**Designation: D7904 – 15**

# Standard Test Method for Determination of Water Vapor (Moisture Concentration) in Natural Gas by Tunable Diode Laser Spectroscopy (TDLAS)[1]

## 1. Scope

1.1 This test method covers online determination of vapor phase moisture concentration in natural gas using a tunable diode laser absorption spectroscopy (TDLAS) analyzer also known as a "TDL analyzer." The particular wavelength for moisture measurement varies by manufacturer; typically between 1000 and 10 000 nm with an individual laser having a tunable range of less than 10 nm.

1.2 Process stream pressures can range from 700-mbar to 700-bar gage. TDLAS is performed at pressures near atmospheric (700- to 2000-mbar gage); therefore, pressure reduction is typically required. TDLAS can be performed in vacuum conditions with good results; however, the sample conditioning requirements are different because of higher complexity and a tendency for moisture ingress and are not covered by this test method. Generally speaking, the vent line of a TDL analyzer is tolerant to small pressure changes on the order of 50 to 200 mbar, but it is important to observe the manufacturer's published inlet pressure and vent pressure constraints. Large spikes or steps in backpressure may affect the analyzer readings.

1.3 The typical sample temperature range is -20 to 65°C in the analyzer cell. While sample system design is not covered by this standard, it is common practice to heat the sample transport line to around 50°C to avoid concentration changes associated with adsorption and desorption of moisture along the walls of the sample transport line.

1.4 The moisture concentration range is 1 to 10 000 parts per million by volume (ppmv). It is unlikely that one spectrometer cell will be used to measure this entire range. For example, a TDL spectrometer may have a maximum measurement of 1 ppmv, 100 ppmv, 1000 ppmv, or 10 000 ppmv with varying degrees of accuracy and different lower detection limits.

1.5 TDL absorption spectroscopy measures molar ratios such as ppmv or mole percentage. Volumetric ratios (ppmv and %) are not pressure dependant. Weight-per-volume units such as milligrams of water per standard cubic metre or pounds of water per standard cubic foot can be derived from ppmv at a specific condition such as standard temperature and pressure (STP). Standard conditions may be defined differently for different regions and entities. The moisture dew point can be estimated from ppmv and pressure. Refer to Test Method D1142 and ISO 18453.

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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.* **Some specific hazards statements are given in Section 8 on Hazards.**

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1142 Test Method for Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature

D4150 Terminology Relating to Gaseous Fuels

D5503 Practice for Natural Gas Sample-Handling and Conditioning Systems for Pipeline Instrumentation

D5454 Test Method for Water Vapor Content of Gaseous Fuels Using Electronic Moisture Analyzers

2.2 *ISO Standards:*[3]

ISO 10715 Natural Gas Sampling Guidelines

ISO 18453 Natural Gas—Correlation Between Water Content and Water Dew Point

## 3. Terminology

3.1 *Definitions:* Also refer to Terminology D4150.

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[1] This test method is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.12 on On-Line/At-Line Analysis of Gaseous Fuels.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

3.1.1 *absorption spectroscopy, n*—refers to spectroscopic techniques that measure the absorption of electromagnetic radiation (such as light), as a function of frequency or wavelength, because of its interaction with a sample.

3.1.2 *adsorption, n*—adhesion of molecules to a solid surface forming a molecular or atomic film.

3.1.3 *chemometrics, n*—field of science relating measurements made on a chemical system or process to the state of the system via application of mathematical or statistical methods.

3.1.4 *desorption, n*—phenomenon whereby a substance is released from a surface (the opposite of adsorption).

3.1.5 *heat trace, n*—ribbon-shaped tape that uses electrical resistance or steam to generate heat.

3.1.5.1 *Discussion*—Heat trace tape is attached to sample tubing and other sample conditioning components to avoid condensation and stabilize the temperature of the wetted components and the gas stream.

3.1.6 *moisture dew point, n*—A temperature and pressure at which water vapor begins to condense into liquid.

3.1.6.1 *Discussion*—For a given concentration of water vapor, the dew point temperature is a function of pressure. A dew point curve is shown in Fig. 1 with the dew point temperature on the *x*-axis and pressure on the *y*-axis.

