Designation: F1397 – 93 (Reapproved 2012)

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

This standard is issued under the fixed designation F1397; 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 testing components for total moisture contribution to a gas distribution system at ambient temperature. In addition, the test method allows testing at elevated ambient temperatures as high as 70°C and of the component moisture capacity and recovery.

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

1.3 *Limitations:*

1.3.1 This test method is limited by the sensitivity of current instrumentation, as well as by 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 applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 5.

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

2.1.11 *V-3, V-4*—inlet and outlet valves of test loop, respectively.

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[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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2.1.12 *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 *Abbreviations:*

2.2.1 *MFC*—mass flow controller.

2.2.2 *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).

2.2.3 *ppbw*—parts per billion by weight (such as ng/g).

2.2.4 *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).

2.2.5 *ppmw*—parts per million by weight (such as µg/g).

2.3 *Symbols:*

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

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

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

2.3.4 Q_2—the total sample flow through the analytical system.

2.3.5 Q_s—the flow through the spool piece or component.

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

2.3.7 T_s—the temperature of the spool piece or component.

2.3.7.1 *Discussion*—The thermocouple must be in contact with the outside wall of the component or spool piece.

3. Significance and Use

3.1 The purpose of this test method is to define 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 the purposes of qualification for this installation.

4. Apparatus

4.1 *Materials:*

4.1.1 *Nitrogen or Argon,* clean, dry, as specified in 8.4.

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 (0.8 in.). The spool piece should have the same end connections as the test component.

4.1.3 *Tubing,* used downstream of the purifier 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 (2.4 in.) to minimize the effect (adsorption/desorption) of the sample line on the result. The sample line shall have no more than two mechanical joints.

4.1.3.1 *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.

4.1.4 *Valves,* must be diaphragm or bellows type and 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 *Moisture Analyzer*—Moisture analyzers (such as electrolytic, piezo-electric, chilled mirror, or opto-electronic) are used to measure moisture levels. The 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 (±20 ppbv) before or after the tests require that all results be rejected. The analyzer must be capable of accurately recording changes in moisture concentrations on a real time basis (see Appendix X1.1).

4.2.2 *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 8.3. Sampling is to be performed via a tee in the line, with a run 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 total flow through the analyzer (see Fig. 1).

4.3 *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 moisture analysis. Good laboratory practices must also be understood.

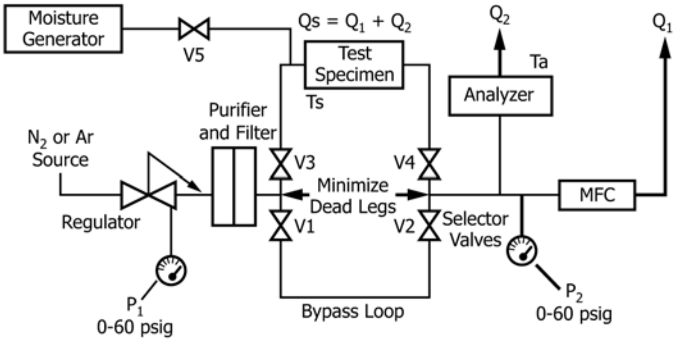


FIG. 1 Test Schematic

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 the 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.

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

6. Preparation of Apparatus

6.1 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 9.2 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 gas filter (pore size rating of ≤0.02 µm) before it is delivered to the test component.

6.2 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 moisture 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.

6.3 A moisture analyzer capable of detecting moisture concentration levels down to 10 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 moisture is used as the zero moisture 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.

6.4 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.

6.5 The temperature of the spool piece, test specimen, analyzer cell compartment, and the moisture concentration measured by the analyzer can either be recorded continuously by a multichannel data logger or collected and stored in a computer using a data acquisition program.

6.6 A moisture generator capable of generating moisture concentration levels over the range of 100 ppb to 2000 ppb is connected upstream of the test component through valve V-5.

7. Calibration

7.1 Calibrate instruments regularly in accordance with manufacturer's instructions.

7.2 *Moisture Analyzer Calibration* —Zero gas must contain moisture below the MDL of the instrument, supplied by purified gas, with the purifier in close proximity to the analyzer. Use the instrument's internal standard, if available, is to be used for the span calibration. If such a standard is not available, calibrate the analyzer with an external moisture generator according to the manufacturer's instructions.

8. Conditioning

8.1 *Pressure*—Test the test component at 200 kPa gage (30 psig) as measured by P_2.

