



Designation: F 1396 – 93 (Reapproved 1999)

## Standard Test Method for Determination of Oxygen Contribution by Gas Distribution System Components<sup>1</sup>

This standard is issued under the fixed designation F 1396; 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 ( $\epsilon$ ) 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 covers a procedure for testing components for oxygen contribution to ultra-high purity gas distribution systems at ambient temperature. In addition, this test method allows testing of the component at elevated ambient temperatures as high as 70°C.

1.2 This test method applies to in-line components containing electronics grade materials such as those used in a semiconductor gas distribution system.

#### 1.3 Limitations:

1.3.1 This test method is limited by the sensitivity of current instrumentation, as well as the response time of the instrumentation. This test method is not intended to be used for test components larger than 12.7-mm (1/2-in.) outside diameter nominal size. This test method could be applied to larger components; however, the stated volumetric flow rate may not provide adequate mixing to ensure a representative sample. Higher flow rates may improve the mixing but excessively dilute the sample.

1.3.2 This test method is written with the assumption that the operator understands the use of the apparatus at a level equivalent to six months of experience.

1.4 The values stated in SI units are to be regarded as the standard. 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.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.10 on Processing Environments.

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### 2. Terminology

#### 2.1 Definitions:

2.1.1 *baseline*—the instrument response under steady state conditions.

2.1.2 *glove bag*—an enclosure that contains a controlled atmosphere. A glove box could also be used for this test method.

2.1.3 *heat trace*—heating of a component, spool piece, or test stand by a uniform and complete wrapping of the item with resistant heat tape.

2.1.4 *minimum detection limit (MDL) of the instrument*—the lowest instrument response detectable and readable by the instrument, and at least two times the amplitude of the noise.

2.1.5 *response time*—the time required for the system to reach steady state after a change in concentration.

2.1.6 *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.

2.1.7 *standard conditions*—101.3 kPa, 0.0°C (14.73 psia, 32°F).

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

2.1.9 *test stand*—the physical test system used to measure impurity levels.

2.1.10 *zero gas*—a purified gas that has an impurity concentration below the MDL of the analytical instrument. This gas is to be used for both instrument calibration and component testing.

#### 2.2 Symbols:

2.2.1  $P_1$ —The inlet pressure measured upstream of the purifier and filter in the test apparatus.

2.2.2  $P_2$ —The outlet pressure measured downstream of the analyzer in the test apparatus.

2.2.3 *ppbv*—Parts per billion by volume assuming ideal gas behavior, equivalent to nmole/mole (such as nL/L). The same as molar parts per billion (ppb).

2.2.4 *ppbw*—Parts per billion by weight (such as ng/g).

2.2.5 *ppmv*—Parts per million by volume assuming ideal gas behavior, equivalent to  $\mu\text{mole/mole}$  (such as  $\mu\text{L/L}$ ). The same as molar parts per million (ppm).

2.2.6 *ppmw*—Parts per million by weight (such as  $\mu\text{g/g}$ ).

2.2.7  $Q_1$ —the bypass sample flow not going through the analytical system.

2.2.8  $Q_2$ —the total sample flow through the analytical system.

2.2.9  $Q_s$ —the flow through the spool piece or component.

2.2.10  $T_a$ —the temperature of the air discharged by the analyzer's cooling exhaust.

2.2.11  $T_s$ —the temperature of the spool piece or component.

2.2.11.1 *Discussion*—Precautions must be taken to insure that the temperature measured by the thermocouple is as close as possible to that of the spool piece and test component. Appropriate insulation and conductive shield should be used to achieve as uniform a temperature as possible. The thermocouple must be in contact with the outside wall of the component or spool piece.

2.2.12  $V-1$ ,  $V-2$ —inlet and outlet valves of bypass loop, respectively.

2.2.13  $V-3$ ,  $V-4$ —inlet and outlet valves of test loop, respectively.