3.1.7 *nanometre, n*—unit of length; 1 nm = 1/1 000 000 000th of a metre.

3.1.8 *selectivity, n*—refers to the extent to which TDLAS can detect moisture in gas matrices without significant interferences from other components in the mixture.

3.1.9 *standard condition for temperature and pressure, STP, n*—standard set of conditions established to allow the comparison of different sets of data.

3.1.10 *tunable diode laser absorption spectroscopy, TDLAS, n*—technique for measuring the concentration of a specific component, such as water vapor, in a gaseous sample by absorption spectrometry using tunable diode lasers.

## 4. Summary of Test Method

4.1 A representative sample of the gas is extracted from a process pipe or pipeline and is transferred by a sample transport line through an appropriately designed sampling system to the inlet of a moisture analyzer. The sample must be conditioned with a minimum, preferably negligible, impact on the moisture concentration. The gas flows continuously through the analyzer and is vented to atmosphere, or to flare, or back to the process stream depending on application and regulatory requirements.

4.2 The gas sample stream flows through the measurement cell. An overall diagram of the system is shown in Fig. 2. A solid state laser with a narrow wavelength range is used as a light source. Electronics drive the laser and a thermoelectric cooler, which precisely stabilizes the laser temperature. The laser generates a near-infrared beam of light that passes through the cell window, is typically reflected using a mirror (or mirrors) within the cell, and then returns back through the window and into a photodiode detector. The photodiode signal is used to determine how much light is absorbed at specific wavelengths.

4.3 Fig. 3 is a graph of typical regions in the near-infrared spectrum where water will be absorbed. In the graph, the *x*-axis indicates the wavelength. The *y*-axis indicates the "transmission" of light where 1.0 (or 100%) is the maximum. Where the transmission is less than 1.0, absorbance by water is indicated. The vertical lines within the graph indicate the magnitude of absorption at specific wavelengths. Each individual absorption line can be potentially utilized for TDLAS moisture measurement. The actual wavelength used will vary based on manufacturer, background composition, measurement specification requirements, and laser availability.