8.2 *Temperature*—T_s is to be in the ambient temperature range of 18 to 26°C (64 to 78°F) and the higher range of 69 to 71°C (156 to 160°F). T_a must not deviate more than ±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.

8.3 The flow rate Q_s for components is 1 standard L/min with ±2 % tolerance.

8.4 The test gas shall be purified nitrogen or argon with a maximum moisture concentration not exceeding a moisture concentration level of 20 ppb. Gas quality must be maintained at flow specified in 8.3. The test gas must be passed through a gas 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.

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

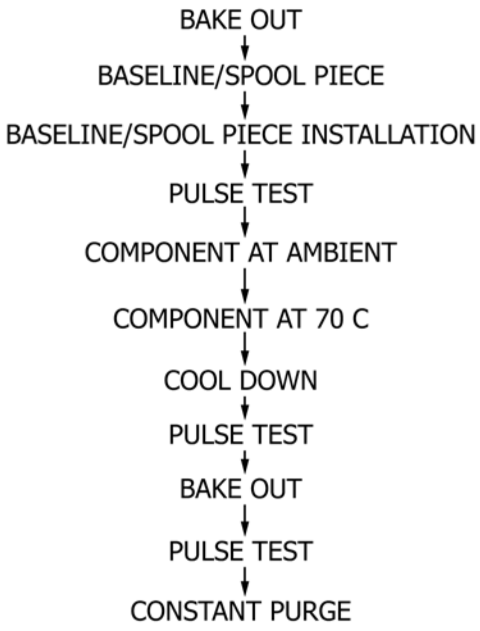


FIG. 2 Test Procedure Sequence

leg) at 94°C (200°F) until outlet moisture concentration is stable (<40 ppbv). Flow of the gas is specified in 8.3. 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 8.3. Flow for 30 min after the moisture concentration values have 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 moisture of the outlet and the T_s, as specified in 8.2. 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 (<40 ppbv). 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. Flow through the analyzer must be maintained 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.

9.4 Initiate flow in accordance with 8.3. Monitor T_s and T_a in accordance with 8.2. Monitor moisture 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. Monitor the moisture of the outlet and the T_s, as specified in 8.2. 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 (<40 ppbv). Cool until the lower T_s is reached.

9.5 Switch the spool piece input from the dry base gas source to a gas source containing a moisture concentration of 2 ppm. Record the time, t_0, the gas is introduced from the moisture generator through valve V-5, and wait for the period of 1 min.

NOTE 1—A lower moisture concentration input will be preferrable if an atmospheric pressure ionization mass spectrometer (APIMS) is used for analysis.

9.5.1 Switch the gas flow back to the dry gas source again. Allow the system to return to its baseline moisture concentration.

9.6 Monitor the time-dependent moisture concentration at the spool piece outlet. Record the time delay from t_0 to the time when measurable increase in moisture level is recorded by the moisture analyzer "induction time." Also record the maximum moisture concentration achieved "peak height," and the time from peak maximum to reestablish baseline (<40 ppb).

9.7 Repeat 9.5 and 9.6 twice (three pulses total) or until reproducible induction times and peak heights are obtained.

9.8 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 2—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 outlined 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.

9.8.1 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.

9.9 Initiate flow in accordance with 8.3. Monitor T_s from T_a in accordance with 8.2. Monitor moisture until a stable baseline is reestablished in accordance with 6.2 (<40 ppbv). The test is to be terminated after 3 h if a stable baseline is not achieved.

9.10 Utilizing heat tape, heat the test component and upstream tubing to within 80 mm of the upstream valve. Monitor the moisture of the outlet and the T_s, as specified in 8.2. 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 (<40 ppbv). Terminate the test in 3 h if a stable baseline is not achieved. Cool until the lower T_s is reached.

9.11 Switch the test-component input from the dry base gas source to a gas source containing a moisture concentration of 2 ppm. Record the time, t_0, the gas is introduced from the moisture generator through valve V-5, and wait for the period of 1 min for tubing, valves and samples of low surface area. For high surface area samples such as filters, wait for a period of 20 min.

NOTE 3—A lower moisture concentration input will be preferrable if an APIMS is used for analysis.

9.11.1 Switch the gas flow back to the dry gas source again. Allow the system to return to its baseline moisture concentration.

9.12 Monitor the time-dependent moisture concentration at the component outlet. Record the time delay from t_0 to the time when measurable increase in moisture level is recorded by the moisture analyzer "induction time." Also record the maximum moisture concentration achieved "peak height," and the time from peak maximum to reestablish baseline (<40 ppb).

9.13 Repeat steps 9.11 and 9.12 twice (three pulses total) or until reproducible induction times and peak heights are obtained.

