



Designation: F1397 – 93 (Reapproved 2020)

Standard Test Method for Determination of Moisture Contribution by Gas Distribution System Components¹

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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 ($\frac{1}{2}$ -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, health, and environmental practices and deter-*

mine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 5.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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.

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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.

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 $\mu\text{mole/mole}$ (such as $\mu\text{L/L}$). The same as molar parts per million (ppm).

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

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 (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 (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 (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

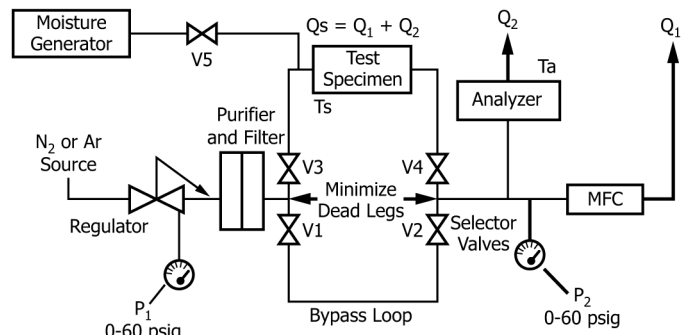


FIG. 1 Test Schematic

handling of test components for trace moisture 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 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 $\leq 0.02 \mu\text{m}$) 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 $\pm 2^\circ\text{C}$ (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.

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

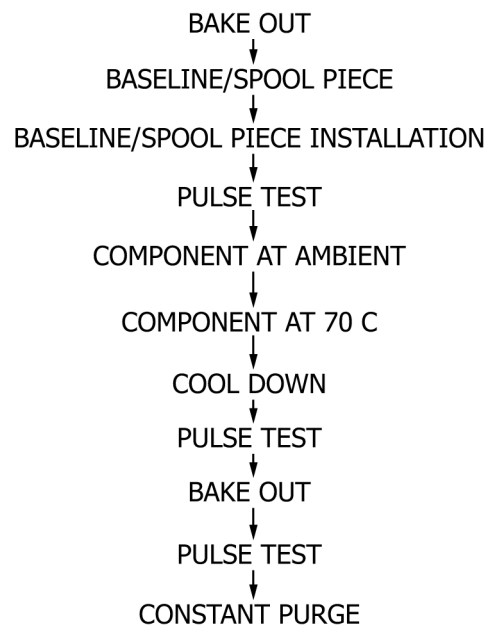


FIG. 2 Test Procedure Sequence

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 preferable 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 preferable 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

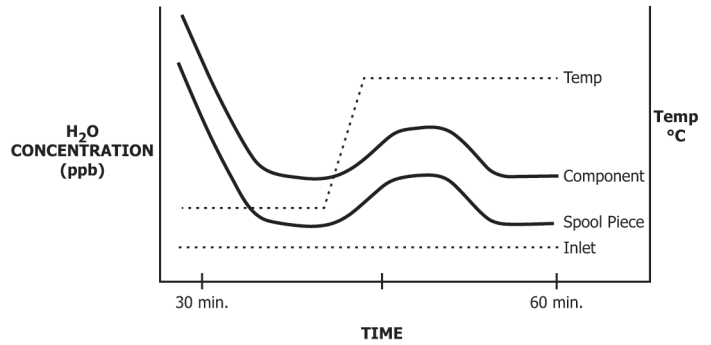


FIG. 3 Component Moisture Contribution

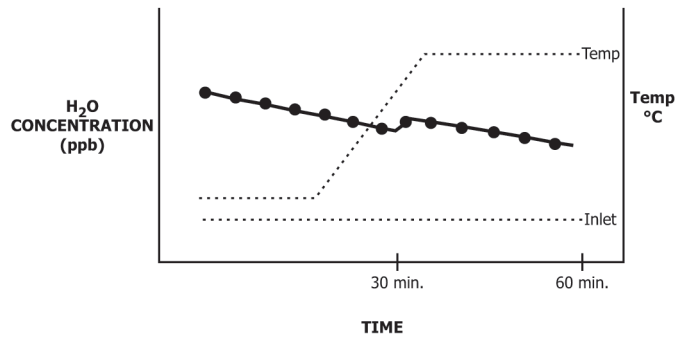


FIG. 4 Net Component Moisture Contribution

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.

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)