Standard Test Method for
Determination of Oxygen Contribution by Gas Distribution System Components[1]

This standard is issued under the fixed designation F1396; 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 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 (½-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 applica-bility of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

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_I—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 μmole/mole (such as μL/L). The same as molar parts per million (ppm).

2.2.6 *ppmw*—Parts per million by weight (such as μg/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 (¼ 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°C (200°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 (±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 (⅛-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.
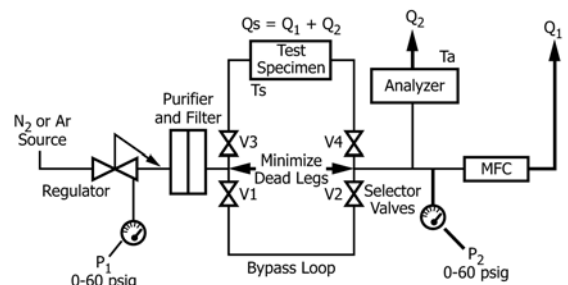

FIG. 1 Test Schematic

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.

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°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 μm 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 ≤ 0.02 μm) 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 disconnecting. 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
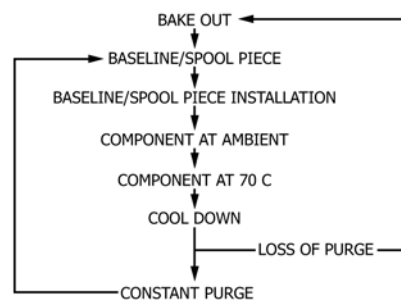

FIG. 2 Test Procedure Sequence

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.

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
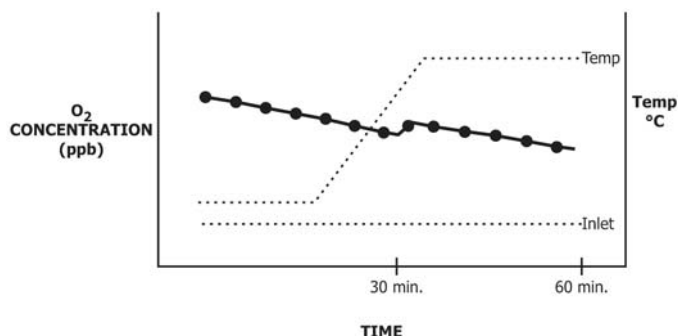

FIG. 4 Net Component Oxygen Contribution

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 valves 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 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

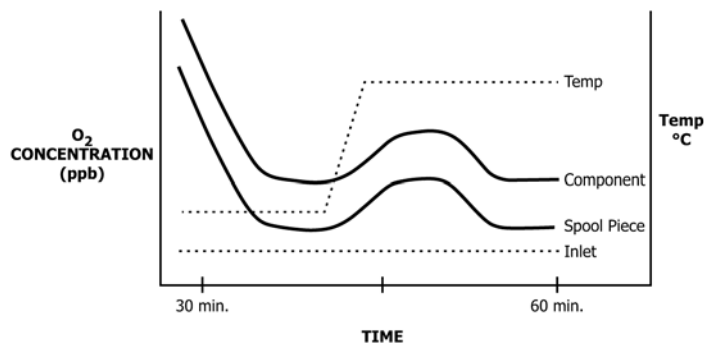11.1 The precision and bias for this test method are being determined.

FIG. 3 Component Oxygen Contribution

Date: _____
Test Location: _____
Test Component: _____
    Model Number: _____
Analyzer: _____
    Model Number: _____
    Last Calibration Date: _____
Base Gas: _____

Operator: _____
Spool Piece: _____
    Serial Number: _____
    Serial Number: _____

Flow Meter: _____
    Calibration Date: _____
    Cal Pressure: _____
MFC: _____
    Calibration Date: _____
    Cal Pressure: _____

Model Number: _____
Cal Temperature: _____ °C
Cal Gas: _____
Model Number: _____
Cal Temperature: _____ °C
Cal Gas: _____

I. Bake-out (if warranted)
T_a: _____ °C Q_1: _____ standard L/min $Q_s (Q_1+Q_2)$: _____ standard L/min
T_s: _____ °C Q_2: _____ standard L/min P_2 _____ mm Hg

Barometric Pressure: _____ mm Hg
Ambient Temperature: _____ °C
Data Acquisition Rate: _____
Data File Name: _____ . _____
Attach data presentation in graphical form.

