Analyze as follows, using the appropriate method.
- 4.3.9.1 *IC*—Inject the water sample into the IC using the appropriate injection method. Use three injections for cations and three injections for anions. Record the identification and quantify by the appropriate mathematical method.
- 4.3.9.2 *GC*—Inject the MeCl<sub>2</sub> sample into the GC using the appropriate injection method. Use three injections. Record the identification and quantify by appropriate mathematical method. Only analyze for those components resolved with retention times greater than the solvent MeCl<sub>2</sub> peak.
- 4.3.9.3 *FTIR*—Analyze the sample by the appropriate method to minimize solvent interference peaks (such as evaporation on a salt plate).
- 4.4 *Data Analysis*
- 4.4.1 *IC*—The IC data is to be reported as micrograms of ion per liter extract fluid, tabulated by ionic species (e.g., Na<sup>+</sup>, SO<sub>4</sub><sup>-2</sup>). The data is also to be reported as mass of each ionic species per test component in the same table.
- 4.4.2 *GC*—The GC data is to be reported as total mass of organics detected per liter extract fluid and total mass of organics per test component.
- 4.4.3 *FTIR*—The FTIR data is to be reported in tabular form, listing frequency of absorption band and organic specie with matching characteristic absorption bands. Analysis of the spectrum leading to identification of materials in the residue will also include reference spectra.
- 4.4.4 The spectra are to be attached for reference. A proper subtraction of the fluid blank spectrum from the sample spectra is acceptable, provided that proper interpretation techniques are followed and the spool piece and fluid blank spectra are virtually identical.



**Table 1 (continued) Ionic/Organic Contribution** (page two of four)

Test Component		
Species:	µg/l Water	µg/Test Component
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Analyzer: \_\_\_\_\_

Model Number: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Last Calibration Date: \_\_\_\_\_

Test Parameters:

Column: \_\_\_\_\_

Elution System: \_\_\_\_\_

Detector: \_\_\_\_\_

**Table 1 (continued) Ionic/Organic Contribution** (page three of four)

## II. Organic Residue by gas chromatography

Methylene Chloride

Grade: \_\_\_\_\_

Manufacturer: \_\_\_\_\_

Lot Number: \_\_\_\_\_

Spool Piece Baseline

Retention Time	$\mu\text{g/l MeCl}_2$	$\mu\text{g/Test Component}$
----------------	------------------------	------------------------------

_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Test Component

Retention Time:	$\mu\text{g/l MeCl}_2$	$\mu\text{g/Test Component}$
-----------------	------------------------	------------------------------

_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Analyzer: \_\_\_\_\_

Model Number: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Last Calibration Date: \_\_\_\_\_

Test Parameters:

Column: \_\_\_\_\_

Column Temperature: \_\_\_\_\_

Injector Temperature: \_\_\_\_\_

Detector: \_\_\_\_\_

Flow: \_\_\_\_\_

**Table 1 (continued) IC/GC/FTIR Contribution Data Table** (page four of four)

## III. Organic Residue by Fourier transform infrared spectroscopy

Methylene Chloride

Grade: \_\_\_\_\_

Manufacturer: \_\_\_\_\_

Lot Number: \_\_\_\_\_

Spool Piece Baseline

Frequency (cm <sup>-1</sup> )	Functional Group
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Test Component

Frequency (cm <sup>-1</sup> )	Functional Group
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Analyzer: \_\_\_\_\_

Model Number: \_\_\_\_\_

Serial Number: \_\_\_\_\_

Last Calibration Date: \_\_\_\_\_

Test Parameters:

Detector: \_\_\_\_\_

Cell: \_\_\_\_\_

Method: \_\_\_\_\_

NOTE: Attach spectra and reference spectra.

**X1. Alternative Tests**

- X1.1 *Liquid chromatography/Mass spectrometry (LC/MS)*—In addition to gas chromatography and Fourier transform infrared spectroscopy, these are quantitative and/or qualitative methods that allow resolution and identification of organic compounds.
- X1.2 *Quadrupole mass spectrometer (QMS)*—The quadrupole mass spectrometer accurately detects hydrocarbon species by ionizing organic moieties in a vacuum. The ionized fragments are detected semi-quantitatively and individually.
- X1.3 *Graphite furnace atomic absorption spectroscopy (GFAAS)*—This instrument quantitatively determines the mass of species based on their absorption of specific wavelengths. An elemental lamp emits the specific radiation, which is passed to a photo-detector after being absorbed by the sample in the furnace. It is highly sensitive to cations but limited in scope for anions.
- X1.4. *Inductively coupled plasma atomic emission spectroscopy (ICP–AES)*—This instrument quantitatively determines the mass of the species based on the emission spectra of the sample placed in the plasma. It is highly sensitive to cations but limited in scope for anions.

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