



Designation: F 1398 – 93 (Reapproved 1999)

Standard Test Method for Determination of Total Hydrocarbon 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 the testing of components for total hydrocarbons (THC) contribution to a gas distribution system 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 in the gaseous form, 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 components larger than 12.7-mm (1/2-in.) outside diameter nominal size. This test method could be applied to larger components; however, the stated volumetric flow rate may not provide adequate mixing to ensure a representative sample. Higher flow rates may improve the mixing but excessively dilute the sample.

1.3.2 Different instrumental methods (such as flame ionization detector (FID), mass spectrometer (MS)) will yield total hydrocarbon (THC) levels that are not comparable due to different sensitivities to different molecular species. Hydrocarbon contaminants of high-purity gas distribution systems can be subdivided into two general categories: (1) noncondensable hydrocarbons ($<C_4$), that are present due to difficulty of removal and relative atmospheric abundance, and (2) condensable hydrocarbons, that are often left behind on component

surfaces as residues. Condensable hydrocarbons include pump oils, degreasing agents, and polishing compound vehicles.

1.3.3 Because of the tremendous disparity of hydrocarbon species, it is suggested that direct comparisons be made only among data gathered using the same detection method.

1.3.4 This test method is intended for use by operators who understand 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 component, spool piece, or test stand by a uniform and complete wrapping of the item with resistant heat tape.

2.1.4 *methane (CH_4) equivalent*—that concentration of CH_4 that causes the same instrument response as the sample.

2.1.4.1 *Discussion*—The calibration gas contains a known concentration of methane (CH_4). Instrument response to zero gas and span gas defines the calibration curve for the analyzer. Instrument response to the sample is the summation of the response for each hydrocarbon reaching the detector. The

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concentration reported is the methane concentration, from the calibration curve that corresponds to the instrument response to the sample. The sample's concentration is equivalent to the methane concentration that would produce the same instrument response.

2.1.5 *minimum detection limit (MDL) of the instrument*—the lowest instrument response above zero detectable that is readable by the instrument and at least two times the amplitude of the noise.

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

2.1.7 *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.

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

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

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

2.1.11 *zero gas*—a gas that has a THC concentration below the MDL of the analytical instrument. This purified gas is used for both instrument calibration and component testing.

2.2 Abbreviations: Abbreviations:

2.2.1 *FID*—flame ionization detector.

2.2.2 *MFC*—mass flow controller.

2.2.3 *MS*—mass spectrometer.

2.2.4 *ppbv*—parts per billion by volume assuming ideal gas behavior, equivalent to nmole/mole (such as nL/L).

2.2.4.1 *Discussion*—The same as molar parts per billion (ppb).

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

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

2.2.6.1 *Discussion*—The same as molar parts per million (ppm).

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

2.2.8 *slpm*—standard liters per minute. The gas volumetric flow rate measured in liters per minute at 0.0°C (32°F) and 101.3 kPa (1 atm).

2.2.9 *THC*—total hydrocarbon.

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 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 located in contact with the outside wall of the component or spool piece.

2.3.8 $V-1$, $V-2$ = inlet and outlet valves of bypass loop, respectively.

2.3.9 $V-3$, $V-4$ = inlet and outlet valves of test loop, respectively.

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 purposes of qualification for this installation.

4. Apparatus

4.1 Materials:

4.1.1 *Test Gas*, purified nitrogen or argon with a maximum THC concentration not exceeding the manufacturer's stated minimum detection limit of the instrument (MDL). Particulate filtration of the test gas to 0.02 μm or finer is required, using a typical electronics grade filter. The filter must be compatible with the 94°C (200°F) bake-out.

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 shall have 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 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 ($\frac{1}{4}$ in.). The length of the sample line from the tee (installed upstream of the pressure gage P_2) to the analyzer shall not be more than 600 mm 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 and must be 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 *THC Analyzer*—The THC 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. The baseline must return to levels <100 ppbv, before and after the tests. Deviations greater than this require that all results be rejected and a new test component be tested. The analyzer must be capable of accurately recording changes in THC concentrations on a real-time basis, within the constraints of the data acquisition system.

4.2.2 *THC analyzer calibration*—Two-point calibration, zero and span, is to be performed regularly. Zero gas is defined as below the manufacturer's stated MDL of the instrument, supplied by purified gas, with the purifier in close proximity to the analyzer. Span gas is analyzed at the lowest possible detection range, which may not be at the lowest detection range of the instrument. Span gas is 5–10 ppmv methane, $\pm 20\%$ for FID. Calibration is based on traceable methane concentration in the base gas (nitrogen or argon).

4.2.3 *Flame Ionization Detector (FID)*—The FID detects hydrocarbon species by ionizing the organic material in a flame. Ions produced in the hydrogen flame yield a measurable current, directly related to the quantity of hydrocarbons introduced to the flame. Burner gases, hydrogen, and air are not to contain measurable amounts of THC. Purifiers are available for this purpose (especially to remove methane). Burner gases must be maintained at a temperature between 18 and 26°C (64 and 78°F).