II. Spool Piece Baseline

    A. Low Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min

      Barometric Pressure: _____ mm Hg
      Ambient Temperature: _____ °C
      Data Acquisition Rate: _____
      Data File Name: _____ . ____
Attach data presentation in graphical form.

Note—Archive all acquired data. Note file name on test data, if applicable.

    B: High Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min

      Barometric Pressure: _____ mm Hg
      Ambient Temperature: _____°C
      Data Acquisition Rate: _____
      Data File Name: _____ . _____
    Attach data presentation in graphical form.

III. Spool Piece

    A. Low Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min
      Barometric Pressure: _____ mm Hg
      Data Acquisition Rate: _____
      Data File Name: _____ . ___
      Ambient Temperature: _____ °C

      Total time for disconnection/installation: _____
    Attach data presentation in graphical form.

    B: High Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min
      Barometric Pressure: _____ mm Hg
      Ambient Temperature: _____ °C
      Data Acquisition Rate: _____
      Data File Name: _____ . ____
Attach data presentation in graphical form.

IV. Test Component

    A. Low Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min

      Barometric Pressure: _____ mm Hg
      Data Acquisition Rate: _____
      Data File Name: _____
      Ambient Temperature: _____ °C

    Total time for disconnection/installation: _____
    Attach data presentation in graphical form.

    B: High Temperature

      T_a: _____ °C Q_1: _____ standard L/min $Q_s(Q_1+Q_2)$: _____ standard L/min
      T_s: _____ °C Q_2: _____ standard L/min

      Barometric Pressure: _____ mm Hg
      Ambient Temperature: _____ °C
      Data Acquisition Rate: _____
      Data File Name: _____ . ____
Attach data presentation in graphical form.

FIG. 5 Sample Oxygen Contribution Data Table

12. Keywords

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

APPENDIXES

(Nonmandatory Information)

X1. ALTERNATIVE TEST

X1.1 This test method may also be conducted using an atmospheric pressure ionization mass spectrometer (APIMS). APIMS is a complex technique that will ultimately yield better sensitivities (possibly parts per trillion (pL/L)) than vacuum (MS) techniques.

X2. APPLICATION NOTES

X2.1 *Mass Contribution Calculation*—Calculate the total mass of oxygen contributed by the component under test from the concentration versus time curve shown in Fig. 3. The following symbols apply in the derivation:

m_B = mass of base gas,
m_I = mass of measured oxygen,
MW_B = molecular weight of base gas,
MW_I = molecular weight of oxygen,
n_B = number of moles of base gas,
n_I = number of moles of oxygen,
p_B = pressure of base gas,
p_I = pressure of oxygen,
p_T = total system pressure,
P_R = pressure at which MFC was calibrated,
$Q_{s,m}$ = total mass flow through test component,
$Q_{s,v}$ = total volume flow through test component,
ρ_R = density of base gas at T_R and P_R,
R = universal gas constant,
T = system temperature,
t = time,
T_R = temperature at which MFC was calibrated,
V_B = volume occupied by base gas, and
V_I = volume occupied by oxygen.

X2.2 Most commercial instruments report the measured oxygen concentration in units of ppmv. The concentration axis is first converted to ppmw. The relationship between them may be derived as follows:

X2.2.1 Recognizing that at low oxygen concentrations:

$$V_B >>> V_I \text{ and } m_B >>> m_I$$

X2.2.2 The equations defining ppmv and ppmw may be simplified to:

$$ppmv \sim (V_I/V_B) \times 10^6$$

$$ppmw \sim (m_I/m_B) \times 10^6$$

X2.2.3 At low pressure assume both gases obey the ideal gas law:

$$p_I V_I = n_I RT \tag{X2.1}$$

$$p_B V_B = n_B RT \tag{X2.2}$$

X2.2.4 Dividing Eq X2.1 by Eq X2.2:

$$p_I V_I / p_B V_B = n_I / n_B \tag{X2.3}$$

X2.2.5 Using Amagat's law of partial volumes, $p_I = p_B = p_T$ and Eq X2.3 becomes:

$$V_I/V_B = n_I/n_B \tag{X2.4}$$

with:

$$n_I = m_I/MW_I \tag{X2.5}$$

$$n_B = m_B/MW_B \tag{X2.6}$$

X2.2.6 Substituting Eq X2.5 and Eq X2.6 into Eq X2.4 and multiplying by 10^6:

$$(V_I/V_B) \times 10^6 = (m_I/m_B) \times (MW_B/MW_I) \times 10^6 \tag{X2.7}$$

X2.2.7 Substituting the simplified definitions of ppmv and ppmw into Eq X2.7:

$$ppmv = ppmw \times (MW_B/MW_I) \tag{X2.8}$$

$$ppmw = ppmv \times (MW_I/MW_B) \tag{X2.9}$$

X2.2.8 Eq X2.9 is required for converting instrument response in ppmv to ppmw.

