Designation: F1374 – 92 (Reapproved 2020)

Standard Test Method for Ionic/Organic Extractables of Internal Surfaces-IC/GC/FTIR for Gas Distribution System Components[1]

This standard is issued under the fixed designation F1374; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1151 Practice for Ion Chromatography Terms and Relationships

2.2 *Union Carbide Standard:*

Techniques for Measuring Trace Gas Impurities in High Purity Gases[3]

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.

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.10 on Contamination Control.

Current edition published April 15, 2020. Published May 2020. Originally approved in 1992. Last previous edition approved in 2012 as F1374–92(2012). DOI: 10.1520/F1374-92R20.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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 µL/L).

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

3.2.10 *psid*—pounds per square inch differential.

3.2.11 *scfm*—standard cubic feet per minute.

3.2.12 *slpm*—standard litre per minute. The gas volumetric flow rate measured in litres per minute at 0.0°C (32°F) and 101.3 kPa (1 atm).

4. Significance and Use

4.1 The purpose of this test method is to define a procedure 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.

5. Apparatus

5.1 *Materials:*

5.1.1 *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 MΩ water [corrected for 18°C (64°F)] must be used for the ionic extractables determination. Total oxidizable carbon must be less than 50 ppbw. The water must be filtered through a 0.2-µm (or finer) filter. Electronics grade (or better) IPA is to be used for the organic extractables determination.

5.1.2 *Spool Piece*— a straight run of electropolished 316-L 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.

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

5.1.4 *Gloves*—made of powder free latex or natural rubber and resistant to the test fluids used in this test method.

5.2 *Instrumentation:*

5.2.1 *Ion Chromatograph*—The IC is an analytical instrument that detects ionic species in deionized (DI) water. The eluant 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 test 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.

Date: _____
Operator: _____
Test Location: _____
Spool Piece ID: _____
Test Component: _____
Model Number: _____
Serial Number: _____
Ambient Temperature: _____ °C
Start Time/Date of Soak: _____
Finish Time/Date of Soak: _____
I. Ionic Contribution by ion chromatography
D.I. Water
Resistivity: _____
TOC: _____
Spool Piece Baseline

Species:	µg/l Water	µg/Test Component

Test Component

Species:	µg/l Water	µg/Test Component

Analyzer: _____
Model Number: _____
Serial Number: _____
Last Calibration Date: _____
Test Parameters:
Column: _____
Elution System: _____
Detector: _____

FIG. 1 Ionic/Organic Contribution Data Table Illustration

5.2.2 *Gas chromatograph*—The 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.

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

II. Organic Residue by gas chromatography
Isopropanol
Grade: _____
Manufacturer: _____
Lot Number: _____

Spool Piece Baseline Retention Time:	µg/l IPA	µg/Test Component

Test Component Retention Time:	µg/l Water	µg/Test Component

Analyzer: _____
Model Number: _____
Serial Number: _____
Last Calibration Date: _____
Test Parameters:
Column: _____
Column Temperature: _____
Injector Temperature: _____
Detector: _____
Flow: _____

FIG. 2 Ionic/Organic Contribution Data Table Illustration

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

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 practices 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 ± 2°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 ± 2°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.