### 3. Significance and Use

3.1 This test method defines a procedure for testing 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 purposes of qualification for this installation.

### 4. Apparatus

#### 4.1 Materials:

4.1.1 *Nitrogen or Argon*, clean and dry, as specified in 7.5.

4.1.2 *Spool Piece*, that can be installed in place of the test component is required. This piece is to be a straight section of 316L electropolished stainless steel tubing with no restrictions. The length of the spool piece shall be 200 mm. The spool piece has the same end connections as the test component.

4.1.2.1 *Components With Stub Ends*—Use compression fittings with nylon or teflon ferrules to connect the spool piece and test component to the test loop. Keep the purged glove bag around each component for the duration of the test. In the case of long pieces of electropolished tubing, use two glove bags, one at each end.

4.1.3 *Tubing*, used downstream of the test component shall be 316L electropolished stainless steel seamless tubing. The diameter of the sample line to the analyzer shall not be larger than 6.4 mm ( $\frac{1}{4}$  in.). The length of the sample line from the tee (installed upstream of the pressure gage  $P_2$ ) to the analyzer shall not be more than 600 mm, so as to minimize the effect (adsorption/desorption) of the sample line on the result. The sample line shall have no more than two mechanical joints.

4.1.4 *Valves*, diaphragm or bellows type, capable of unimpaired operation at  $94^\circ\text{C}$  ( $200^\circ\text{F}$ ). The use of all-welded, all-metal valves is preferred.

#### 4.2 Instrumentation:

4.2.1 *Oxygen Analyzer*—The oxygen analyzer is to be placed downstream of the test component. Accurate baseline readings must be obtained prior to and subsequent to each of the tests. Excessive deviations in baseline levels ( $\pm 10$  ppbv) before or after the tests require that all results be rejected. The analyzer must be capable of accurately recording changes in oxygen concentrations on a real time basis.

4.2.2 *Oxygen Analyzer Calibration*—Zero gas shall be at an oxygen level below the MDL of the instrument, supplied by purified gas, with the purifier in close proximity to the analyzer. The instrument's internal standard, if available, is to be used for the span calibration. Alternatively, span gas from a cylinder may be used.

4.3 *Pressure and Flow Control*—Upstream pressure is to be controlled with a regular upstream of the test component. Flow is to be controlled at a point downstream of the sampling port and monitored at that point. A mass flow controller is preferred for maintaining the flow as described in 7.4. Sampling is to be performed via a tee in the line, with a section of straight tubing before the mass flow controller. All lines must conform to 4.1.3. Inlet pressure is monitored by  $P_1$ . Test flow is the sum of  $Q_1$  and  $Q_2$ .  $Q_1$  is directly controlled, and  $Q_2$  is the measured flow through the analyzer. Refer to Fig. 1.

4.4 *Bypass Loop*—The design of the bypass loop is not restricted to any one design. It could be, for example, a 3.2-mm ( $\frac{1}{8}$ -in) 316L stainless steel coil or a flexible tube section. This allows the flexibility necessary to install test components of different lengths.

### 5. Hazards

5.1 It is required that the user have a working knowledge of the respective instrumentation and that the user practice proper handling of test components for trace oxygen analysis. Good laboratory practices must also be understood.

5.2 It is required that the user be familiar with proper component installation, and that the test components be installed on the test stand in accordance with manufacturer's instructions.

5.3 Do not exceed ratings (such as pressure, temperature, and flow) of component.

5.4 Gloves are to be worn for all steps.

5.5 Limit exposure of the instrument and test component to atmospheric contamination before and during the test.

### 6. Calibration

6.1 Calibrate instruments using standard laboratory practices and manufacturer's recommendations.

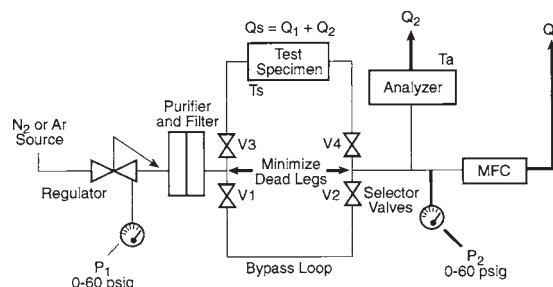


FIG. 1 Test Schematic

## 7. Conditioning

7.1 Ensure that adequate mixing of the test gas is attained.

7.2 *Pressure*—Test component at 200 kPa gage (30 psig) measured at  $P_2$ .