4.2.3.1 The THC data are referred to as ppmv quantity of CH₄ equivalents, corresponding to the sum of the number of equivalent carbons. Simple, low molecular weight hydrocarbons are readily detected and quantified as total THC. Higher molecular weight hydrocarbons and more substituted hydrocarbons may not be detected well. The FID is, therefore, a specific group detector that yields quantification of total hydrocarbons for a specific detectable group.

4.3 *Pressure and Flow Control*—Upstream pressure is to be controlled with a regulator 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. However, a variable area flowmeter plus a back pressure regulator may be used instead. 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 (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 (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 Precautions:

5.1.1 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 organic analysis. Good laboratory practices must also be understood.

5.1.2 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.

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

5.1.4 Gloves are to be worn for all steps.

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

5.1.6 Precautions must be taken to insure that the temperature measured by the thermocouple is as close as possible to that of the spool piece or the test component. Use appropriate insulation and conductive shield to achieve as uniform a temperature as possible.

5.1.7 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 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 gas filter (pore size rating $\leq 0.02 \mu\text{m}$) 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 hydrocarbon 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 total hydrocarbon analyzer capable of detecting hydrocarbon concentration levels down to <50 ppb is connected to the test stand to sample the gas flowing through the test piece. The THC analyzer uses hydrogen fuel for the generation of the flame required for the FID in the instrument. The purified and filtered base gas from the test stand containing <10 ppb THC is used as the zero 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 purified by an electronic grade pressure gage. Flow measurement is carried out by a 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 THC concentration measured by the analyzer can either be recorded continuously by a data logger or collected and stored in a computer using a data acquisition program.

7. Calibration

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

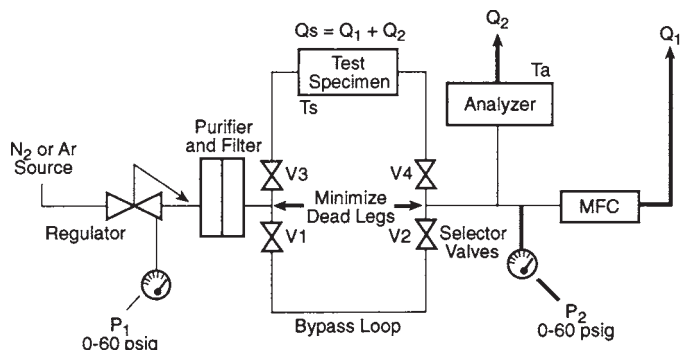


FIG. 1 Test Schematic

7.2 *THC Analyzer Calibration*—Perform two-point calibration, zero and span, regularly. Zero gas is defined as below the manufacturer’s stated MDL of the instrument, supplied by purified gas, with the purifier in close proximity to the analyzer. Analyze span gas at the lowest possible detection range, that may not be at the lowest detection range of the instrument. Span gas is 5 to 10 ppmv methane, $\pm 20\%$ for FID. Base calibration on traceable methane concentration in the base gas (nitrogen or argon).

8. Conditioning

8.1 *Pressure*—The test component is to be tested 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 in the higher mean temperature range of 69 to 71°C (156 to 160°F). T_a must not deviate more than $\pm 1^\circ\text{C}$ (2°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 Gas quality must be maintained at flow specified in 8.3.

9. Procedure (see Fig. 2)

9.1 This test procedure applies to the use of an FID (see Fig. 2).

9.2 *Bake-out*—With the spool piece 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 THC concentration is stable below 50 ppbv. Flow of the gas is specified in 8.3. Cool to lower T_s . Close Valves V-1 and V-2.

9.3 *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 THC concentration has attained a level of <510 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 THC 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 (<100 ppbv). Cool until the lower T_s is reached.

9.4 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.

9.5 Initiate flow in accordance with 8.3. Monitor T_s and T_a in accordance with 8.2. Monitor THC until a stable baseline is reestablished in accordance with 9.3 (<50 ppbv). Utilizing heat tape, heat the spool piece and upstream tubing to within 80 mm of the upstream valve. Turn on current and monitor the THC 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 (<50 ppbv). Cool until the lower T_s is reached.

9.6 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 the spool piece while maintaining flow and recap. 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 as outlined in 9.4 including time required to disconnect and connect. The spool piece must not be removed from the glove bag for the duration of the test.

9.6.1 *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.

9.7 Initiate flow in accordance with 8.3. Monitor T_s and T_a in accordance with 8.2 for ambient temperature. Monitor THC until a stable baseline in accordance with 9.3 is re-established (<100 ppbv). Terminate the test after 3 h if a stable baseline is not achieved.

