

SEMI F27-0997 (Reapproved 1103) TEST METHOD FOR MOISTURE INTERACTION AND CONTENT OF GAS DISTRIBUTION SYSTEMS AND COMPONENTS BY ATMOSPHERIC PRESSURE IONIZATION MASS SPECTROMETRY (APIMS)

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1 Purpose

1.1 This test will determine the quantity of removable moisture and the degree of interaction with trace concentrations of gas phase moisture, of gas distribution systems and components. APIMS is currently the technique of choice for such tests because it is essentially the only commercially available method capable of ppt moisture analysis and because of its superior response time. This method may provide guidelines for the application of other techniques with similar detection limits and response time to APIMS which are not commercially available at this time.

1.2 The results of this test can be used for qualitative ranking of systems and components and can also be used, by a sufficiently sophisticated user, as input for numerical simulation of distribution system behavior.

2 Scope

2.1 *System and Component Types* — This procedure applies to in-line components to be used to contain electronics grade materials in semiconductor gas distribution systems. The following components are expected to yield meaningful results when tested according to the present method: tubing, connectors (fittings), particle filters, valves (check, relief, shut-off, and metering), regulators, flow-through transducers/sensors, mass flow controllers and meters. Components with dead volumes, such as pressure gauges, can be tested according to this method, but the results will be difficult to interpret. Additional criteria besides those considered here need to be developed for testing components with dead volumes.

2.1.1 As gas phase moisture levels are dominated by desorption from surfaces, the procedure is expected to be most useful for components of large surface area such as particle filters and tubing. Components with very small wetted surface areas may have moisture interactions which are too small to measure using this test. Check valves and relief valves can be tested only if their operating parameters are consistent with the test conditions.

2.1.2 Tubing samples must be rather long (3–4 m) to yield useful results, but they can be bent into a U shape in order to be accommodated into a practical test bench. Radii of curvature should be no less than 6 times the internal diameter of the tube and the minimum number of bends should be used. Most ultra-high purity components are currently supplied with metal gasket connectors, so the test bench employed should be fitted with mating connectors. This type of fitting is usually welded to tubing, which is not recommended as it introduces a potential source of uncontrolled variability to the experiments. Instead, a compression fitting on the tube and a suitable adapter should be used. The compression fitting should not be disconnected after initial connection until the test series is over. In this way, deterioration in integrity of the connection can be avoided. The same type of gasket to compression fitting adapter should be used for all tubing samples.

2.1.3 Purifiers require special test procedures and are not addressed here.

2.1.4 Simple systems consisting of components connected in line can be tested by the present method. Complex systems (i.e., those with more than one potential inlet and/or outlet) will show performance which varies depending on the test configuration (which inlets and outlets are chosen). Testing of such systems is not addressed by the current document.

2.2 *Gases* — The procedure will be carried out in nitrogen. The results will provide a ranking with respect to moisture contribution which may be applied with due caution to components intended for use in other gas streams. Other “inert” gases will have different purging characteristics and may dry a component more quickly or slowly. Reactive gases may react chemically with moisture. Considerations relating to corrosion resistance are outside the scope of the present document, although the test procedure may prove useful in corrosion studies.

2.3 *Operating Situations* — Moisture contribution from a component may be the result of contamination arising in its manufacture, or from subsequent exposure to

ambient air or non-dry gas. Thus, it is necessary to consider two main situations:

1. The “initial dry-down” situation, which is determined by the moisture content of the component as received with the effects of manufacturing process and design, surface quality, pretreatment and packaging convoluted together.
2. The “Response to upset” situation, which is determined by the amount of moisture taken up by the component and subsequently released in any exposure after receipt.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard 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 allows the determination of moisture interactions which can be used, for example, to rank components in order of decreasing moisture interaction. Because different degrees of moisture interaction are desirable in different situations, selecting the “best” components requires consideration of how they will be used in a given distribution system, either qualitatively or through numerical simulation of distribution system behavior.

4 Referenced Standard

4.1 SEMI Standard

SEMI C15 — Test Method for ppm and ppb Humidity Standards

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

5 Terminology

5.1 Acronyms and Abbreviations

5.1.1 *APIMS* — Atmospheric Pressure Ionization Mass Spectrometry

5.1.2 *EPSS* — Electropolished Stainless Steel

5.1.3 *MFC* — Mass Flow Controller

5.1.4 *ppm* — molar parts per million ($\mu\text{mole/mole}$). The same as ppmv.

5.1.5 *ppb* — molar parts per billion (nmole/mole). The same as ppbv.

5.1.6 *ppt* — molar parts per trillion (pmole/mole). The same as pptv.

5.2 Definitions

5.2.1 *induction time* — the elapsed time between when humidified gas is input to the test component and when moisture is detected at the moisture analyzer. For a component and test system which are perfectly transparent to moisture the induction time is equal to the residence time of the gas in the system.

5.2.2 *peak height* — the maximum moisture concentration recorded when a moisture input of predefined length and concentration is introduced to a test component.

6 Required Equipment

6.1 *APIMS* — The APIMS used for moisture detection can be of any type. In order to be considered valid, any series of moisture tests shall reference two single point calibrations, one at the beginning and one at the end of the series. These calibrations shall both be made under the same analytical conditions (flow, pressures, plate voltages, etc.) as the tests and the results shall be within 5% of each other. Provided this condition is met, the interval between calibrations may be left to the discretion of the operator; however, it is suggested that calibration should be carried out whenever the equipment is moved and/or every two weeks of operation.

6.2 *Dry Gas and Moisture Generator* — A source of extremely dry nitrogen (less than 100 ppt moisture) and a moisture generator capable of delivering up to 13 slm nitrogen doped with 50 ppb moisture is required. This generator may be the same as used to calibrate the APIMS. The output of the generator shall be verified according to SEMI C15.

6.2.1 Most ultra-high purity components are currently fitted with metal gasket type connectors. The same type of connector should, therefore, be incorporated into the test bench for connection to the test components.

6.3 *Test Blank* — Any series of tests shall include the results of testing a blank. The blank shall be the shortest convenient length (no more than 1 m) of 1/4" electropolished stainless steel (EPSS) tubing with suitable fittings and/or adapters at either end to enable it to be inserted in place of the test specimen.