X2.3 The time axis in Fig. 3 must be converted from units of time to units of mass of base gas. This conversion will be demonstrated for the case where a mass flow controller is on the downstream bypass of the component under test measuring Q_1, (see Fig. 1).

X2.4 Most MFCs report and control based on a set point that is given in units of volume flow at P_R and T_R (the calibration temperature and pressure of the MFC). The MFC is a device that will maintain a given volume flow rate despite small changes in upstream or downstream pressure or fluctuations in temperature. To convert to a mass flow ρ_R, the base gas density at P_R and T_R is needed, and:

$$Q_{1,m} = Q_{1,v} \times \rho_R \tag{X2.10}$$

X2.4.1 Any flow diverted to the analyzer(s) $(Q_{2,m})$ must also be included in the calculation of total mass flow:

$$Q_{s,m} = Q_{1,m} + Q_{2,m} \qquad (X2.11)$$

X2.4.2 If a rotameter is used to calculate $Q_{2,m}$ a separate measurement of room temperature and barometric pressure will be necessary. Once $Q_{s,m}$ is established, the total amount of base gas moving through the component is given by:

$$m_B = Q_{s,m} \times t \qquad (X2.12)$$

X2.4.3 The ppmv versus time curve given in Fig. 3 can now be converted into a ppmw versus mass of base gas curve (refer to Fig. X2.1). A unit area under such a curve is given by:

$$(m_I/m_B) \times m_B = m_I \qquad (X2.13)$$

X2.4.4 The total mass of oxygen measured $(m_{I,T})$ is given by the area under the ppmw versus m_B curve (see Fig. X2.1):

$$m_{IT} = \int_o^{m_{ST}} ppmw\, dm_B \qquad (X2.14)$$

X2.5 Integration may be carried out numerically by the trapezoid, Simpson's, or by any of the other quadrature methods (refer to Fig. X2.2). Alternatively, the data may be fit to a function that can be integrated in closed form and evaluated. The quadrature methods are easily applicable to digitized data and do not require knowledge of the underlying functional form of the data. As a final example, the total mass
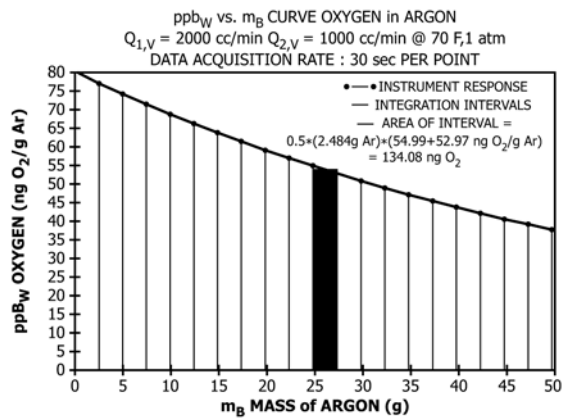

FIG. X2.1 Example Calculation Graph

FIG. X2.2 Example Calculation Graph

of oxygen will be evaluated using the trapezoid rule for the data in the following example:

X2.5.1 *Data Acquisition Rate*—One point every 30 s:

$Q_{1,v}$	= 1000 cm^3/min at approximately 101.3 kPa, 0.0°C (14.73 psia, 32°F)
$Q_{2,v}$	= 2000 cm^3/min at approximately 101.3 kPa, 0.0°C (14.73 psia, 32°F)
ρ_R	= 0.00143 kg/L at approximately 101.3 kPa, 0.0°C (14.73 psia, 32°F)
MW oxygen	= 31.998; *MW argon* = 39.948

$$ppbw = 0.8001 \times ppbv \qquad (X2.15)$$

$$Q_{s,m} = 50 \text{ cm}^3/s \times 0.00143 \text{ kg/L} = 0.0828 \text{ g/s } Ar$$

$$m_B = 0.0719 \text{ g } Ar/s \times t \qquad (X2.16)$$

X2.6 The results of the test are given in Table X2.1. The first column is the time into the run. The second column is the ppbv of oxygen measured at a given time. Column 3 is the mass of argon that has passed through the component, calculated from Eq X2.16. Column 4 is the ppbw of oxygen calculated from Eq X2.15. Column 5 is the area of the trapezoid for each interval. Finally, Column 6 is the total area up to and including the current interval. The graphs of these results are shown in Fig. X2.1 and Fig. X2.2.