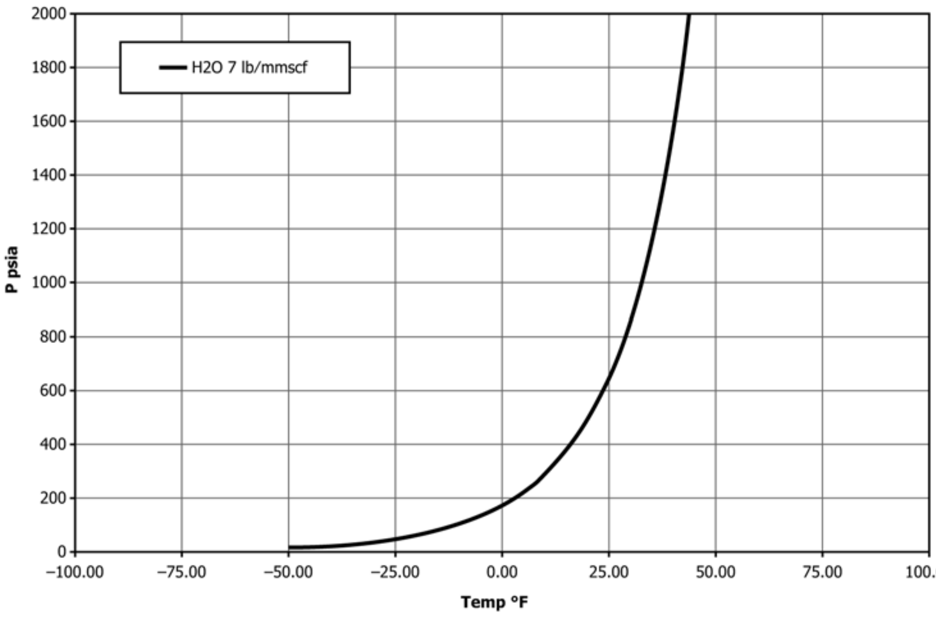

**FIG. 1 Example of Moisture Dew Point Curve Calculated Using Equations in Test Method D1142**

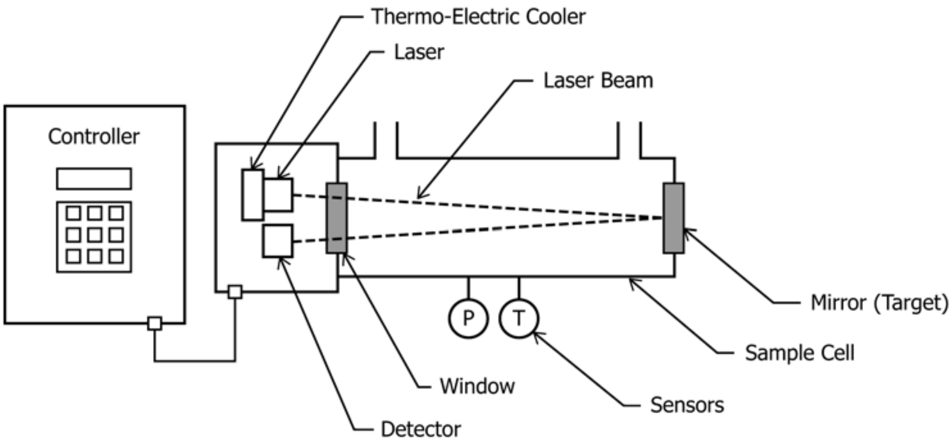

FIG. 2 Main Components of the TDLAS System

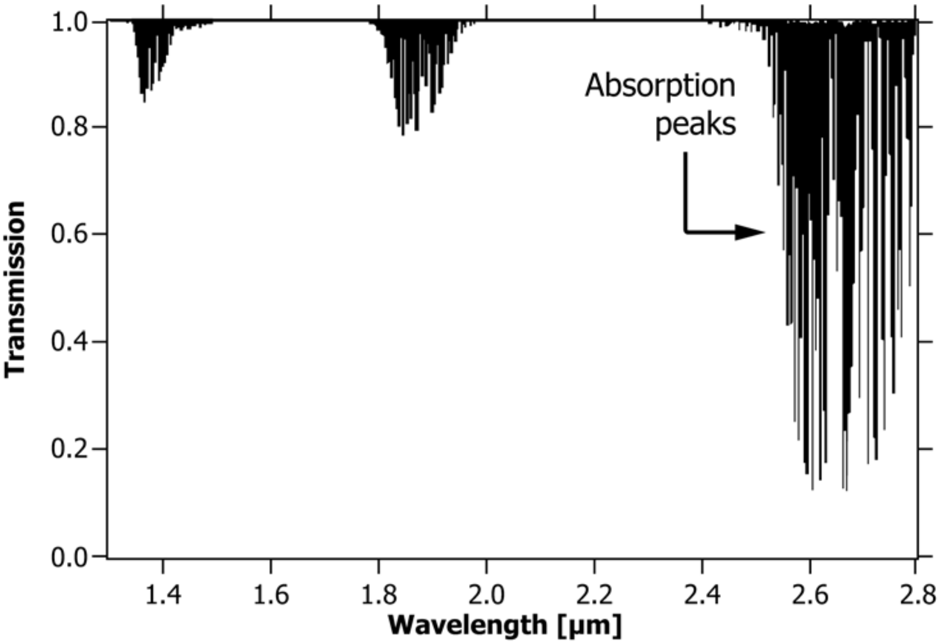

FIG. 3 Water Transmittance in the Near Infrared (NIR) Spectrum
SOURCE: HITRAN

4.4 The sensitivity of the measurement is determined by the absorption as well as the length of the laser beam path (path length) within the sample cell. The path length is fixed and can range from about 30 cm to 30 m depending on the measurement range and the wavelength used. By optimizing the path length and wavelength, linearity less than 0.1% can be readily achieved. The TDLAS manufacturer must be consulted for actual linearity specifications.

4.5 This test method can be used as a guideline for installation so that good moisture measurement can be achieved using a TDLAS analyzer. Also, a procedure is outlined for validating measurement integrity.

## 5. Significance and Use

5.1 Moisture measurement in natural gas is performed to ensure sufficiently low levels for gas purchase contracts and to prevent corrosion. Moisture may also contribute to the formation of hydrates.