7.3 *Temperature*— $T_s$  is to be in the ambient temperature range of 18 to 26°C (64 to 78°F) and in the higher mean temperature range of 69 to 71°C (156 to 160°F).  $T_a$  must not deviate more than  $\pm 2^\circ\text{C}$  (4°F) from the time of calibration to the termination of the test.  $T_a$  must either be within the range of 18 to 26°C (64 to 78°F) or be consistent with the analytical systems manufacturer's specifications, whichever is more stringent.

7.4 The flow rate  $Q_s$  for components is 1 standard L/min with  $\pm 2\%$  tolerance.

7.5 The test gas shall be purified nitrogen or argon with a maximum oxygen concentration not exceeding an oxygen concentration of 10 ppb. Gas quality must be maintained at flow specified in 7.4. The test gas must be passed through a filter having a pore size rating of 0.02  $\mu\text{m}$  or finer. The filter must be compatible with the 94°C (200°F) bake-out.

## 8. Preparation of Apparatus

8.1 A schematic drawing of a recommended test apparatus located inside a clean laboratory is shown in Fig. 1. Deviations from this design are acceptable as long as baseline levels consistent with 4.2.1 can be maintained. Nitrogen or argon gas is purified to remove water and hydrocarbons. The base gas is then filtered by an electronics grade, high purity, point of use filter (pore size rating  $\leq 0.02 \mu\text{m}$ ) before it is delivered to the test component.

8.2 A bypass loop may be used to divert gas flow through the test stand and the analyzer whenever the spool piece or a test component is installed or removed from the test stand. This prevents the ambient air from contaminating the test apparatus and the oxygen analyzer; thus, the analyzer baseline remains the same. A glove bag is used to enclose test component lines of the test apparatus during the installation and removal of the spool piece and the test piece.

8.3 A trace oxygen analyzer capable of detecting oxygen concentration levels down to 2 ppb is connected to the test stand to sample the gas flowing through the test piece. The purified and filtered base gas from the test stand containing  $< 10$  ppb oxygen is used as the zero oxygen gas source for the analyzer. Since the analyzer is sensitive to the sample flow rate, the metering valves within the analyzer should be adjusted to yield the flow rates required by the specification for an inlet pressure of 30 psig. The gas flow rate  $Q_s$  is set to 1 L/min.

8.4 Inlet gas pressure is controlled by a pressure regulator and measured immediately upstream of the purifier by an electronic grade pressure gage. Flow measurement is carried out by a mass flow controller (MFC) located downstream of the analyzer. The outlet pressure of the gas is measured immediately downstream of the analyzer by another electronic grade pressure gage. The MFC along with its digital readout should be calibrated before use to control and display the gas flow rate  $Q_1$ .

8.5 The temperature of the spool piece, test specimen, analyzer cell compartment, and the oxygen concentration

measured by the analyzer can either be recorded continuously by a 25 channel data logger or collected and stored in a computer using a data acquisition program.

## 9. Procedure (see Fig. 2)

9.1 *Bake-Out*—With the spool piece installed and valves V-1, V-2, V-3, and V-4 open, bake out the system (downstream of purifier to upstream of analyzer, exclusive of the exhaust leg) at 94°C (200°F) until outlet oxygen concentration is stable below  $<20$  ppbv. Flow of the gas is specified in 7.4. Cool to lower  $T_s$ . Close valves V-1 and V-2.