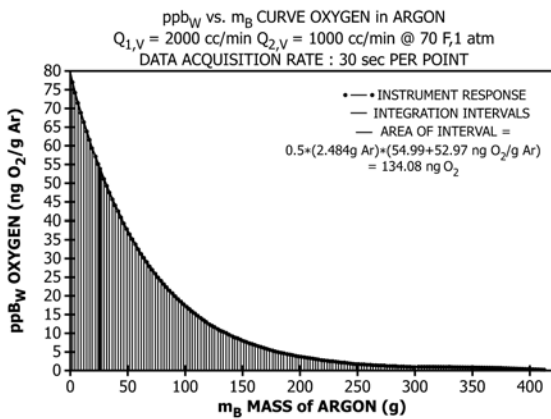X2.7 The final result for this test is 5300.23 ng oxygen, or 5.30µ g.

TABLE X2.1 Data for Sample Calculation

Note 1—Analyzer exhaust 2000 cc/s: bypass 1000 cc/s at 70°F, 1 atm. data acquisition rate one point every 30 s.

Time, seconds	ppbv	m_B, g Ar	ppbw ng O_2/g Ar	Area of Segment ng O_2	Total Area ng O_2
30	96.3194	2.484	43.4401	109.9668	109.967
60	92.7744	4.968	41.8412	105.9194	215.886
90	89.3597	7.452	40.3012	102.0210	317.907
120	86.0708	9.936	38.8179	98.2660	416.173
150	82.9029	12.420	37.3892	94.6493	510.822
180	79.8516	14.904	36.0131	91.1657	601.988
210	76.9126	17.388	34.6876	87.8103	689.798
240	74.0818	19.872	33.4109	84.5783	774.377
270	71.3552	22.356	32.1812	81.4654	855.842
300	68.7289	24.840	30.9967	78.4670	934.309
330	66.1993	27.324	29.8559	75.5790	1009.888
360	63.7628	29.808	28.7570	72.7973	1082.685
390	61.4160	32.292	27.6986	70.1179	1152.803
420	59.1555	34.776	26.6792	67.5372	1220.340
450	56.9783	37.260	25.6972	65.0514	1285.392
480	54.8812	39.744	24.7514	62.6572	1348.049
510	52.8612	42.228	23.8404	60.3510	1408.400
540	50.9156	44.712	22.9630	58.1298	1466.530
570	49.0417	47.196	22.1178	55.9903	1522.520
600	47.2367	49.680	21.3037	53.9295	1576.450
630	45.4981	52.164	20.5196	51.9446	1628.394
660	43.8235	54.648	19.7644	50.0328	1678.427
690	42.2106	57.132	19.0370	48.1913	1726.618
720	40.6570	59.616	18.3363	46.4176	1773.036
750	39.1606	62.100	17.6614	44.7092	1817.745
780	37.7192	64.584	17.0114	43.0636	1860.809
810	36.3310	67.068	16.3853	41.4786	1902.287
840	34.9938	69.552	15.7822	39.9520	1942.239
870	33.7058	72.036	15.2013	38.4815	1980.721
900	32.4652	74.520	14.6418	37.0652	2017.786
930	31.2703	77.004	14.1029	35.7010	2053.487
960	30.1194	79.488	13.5839	34.3870	2087.874
990	29.0109	81.972	13.0839	33.1214	2120.995
1020	27.9431	84.456	12.6023	31.9023	2152.898
1050	26.9146	86.940	12.1385	30.7281	2183.626
1080	25.9240	89.424	11.6917	29.5972	2213.223
1110	24.9699	91.908	11.2614	28.5078	2241.731
1140	24.0509	94.392	10.8469	27.4586	2269.189
1170	23.1656	96.876	10.4477	26.4479	2295.637
1200	22.3130	99.360	10.0632	25.4745	2321.112
1230	21.4918	101.844	9.6928	24.5369	2345.649
1260	20.7008	104.328	9.3360	23.6338	2369.282
1290	19.9389	106.812	8.9924	22.7640	2392.046
1320	19.2050	109.296	8.6615	21.9261	2413.972
7920	0.0050	655.776	0.0023	0.0057	2987.625
7950	0.0048	658.260	0.0022	0.0055	2987.630
7980	0.0047	660.744	0.0021	0.0053	2987.636