5.2 The significance of applying TDLAS for the measurement of moisture in natural gas is TDLAS analyzers may have a very high degree of selectivity and minimal interference in many natural gas streams. Additionally, the sensing components of the analyzer are not wetted by the natural gas, limiting the potential damage from corrosives such as hydrogen sulfide

($H_2S$) and liquid contaminants such as ethylene glycol or compressor oils. As a result, the TDLAS analyzer is able to detect changes in concentration with relatively rapid response. It should be noted that the mirrors of a TDLAS analyzer may be fouled if large quantities of condensed liquids enter the sample cell. In most cases the mirror can be cleaned without the need for recalibration or realignment.

5.3 Primary applications covered in this method are listed in 5.3.1 – 5.3.3. Each application may have differing requirements and methods for gas sampling. Additionally, different natural gas applications may have unique spectroscopic considerations.

5.3.1 Raw natural gas is found in production, gathering sites, and inlets to gas-processing plants characterized by potentially high levels of water ($H_2O$), carbon dioxide ($CO_2$), hydrogen sulfide ($H_2S$), and heavy hydrocarbons. Gas-conditioning plants and skids are normally used to remove $H_2O$, $CO_2$, $H_2S$, and other contaminants. Typical moisture concentration after dehydration is roughly 20 to 200 pppmv. Protection from liquid carryover such as heavy hydrocarbons and glycols in the sample lines is necessary to prevent liquid pooling in the cell or the sample components.

5.3.2 Underground gas storage facilities are high-pressure caverns used to store large volumes of gas for use during peak demand. Underground storage caverns can reach pressures as high as 275 bar. Multistage and heated regulator systems are usually required to overcome significant temperature drops resulting from gas expansion in the sample.

5.3.3 High-quality "sales gas" is found in transportation pipelines, natural gas distribution (utilities), and natural gas power plant inlets. The gas is characterized by a very high percentage of methane (90 to 100 %) with small quantities of other hydrocarbons and trace levels of contaminates.

## 6. Interferences

6.1 TDLAS analyzers can be highly selective. They are capable of measuring the target component with very little interference from background composition, with some limitations. There may be some interference from background components. For example, at some wavelengths, methane may absorb at the same wavelength as moisture. If interferences exist at a particular wavelength, a different wavelength can be employed and other techniques such as chemometrics, background compensation, or differential measurements may be utilized. Since hundreds of possible wavelengths are available in the near-infrared band for measuring moisture, it is not practical to list the potential interferences.

6.2 Background composition changes may also affect the measurement from TDLAS analyzers because of a phenomenon called "collisional broadening." Collisional broadening changes the shape of the absorption "peak." The broadening effect may be different at different wavelengths, or at different pressures and temperatures, or both. TDLAS manufacturers should publish the gas concentration ranges of the various components of the background gas in which the accuracy and repeatability specifications are valid.

## 7. Apparatus

7.1 A TDL analyzer system includes the following subsystems: *(1)* sample extraction, *(2)* sample transport, *(3)* sample conditioning system, *(4)* TDLAS analyzer, and *(5)* vent line.

7.1.1 Sample extraction is required to obtain a representative sample from the pipeline. To maintain the best speed of response, it is recommended to reduce the pressure at the sample point. To avoid condensation that may occur from expanding the gas when it is depressurized (especially when the pipeline pressure is high), it is important to understand the phase diagram of all of the components in the gas (for example, hydrocarbons, alcohols, and water). Use an extraction probe and a regulator as shown in Fig. 4, mounted so that the tip of the probe is in the center third of the pipe diameter. If the dew point of the gas is lower than the ambient temperature after consideration for temperature reduction as a result of gas expansion through the regulator (approximately 3°C per 6 bar), all sampling apparatus such as the probe and regulator may need to be heat traced or enclosed in a heated chamber, or both. According to Practice D5503, "vapor sample must be kept at least 10°C above the hydrocarbon dew point temperature to prevent condensation of the sample."

