

SEMI F35-0998

TEST METHOD FOR ULTRA-HIGH PURITY GAS DISTRIBUTION SYSTEM INTEGRATION VERIFICATION USING NON-INVASIVE OXYGEN MEASUREMENT

NOTE: This document that was previously designated as SEMI E71 (Test Method for Ultra-High Purity Gas Distribution System Integration Verification Using Non-Invasive Oxygen Measurement) has been re-designated as SEMI F35.

1 Purpose

1.1 This test method defines a procedure to monitor the integrity of ultra-high purity (UHP) gas distribution systems by detecting the ingress of atmospheric oxygen. This test method would be used to evaluate an “active” UHP gas distribution system on a continuous basis by using *non-invasive* O₂ measurement, without requiring an interruption of the process tools using the UHP gases of interest.

1.2 This test method should be used to protect semiconductor fabrication processes using the UHP gases, which may be sensitive to contamination by any of the common atmospheric impurities, such as N₂, O₂, H₂O, CO₂, before product yield problems develop.

1.3 This is the first such test method that describes a noninvasive leak detection and locating procedure. It differs from SEMI F1, which is an invasive technique for identifying leak sources using a mass spectrometer and a helium tracer gas.

2 Scope

2.1 This test method applies to UHP gas distribution systems used in semiconductor manufacturing facilities and comparable research and development areas.

2.2 This test method applies to bulk gas distribution systems carrying UHP gases such as N₂, Ar, He, H₂, N₂O, SF₆, and many halocarbons. In most cases, O₂ is present only in ultra-low trace levels (typically less than 1.0 ppb).

2.3 This test method will provide real-time monitoring of UHP gas distribution systems, resulting in meaningful system integrity verification, atmospheric contaminant trending analysis, and leak locating.

2.4 This test method will provide the user with sufficient information to identify and troubleshoot sources of atmospheric leakage into the UHP gas distribution system.

2.5 This test method includes the specification of the required O₂ analytical equipment, standard methods for proper use of the O₂ analytical equipment, and manipulation of the O₂ data in identifying atmospheric leak sources.

3 Limitations

3.1 This test method will only be successful in identifying the presence and location of leaks from atmospheric air using O₂ as the tracer gas representative of all atmospheric air contaminants (i.e., N₂, O₂, H₂O, CO₂). There is no capability to identify cross-contaminant species between different UHP gases which only contain ultra-low trace levels of O₂ or outward leaks to atmosphere.

3.2 This test method will not focus specifically on the process of detecting the exact location of every possible atmospheric air leakage source. Rather, it will describe techniques and examples demonstrating how atmospheric air leaks can be reliably identified and located, in general.

3.3 Results from this test method may not ensure that other common atmospheric impurities (e.g., H₂O) are below their required specification limits after a leak has been identified, corrected, and O₂ levels have returned to normal. For example, since O₂ diffuses more quickly and has a weaker surface adsorption than H₂O, O₂ will be the first atmospheric impurity detected. However, H₂O will likely be the last atmospheric impurity to be purged out once a leak has been identified and repaired.

3.4 This test method is not a direct substitution for SEMI F1.

3.5 This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address the safety considerations 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 Referenced Documents

4.1 SEMI Standard

SEMI F1 — Specification for Leak Integrity of HighPurity Gas Piping Systems and Components

5 Terminology

5.1 *atmospheric impurities* — The common impurities to UHP gases that are found in atmospheric air.

NOTE: Impurities such as N₂, O₂, H₂O, CO₂.

5.2 *contaminant signature* — Typical baseline performance of a specific sample point within a large distribution system with respect to its O₂ impurity concentration. This is considered to be a normal and acceptable impurity level.

5.3 *critical sample point* — Gas sample point which is monitored continuously because it is deemed in a critical location in the distribution system, or that which is most sensitive to causing product/process quality problems from the ingress of atmospheric air impurities.

5.4 *investigative sample point* — Gas sample point which is selected for spot checking analysis as part of an investigation to locate an atmospheric air leak source.

5.5 *low detectable limit* — Defined as the smallest level of O₂ measurement which can be quantified after performing a blank test on zero gas. For our purposes, this is determined to be 2 times the total peak-to-peak noise over 8 hours while the O₂ analyzer is sampling on zero gas.

5.6 *ppbv* — Parts per billion by volume.

5.7 *point of use* — The connection point where the gas distribution system connects to the process tool which uses the UHP gas.

5.8 *sensitivity* — Defined as the smallest detectable step change in O₂ that the analyzer is capable of sensing.

5.9 *ultra-high purity (UHP) gas distribution system* — Semiconductor fab gas delivery system which typically contains impurities from atmospheric air of < 0.2 ppb at the input source, and atmospheric impurities at the point-of-use of typically < 1 ppb.

5.10 *weekly zero drift* — Total analyzer drift while measuring zero gas over a 1-week time period. Includes all components of analyzer drift.

5.11 *zero gas* — Test gas which is known to contain less than 0.1 ppb of O₂ as an impurity.

6 Summary of Method

6.1 The user must identify critical sampling locations in the UHP gas distribution system. These points are either specific process tool points-of-use or key lateral branch lines, which must avoid atmospheric contamination, or representative of a critical purity zone containing process tools that are particularly sensitive to atmospheric contaminants.

6.2 Connect O₂ analysis equipment to these critical sample points for continuous monitoring.

6.3 Qualify the performance of the O₂ analysis equipment before collecting data.

6.4 Begin continuous monitoring of critical sample points. Identify typical or baseline “contaminant signature” performance of the UHP gas distribution system.