9.2 *Baseline*—Flow gas through the test stand with the spool piece installed on the test stand. Use the flow rate as defined in 7.4. Flow for 30 min after the oxygen concentration has attained a level of  $<20$  ppbv. Utilizing heat tape, heat the spool piece and upstream tubing to within 80 mm of the upstream valve. Monitor the oxygen of the outlet and the  $T_s$ , as specified in 7.3. The time required to reach the higher  $T_s$  must be less than or equal to 10 min. Continue testing for 30 min after a stable baseline is reestablished ( $<20$  ppbv) as specified in 9.1. Cool until the lower  $T_s$  is reached.

9.3 Place the spool piece, test component (in original bagging), and fittings into a glove bag or nitrogen tent without bagging. Purge the glove bag with approximately five glove bag volumes of inert gas. Disconnect the spool piece while maintaining the flow through the system. Maintain the spool piece in the proximity of the positive flow. Reinstall the spool piece on the test stand. The entire disconnection and reinstallation must be performed within 2 min. Maintain flow through the analyzer during disconnection and installation via the bypass loop, using valves V-1, V-2, V-3, and V-4 (if V-1 and V-2 are open, then V-3 and V-4 will be closed). During disconnection, open valves V-1 and V-2 first, then close V-3 and V-4. After connection, reverse the order. Keep the purged glove bag around each component for the duration of the test. In the case of long pieces of electropolished tubing, use two glove bags, one at each end.

9.4 Initiate flow through the spool piece in accordance with 8.4. Monitor  $T_s$  and  $T_a$  in accordance with 8.3. Monitor oxygen until a stable baseline, in accordance with 9.2, is reestablished ( $<20$  ppbv). Utilizing heat tape, heat the spool piece and upstream tubing to within 80 mm of the upstream valve. Turn on the current and monitor the oxygen of the outlet and the  $T_s$ , in accordance with 8.3. The time required to reach the higher  $T_s$  must be less than or equal to 10 min. Continue testing until a stable baseline is reestablished ( $<20$  ppbv). Cool until the lower  $T_s$  is reached.

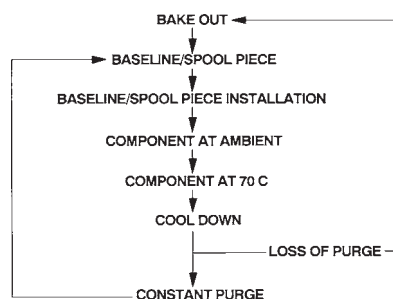


FIG. 2 Test Procedure Sequence

9.5 Without disconnecting the spool piece, place the test component and the fittings in a glove bag or nitrogen tent flushed with clean, dry nitrogen. Open valves V-1 and V-2 first, then close V-3 and V-4. Disconnect and recap the spool piece while maintaining flow. Maintain flow through the analyzer continuously with valves V-1 and V-2 during disconnection and installation. Remove the test component caps and install the test component. Open V-3 and V-4 first then close V-1 and V-2. The time from disconnection of the spool piece to installation of the test component must be less than 2 min.

NOTE 1—The installation conditions of the test component, as well as glove bag conditions, must be the same as the installation conditions for the spool piece outlines in 9.2, including time to disconnect and connect. The spool piece must not be removed from the glove bag for the duration of the test.

9.5.1 When testing valves, MFCs, and regulators, with valves V-3 and V-4 closed, and all gas flowing through the bypass, connect the test component to the test stand. The component will be installed in the “as received” condition (either open or closed). After installation, place the component in the fully open condition.

9.6 Initiate flow in accordance with 8.4. Monitor  $T_s$  and  $T_a$  in accordance with 8.3. Monitor oxygen until a stable baseline in accordance with 9.2 is reestablished (<20 ppbv). Terminate the test after 3 h if a stable baseline is not achieved.

