



Standard Test Method for Ionic/Organic Extractables of Internal Surfaces-IC/GC/FTIR for Gas Distribution System Components¹

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INTRODUCTION

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

1. Scope

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

1.2 This test method applies to in-line components containing electronics grade materials in the gaseous form.

1.3 Limitations:

1.3.1 This test method 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 Fourier transform infrared spectroscopy (FTIR) method can be used as either a qualitative or a quantitative tool. In addition, the gas chromatography (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 test method.

1.3.2 This test method is intended for use by operators who understand the use of the apparatus at a level equivalent to twelve months of experience.

1.4 The values stated in SI units are to be regarded as the standards. The inch-pound units given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 ASTM Standards:

E 1151 Practice for Ion Chromatography Terms and Relationships²

2.2 Union Carbide Standard:

Techniques for Measuring Trace Gas Impurities in High Purity Gases³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *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.

3.1.2 *standard conditions*—101.3 kPa, 0.0°C (14.73 psia, 32.0°F).

3.1.3 *test component*—any device being tested, such as a valve, regulator, or filter.

3.1.4 *test fluid blank*—a volume of test solvent adequate for analysis.

3.1.4.1 *Discussion*—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 that does not contaminate the fluid blank.

3.2 Abbreviations:

3.2.1 *FTIR*—Fourier transform infrared spectroscopy.

3.2.2 *GC*—gas chromatography.

3.2.3 *IC*—ion chromatography.

3.2.4 *IPA*—isopropanol (2-propanol).

3.2.5 *MS*—mass spectrometry.

3.2.6 *ppbv*—parts per billion by volume (such as nL/L).

3.2.7 *ppbw*—parts per billion by weight (such as ng/g).

3.2.8 *ppmv*—parts per million by volume (such as μ L/L).

3.2.9 *ppmw*—parts per million by weight (such as μ g/g).

3.2.10 *psid*—pounds per square inch differential.

3.2.11 *scfm*—standard cubic feet per minute.

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² *Annual Book of ASTM Standards*, Vol 14.02.

³ Available from Linde Division Union Carbide, 175 E. Park Drive, Tonawanda, NY 14151.

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.

NOTE 1—Since the peak of the solvent will be large, it will obscure those species that have a carbon number or retention time below that of the solvent.

5.2.3 *Fourier transform infrared spectrometer*—The FTIR is an analytical instrument that qualitatively or quantitatively identifies contaminants based on characteristic frequencies of absorption of infrared radiation. By identifying combinations of absorption frequencies, identification of contaminants can be made (see Appendix X1).

6. Hazards

6.1 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.

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

6.3 Gloves are to be worn at all times. After use, the gloves are to be rinsed in the appropriate test fluid.

NOTE 2—An alternative could be the use of disposable gloves, using a new glove for each test.

7. Calibration

7.1 Calibrate instruments using standard laboratory prac-

III. Organic Residue by Fourier transform infrared spectroscopy

Isopropanol

Grade: _____

Manufacturer: _____

Lot Number: _____

Spool Piece Baseline

Frequency (cm ⁻¹)	Identity	Library Reference
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Test Component

Frequency (cm ⁻¹)	Identity	Library Reference
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Analyzer: _____

Model Number: _____

Serial Number: _____

Last Calibration Date: _____

Test Parameters:

Detector: _____

Cell: _____

Method: _____

NOTE: Attach spectra and reference spectra.

FIG. 3 Ionic/Organic Contribution Data Table Illustration

tices and manufacturer's recommendations.

8. Test Procedure

8.1 *Temperature*—The test component and the spool piece are to be tested at a constant temperature $\pm 2^\circ\text{C}$ in the range of 26°C (18 to 78°F). Solvents used must be at the same temperature.

8.2 Rinse the outside of the spool piece with the test fluid (DI water (ionic) or IPA (organic)) to be used for analysis. Rinse the caps and rinse any gaskets or ferrules to be used with the respective test fluid.

8.3 Remove the caps accompanying the spool piece. Install the stainless steel cap, rinsed as in 8.1, on one end of the spool piece. Using a buret or graduated pipet, measure the amount of test fluid required to completely fill the internal volume of the spool piece. Extreme care must be taken to avoid overfilling the spool piece. Overfilling invalidates the test. Cap the open end.

8.4 Invert the spool piece 20 times across the long axis, one inversion every 30 s. Allow the spool piece to rest along the horizontal axis for 24 h at a constant temperature $\pm 2^\circ\text{C}$ (see 8.1).

NOTE 3—Since air is likely to be introduced when capping, the inversion insures uniformity of the fluid.

8.5 After 24 h, invert the spool piece 20 times across the long axis, one inversion every 30 s.

8.6 Remove one cap and sample the fluid. Recap the spool piece immediately to limit evaporation of the solvent. If multiple samples are to be run, agitate the spool piece (four inversions in accordance with 8.4) prior to each sample.

8.7 If any species are detected, the spool piece is to be cleaned with the appropriate test fluid and analysis to be repeated until it is suitable for use as a control.

8.8 Repeat 8.2-8.6 simultaneously with the test component and the spool piece.

8.9 For the following components, observe the given guidelines.

8.9.1 *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, take samples from both the inlet and outlet.

8.9.2 *Valves*—Make sure that all valves are fully open before starting the test. If the minimum orifice is $<20\%$ of the tube nominal diameter, take samples from both ends.

8.9.3 *Electropolished Tubing*—An entire random length (stick) of tubing must be used for this test. This is to ensure that the sample is representative of the entire tube. If it is not possible to invert the sample, it may be rolled.

8.9.4 *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. Take samples from both the inlet and outlet.

8.10 Analyze as follows, using the appropriate method:

8.10.1 *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.

8.10.2 *GC*—Inject the IPA 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 IPA peak.

8.10.3 *FTIR*—Analyze the sample by the appropriate method to minimize solvent interference peaks (such as evaporation on a salt plate).

9. Report

9.1 Report the following information:

9.1.1 *IC*—Report the IC data as micrograms of ion per litre extract fluid, tabulated by ionic species (for example Na⁺, SO₄⁻²). Also report the data as mass of each ionic species per test component in the same table, (see Fig. 1),

9.1.2 *GC*—Report the GC data as total mass of organics detected per litre extract fluid and total mass of organics per test component, (see Fig. 2),

9.1.3 *FTIR*—Report the FTIR data 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, (see Fig. 3), and

9.2 Attach the spectra 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.

10. Precision and Bias

10.1 Precision and bias for this test method are being determined.

11. Keywords

11.1 components; contamination; extraction; gas distribution components; ionic extractables; organic extractables; surface contamination

APPENDIX

(Nonmandatory Information)

X1. ALTERNATIVE TESTS

X1.1 *Liquid Chromatography/Mass Spectrometry (LC/MS)*—In addition to gas chromatography and Fourier transform infrared spectroscopy, these are quantitative or qualitative methods, or both, 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 that is passed to a photodetector 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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