6.5 Use continuous trend data to identify problematic events (contaminant spikes) and/or significant changes from the “contaminant signature” performance level.

6.6 Use additional investigative sample point measurements (spot checks) along with observable correlations to gas distribution system usage to locate and correct atmospheric leak sources.

7 Interferences

7.1 While sampling from any point in the gas distribution system, it is critical that the sample be delivered to the O₂ analytical equipment without introducing additional atmospheric O₂. This could create significant errors in the analysis. Observing a higher O₂ reading after reducing the sample flow to the analyzer may be an indication that there is a leak between the sample point and the O₂ analysis equipment.

7.2 An O₂ analyzer may become inaccurate due to improper calibration or simply due to long-term analyzer drift. This will cause inaccurate O₂ readings which do not represent the true performance of the UHP gas distribution system. The O₂ analyzer must be operated per the manufacturer’s recommendation for calibration technique, calibration frequency, and other routine maintenance.

7.3 Some O₂ analytical methods have cross-sensitivity to other gaseous components which may be found in UHP gases (i.e., H₂, CO, CH₄, and other hydrocarbons).

8 Requirements

8.1 *Personnel Qualification* — Personnel performing these tests in accordance with this test method shall have suitable training and experience. Such personnel shall, as a minimum, be knowledgeable of:

8.1.1 The operation and calibration of the specific equipment used in performing this test.

8.1.2 The proper procedures in handling UHP gases as agreed upon by supplier and user.

8.1.3 The proper safety procedure in handling combustible or toxic gases as agreed upon by supplier and user.

8.1.4 The behavior of atmospheric leak sources relative to position, pressure/flow, and gas usage.

9 Apparatus

9.1 An analyzer must be selected which is capable of continuous ultra-low trace O₂ measurements, with the ability to report measurements with at least once per minute frequency.

9.2 The ultra-low trace O₂ analyzer shall meet or exceed the following specifications. These specifications are appropriately defined in Section 5:

Sensitivity	< 0.1 ppb
Low Detectable Limit	< 0.3 ppb
Weekly Zero Drift	< 0.5 ppb
Response Time	< 10 minutes for 90% of a 0–1 ppb O ₂ step change

NOTE: The O₂ analysis must be conducted using an analytical method which meets or exceeds the specifications required by this test method. If not, the user will not be able to confidently distinguish true gas distribution system impurity trends from false analyzer trends.

10 Safety Precautions

10.1 This test method is not a replacement for safety regulations. It is the responsibility of the user to ensure that the UHP gas distribution systems under analysis comply with applicable safety regulations, as agreed to between gas supplier and user.

10.2 It is also the responsibility of the user to comply with applicable safety regulations governing the operation of the required O₂ analytical equipment, as specified by the analyzer manufacturer.

11 Identify Sample Point Locations

11.1 Identify the critical sample point locations in the UHP gas distribution system which allow for a continuous gauging of overall system integrity. These shall include, at a minimum:

11.1.1 An exit purity O₂ measurement of the UHP gas at the furthest point of the gas distribution system within the fab.

11.1.2 Sample point locations representing sub-sections of the UHP gas distribution system, which are deemed critical for the avoidance of atmospheric impurities.

11.1.3 Point-of-use process tool locations which are deemed critical for the avoidance of atmospheric impurities.

11.2 A source purity O₂ measurement of the UHP gas entering the gas distribution system is suggested to be

used as a reference comparison against other points within the fab.

11.3 Additional investigative (spot check) O₂ measurements may be required at sample point locations which aid in deducing the location of suspected atmospheric leak sources. This will vary from situation to situation. (See Section 16.)

11.4 The exact sample tap location must be representative of the measurement point of interest. Avoid measurement of dead-leg locations.

12 Preparation of Sample Point Locations

12.1 Thoroughly pre-purge the sample point prior to connecting the O₂ analyzer.

12.2 Thoroughly cycle-purge any regulators or other components in the sample system. Cycle the pressure at least 20 times with a high/low pressure ratio of at least 5 times.

12.3 Connect the O₂ analyzer to the sample point using appropriate UHP gas lines and fittings. Avoid using long lengths of bellows-type tubing because of long purge-down requirements. Avoid using Teflon or plastic tubing of any kind. Teflon and most other plastics are extremely permeable to O₂ and will cause atmospheric O₂ leakage. Only a few feet of plastic tubing can introduce ppm levels of O₂.

12.4 Establish flow from the sample point to the O₂ analyzer per the manufacturer's recommendations.

13 Calibration and Qualification

13.1 Follow the manufacturer's recommendations for initial start-up and calibration of the O₂ analyzer.

13.2 After initial start-up and calibration, adhere to the manufacturer's recommendations for routine calibration and maintenance needed to achieve the required performance specification as described in Section 9.2.

13.3 There will be two modes of O₂ measurement:

13.3.1 Critical Sample Point O₂ Measurement

13.3.1.1 Applicable to O₂ measurements performed continuously on the critical sample point locations identified earlier.

13.3.1.2 Prior to continuous data collection use of the analyzer, validate it by demonstrating the capability required in the specification for Weekly Zero Drift. Chart the zero baseline drift and peak-to-peak noise while on zero gas for 1 week.

13.3.1.3 Re-validate the O₂ analyzer performance by running a 24-hour blank test on zero gas monthly.