9.14 *Bake-Out Test*— Heat the component to maximum rated temperature for 3 h. Cool to T_a. Repeat 9.11 to 9.13.

9.15 *Pulse test After Bake-Out*—The first pulse applied after bakeout will probably result in no output. Continue to apply pulses until consistent output is observed that is similar to output obtained from unbaked sample, that is, until the component is equilibrated with the background moisture level.

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

9.17 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 standard L/min, 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.

9.18 *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, Fig. X2.1 and Fig. X2.2 for a numerical example.

10.2 *Data Collection*— Present a plot of moisture concentration versus time 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. Use a dual *y*-axis to plot T_s versus time on the same plot (refer to Fig. 3).

10.3 *Data Handling*— To determine moisture 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 (refer to Fig. 4).

10.4 Complete the table given in Fig. 5 and continued in Fig. 6, and Fig. 7.

11. Precision and Bias

11.1 The use of this test method will provide results with a certain minimum absolute error. Due to the difficulty of obtaining accurate standards for moisture analyzer calibration, combined with the moisture response of any particular moisture analyzer, the propagation of errors can easily yield results that, deviate by more than 200 % from absolute. This does not invalidate the use of this test method for comparing test components.

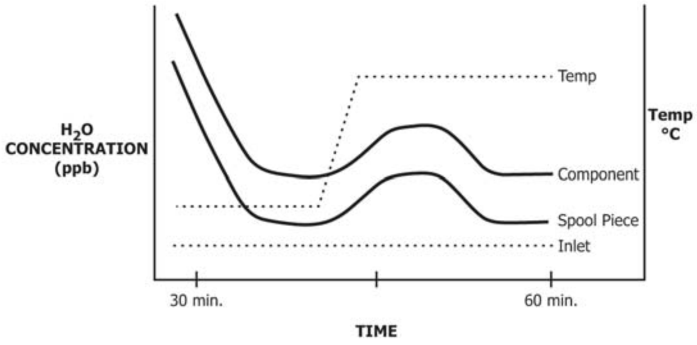


FIG. 3 Component Moisture Contribution

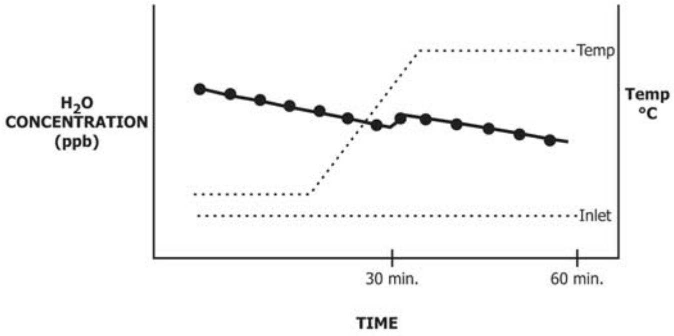


FIG. 4 Net Component Moisture Contribution

11.2 By using a given analyzer performing multiple analyses of the members of a test component batch, a reliable comparison of moisture contribution by the members of that batch can be obtained. The percentage differences between the moisture content of the individual components can be significant, even if the absolute moisture values are not. This means that, although this test method can provide a means of comparing one component to others, these results should not be considered absolute moisture values.

12. Keywords

12.1 components; contamination; gas distribution; moisture analyzer; moisture contribution; semiconductor processing; water outgassing

Date: ______________ Operator: ______________

Test Location: __________ Spool Piece: ______________

Test Component: ______________________________

Model Number: ______ Serial Number: ____________

Analyzer: ______________________________

Model Number: ______ Serial Number: ____________

Last Calibration Date: __________

Base Gas: ______________________________

Flow Meter: __________ Model Number: __________

Calibration Date: ____ Cal Temperature: ________ °C

Cal Pressure: ______ Cal Gas: __________

MFC: ______________ Model Number: __________

Calibration Date: ____ Cal Temperature: ________ °C

Cal Pressure: ______ 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.

