



Designation: F1374 – 92 (Reapproved 2012)

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

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 and health practices and determine the applica-*

bility of regulatory limitations prior to use. Specific hazard statements are given in Section 6.

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.

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.

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.

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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.

- 3.2.2 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

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

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 polytetrafluoroethylene 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.

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