9.7 Utilizing heat tape, heat the test component and upstream tubing to within 80 mm of the upstream valve. Monitor the oxygen of the outlet and the  $T_s$ , as specified in 7.3. The time required to reach the higher  $T_s$  must be less than or equal to 10 min. Continue testing until a stable baseline is reestablished (<20 ppbv). Terminate the test in 3 h if a stable baseline is not achieved. Cool until the lower  $T_s$  is reached.

9.8 Without disconnecting the test component, place the spool piece in a glove bag or nitrogen tent purged with clean, dry nitrogen. Open V-1 and V-2 first, then close V-3 and V-4. Disconnect and cap the test component while maintaining flow. Remove the spool piece caps and install the spool piece. Open Valves V-3 and V-4. The time from disconnection of the test

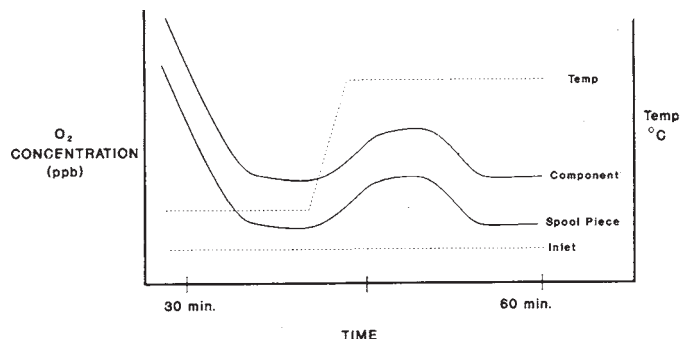


FIG. 3 Component Oxygen Contribution

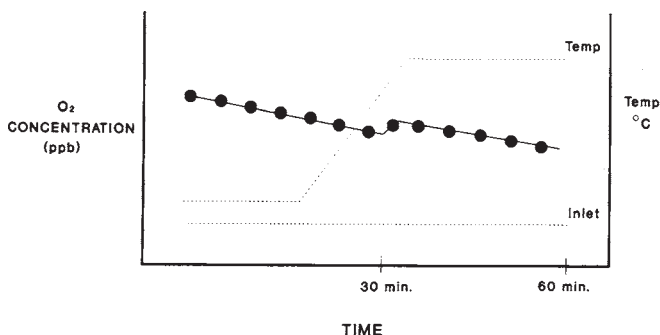


FIG. 4 Net Component Oxygen Contribution

component to installation of the spool piece must be less than 2 min. The 2-min limit reduces the exposure of the test piece to the glove bag environment. Maintain flow through the analyzer via Valves V-1 and V-2 during disconnection and installation.

9.9 With valves V-1, V-2, V-3, and V-4 open, maintain a purge through the system with a purified gas.  $Q_1$  may be reduced to a minimum of 0.5 slpm, or 10 % of the mass flow controller range. Start the next test run in accordance with 9.2 (establishment of stable baseline). If the gas flow is not maintained, the system must be baked out, in accordance with 9.1, prior to further testing.

9.10 *Sampling Frequency*—Perform and record sampling continuously (or at a maximum of 1-min intervals for digitally acquired data) during the specified time period.

## 10. Report

10.1 Refer to Table X2.1 for a numerical example.

10.2 *Data Collection*—A plot of oxygen concentration versus time is presented for (1) the respective spool piece baseline and (2) the test component. The elevated temperature data are plotted as a continuation of the ambient temperature data. A dual y-axis is used to plot  $T_s$  versus time on the same plot (refer to Fig. 3).

10.3 *Data Handling*—To determine the oxygen contribution of the test component, compute the difference between the spool piece plot and the component plot derived in 10.2 and graph the results. A dual y-axis (concentration ppbv and  $T_s$ ) is used (see Fig. 4).

10.4 Complete the table given in Fig. 5.

## 11. Precision and Bias

11.1 The precision and bias for this test method are being determined.

## 12. Keywords

12.1 components; contamination; gas distribution; oxygen analyzer; oxygen contribution; oxygen outgassing; semiconductor processing