FIG. 5 Moisture Contribution Data Table (A)

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

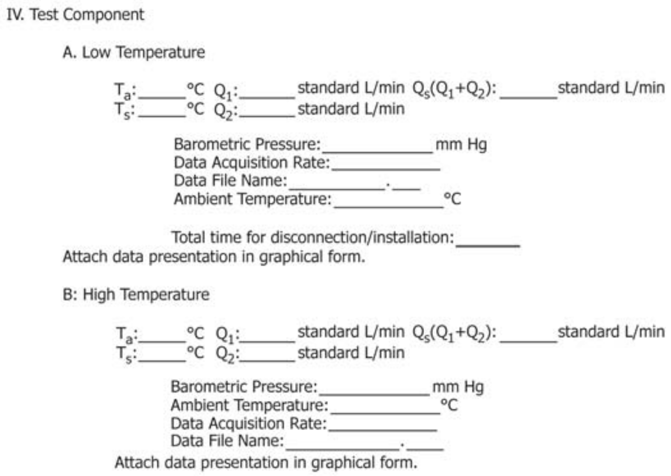
Ambient Temperature: __________ °C

Data Acquisition Rate: __________

Data File Name: __________ . ___

Attach data presentation in graphical form.

FIG. 6 Moisture Contribution Data Table (B)



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. 7 Moisture Contribution Data Table (C)

APPENDIXES

(Nonmandatory Information)

X1. ALTERNATIVE TEST

X1.1 This test method may 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*—This appendix demonstrates the calculation of total moisture 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 moisture,
MW_B = molecular weight of base gas,
MW_I = molecular weight of moisture,
n_B = number of moles of base gas,
n_I = number of moles of moisture,
p_B = pressure of base gas,
p_I = pressure of moisture,
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 moisture.

X2.2 Most commercial instruments report the measured moisture 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 moisture concentrations:

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

The equations defining ppmv and ppmw may be simplified to:

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

X2.2.2 At low pressures, assume both gases obey the ideal gas law:

$$p_I V_I = n_I RT \quad \text{(X2.1)}$$
$$p_B V_B = n_B RT \quad \text{(X2.2)}$$

Dividing Eq X2.1 by Eq X2.2:

$$p_I V_I / p_B V_B = n_I / n_B \quad \text{(X2.3)}$$

Using Amagat's law of partial volumes:

$$p_I = p_B = p_T$$

and:

Eq X2.3 becomes:

$$V_I / V_B = n_I / n_B \quad \text{(X2.4)}$$

with:

$$n_I = m_I / MW_I \quad (X2.5)$$

$$n_B = m_B / MW_B \quad (X2.6)$$

X2.2.3 Substituting Eq X2.5 and Eq X2.6 into Eq X2.4 and multiplying by 10^6:

$$(V_I/V_B) \times 10^6 = (m_I/m_B) \times (MWB/MW_I) \times 10^6 \quad (X2.7)$$

X2.2.4 Substituting the simplified definitions of ppmv and ppmw into Eq X2.7:

$$ppmv = ppmw \times (MW_B/MW_I) \quad (X2.8)$$

$$ppmw = ppmv \times (MW_I/MW_B) \quad (X2.9)$$

X2.2.5 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).

X2.3.1 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:

$$Q_{1,m} = Q_{1,v} \times \rho_R \quad (X2.10)$$

X2.3.2 Any flow diverted to the analyzer(s) ($Q_{2,m}$) must also be included in the calculation of total mass flow:

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

X2.3.3 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:

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

X2.3.4 The ppmv versus time curve given in Fig. 3 can now be converted into a ppmw versus mass of base gas curve. A unit area under such a curve is given by:

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

X2.3.5 The total mass of moisture measured ($m_{I,T}$) is given by the area under the ppmw versus m_B curve:

$$m_{I,T} = \int_0^{m_{B,T}} \text{ppmwd}m_B \quad (X2.14)$$

X2.3.6 Integration may be carried out numerically by the trapezoid, Simpson's, or by any of the other quadrature methods. 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 of moisture from a component will be evaluated using the trapezoid rule for the data in the following example, (data acquisition rate is 1 point every 30 s):

$$Q_{1,V} = 1000\ \text{cm}^3/\text{min at } 101.3\ \text{kPa}, 21°C\ (14.73\ \text{psia}, 70°F)$$

$$Q_{2,V} = 2000\ \text{cm}^3/\text{min at } 101.3\ \text{kPa}, 21°C\ (14.73\ \text{psia}, 70°F)$$

$$\rho_R = 0.001656\ \text{g/cm}^3 \text{ at } 101.3\ \text{kPa}, 21°C\ (14.73\ \text{psia}, 70°F)$$

$MW\ moisture = 18.015$; $MW\ argon = 39.948$

$$ppbw = 0.4510 \times ppbv \quad (X2.15)$$

$$Q_{s,m} = 50\ \text{cm}^3/\text{s} \times 0.001656\ \text{g/cm}^3 = 0.0828\ \text{g/s Ar}$$

$$m_B = 0.0828\ \text{g Ar/s} \times t \quad (X2.16)$$

X2.3.7 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 moisture measured at a given time. Column 3 is the mass of argon that has passed through the component, calculated from Eq X2.11. Column 4 is the ppbw of moisture equivalent calculated from Eq X2.10. 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.