9.8 Utilizing heat tape, heat the test component and upstream tubing to within 80 mm of the upstream valve. Turn on the current and monitor the THC 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

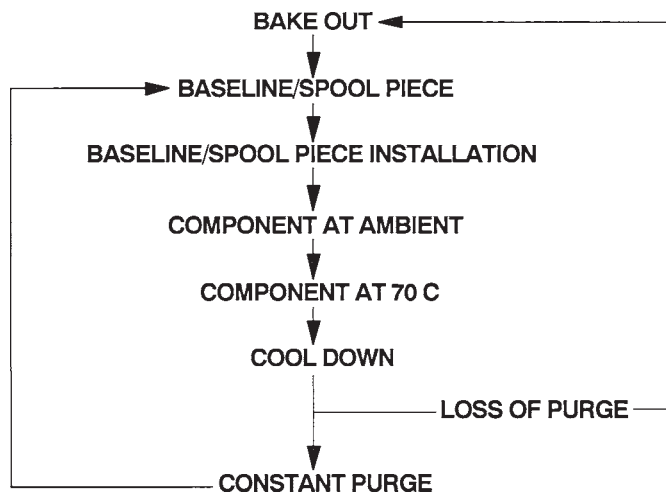


FIG. 2 Test Procedure Sequence

baseline is reestablished (<50 ppbv). Terminate the test in 3 h if a stable baseline is not achieved. Cool until the lower T_s is reached.

9.9 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 the test component while maintaining flow and cap. 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.

9.10 With Valves V-1, V-2, V-3, and V-4 open, maintain a purge through the system with a purified gas. Q_f 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 9.3 (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.

9.11 *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 *Data Analysis*— Refer to X2.1 for a numerical example.

10.2 Report the following information:

10.2.1 *Data Collection*— Present a plot of THC concentration versus time for (1) the respective spool piece baseline and

(2) the test component. Plot the elevated temperature data 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).

10.2.2 *Data Handling*— To determine THC contribution of the test component, compute the difference between the spool piece plot and the component plot derived in 10.1 and graph the results. Use a dual y-axis (concentration ppbv and T_s) (refer to Fig. 4).

10.2.3 Complete the table given in Figs. 5-7.

11. Precision and Bias

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

12. Keywords

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

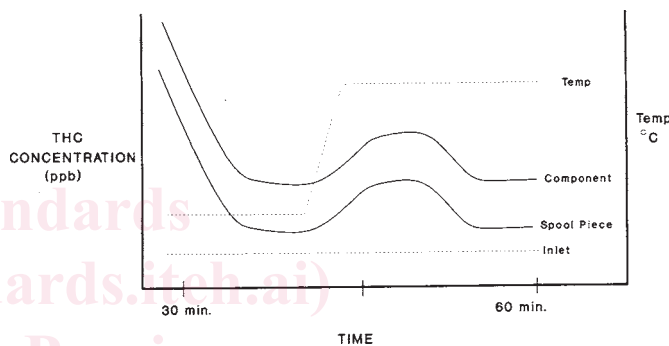


FIG. 3 Component THC Contribution

ASTM F1398-93(1999)

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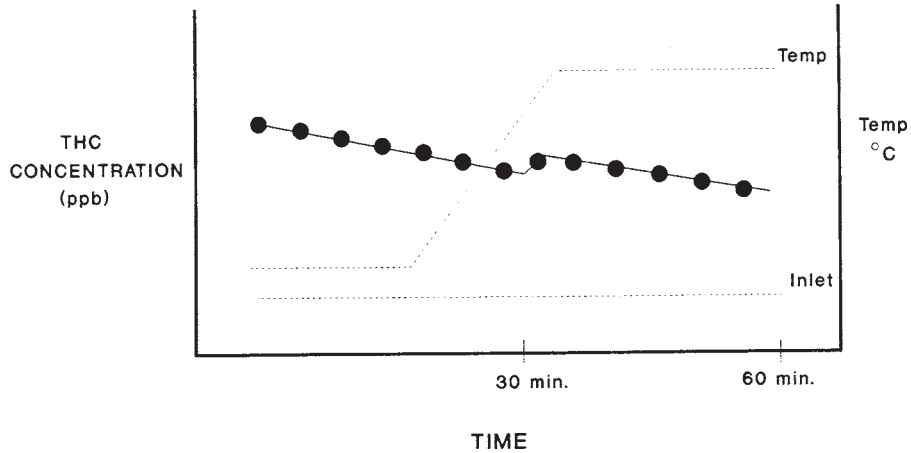


FIG. 4 Net Component THC Contribution

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)
 T₁: _____ °C Q₁: _____ standard L/min Q₁(Q₁+Q₂): _____ standard L/min
 T₂: _____ °C Q₂: _____ standard L/min P₂: _____ 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

T₁: _____ °C Q₁: _____ standard L/min Q₁(Q₁+Q₂): _____ standard L/min
 T₂: _____ °C Q₂: _____ 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 Hydrocarbon Contribution Data Table