X2.3.8 The final result for this test is 2987.636 ng moisture, or 2.99 µg.

X2.4 The use of this calculation for the determination of moisture mass contribution is only quantitative if the instrumentation is gravimetrically calibrated prior to the test procedure.

X2.5 *Avoiding Misinterpretation of Results:*

X2.5.1 The use of this procedure will provide results with a certain minimum absolute error. Due to the difficulty of obtaining accurate standards for moisture analyzer calibration, combined with the moisture response of any particular moisture analyzer, the propagation of errors can easily yield results that deviate by more than 200 % from absolute. This does not invalidate the use of this procedure for comparing test components.

X2.5.2 By using a given analyzer performing multiple analyses of the members of a test component batch, a reliable comparison of moisture contribution by the members of that batch can be obtained. The percentage differences between the moisture content of the individual components can be significant, even if the absolute moisture values are not. This means that, although this test method can provide a means of comparing one component to others, one should not consider these results absolute moisture values.

TABLE X2.1 Data for Sample Calculation

NOTE 1—Analyzer exhaust 2000 cc/s. Bypass 1000 cc/s at approximately 70°F, 1 atm data acquisition rate one point every 30 s.

Time, s	ppbv	m_B, g Ar	ppbw ng H_2O/g Ar	Area of Segment ng H_2O	Total Area ng H_2O
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
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

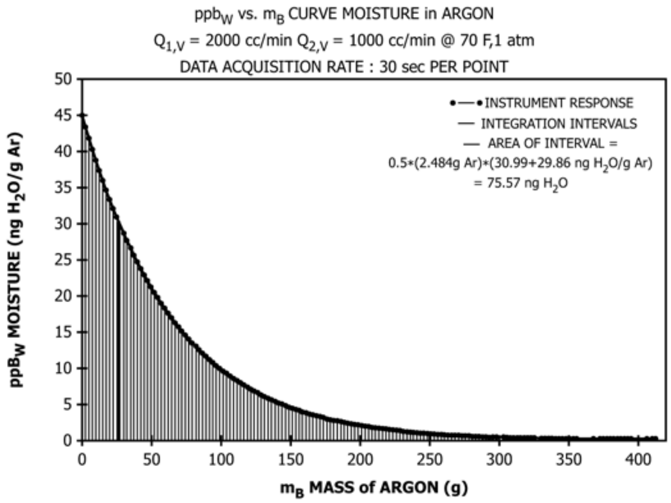


FIG. X2.1 Example Calculation Graph

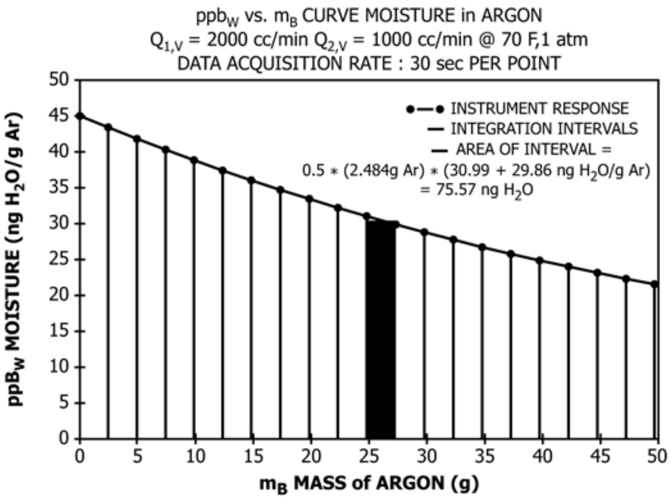


FIG. X2.2 Example Calculation Graph

X3. INCREMENTAL COMPONENT CAPACITY FOR MOISTURE

X3.1 The incremental capacity of a component for moisture is the amount of "extra" moisture that can be taken up from a given baseline concentration. This may be determined after baking by calculating the area under a pulse once consistent output is obtained (the "equilibrium" condition just mentioned), and summing the difference between this area and the area obtained for each of the preceding pulses as follows: where:

$$\text{Incremental component capacity for moisture} = \sum_{\text{all peaks}} \left[(\text{peak area at equilibrium}) - (\text{peak area observed}) \right].$$

X3.1.1 The moisture concentration must be allowed to return to its starting baseline concentration after each pulse for an accurate capacity determination.

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