7.1.2 *Sample Transport*—The sample transport line carries the sample from the sample extraction point to the analyzer. The length of the sample transport tubing should be as short as possible. Heat trace is absolutely necessary if the environmental temperature is close to the dew point of the sample gas. Heat trace prevents water condensation and adsorption and desorption from the walls of the tubing caused by ambient temperature changes (Fig. 5). When heat trace is employed, the entire length of tubing must be heated and insulated with no gaps. Electropolished tubing is recommended for use in analytical sample transport to reduce adsorption and desorption

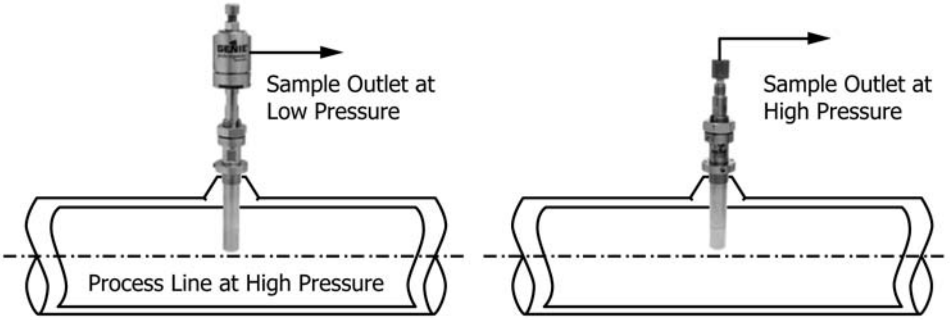

FIG. 4 Heat-Trace Tubing with Self-Regulating Heat Tape Bundled with Insulation and Protective Jacket

FIG. 5 Sample Probe with Integrated Pressure Regulator (Left) and Sample Probe with No Regulator (External Pressure Regulator to be Used) (Right)

effects and to optimize speed of response in the sample transport. For a moisture concentration below 10 ppmv, the transport line may be coated with fused silica or an equivalent for additional resistance to adsorption and desorption effects.

7.1.3 The sample conditioning system is typically installed immediately before the analyzer and contains a regulator to perform a final pressure drop, a means to control and measure flow, and filters to remove particulates and reject liquids. Additionally, the sample conditioning system may provide a means for bypassing the analyzer, to connect a reference gas for validating the analyzer, and an outlet for venting the gas. Sample system recommendations are described in detail in Practice D5503.

7.1.3.1 Similar to the sample transport line, the sample conditioning system may require heat to eliminate condensation and reduce erratic readings caused by adsorption/desorption effects. Typically, if heat is required, the sample panel will be installed in a heated building or a heated enclosure. Components such as valves, regulators, and fittings that are used in the sample extraction, sample transport, and sample conditioning shall be designated as "instrument or analytical grade." Non-instrument-grade components may have very rough wetted surfaces, be constructed with hydroscopic materials, or have internal voids and pockets in which moisture can be trapped and released unpredictably, causing erratic moisture readings.

7.1.3.2 The sample vent line transports the sample from the analyzer to a safe location. It should be an unrestricted line. Many users vent the analyzer to a high point. If so, a flame arrestor on the vent may be used to protect against lightning strikes at the vent. Also, a 180° bend with at least a 30-mm radius should be applied to the end of the vent to minimize rain and wind intrusion. Additionally, a screen of some kind on the end should be used to discourage insects that might plug the vent. Never vent the analyzer inside a building or enclosure.

## 8. Hazards

8.1 The process line may contain very high-pressure gas (tens of MPa). The sample from the process line shall be dropped close to atmospheric pressure before going into the TDL cell. The recommended practice is to use a 0- to 300-kPa regulator set at 160 to 180 kPa at the extraction point plus a 0- to 150-kPa regulator set at 70 kPa at the analyzer. This allows for longer sample runs, better pressure control, and protection against the failure of one regulator.

8.2 Observe all safety regulations related to the use of combustible gases and equipment in hazardous area locations.

## 9. Calibration and Standardization

9.1 TDL analyzers are calibrated by the manufacturer in the factory. The manufacturer may allow for reading offsets to be applied for external calibration to a trusted reference. However, it is generally not necessary to calibrate the analyzer in the field.

9.2 The procedure in Section 10 includes methods for validating the original factory calibration of a TDL analyzer.

## 10. Procedure

10.1 Connect the sample extraction, sample transport, and sample-conditioning equipment as described in Section 7. Observe the manufacturer's installation recommendations.

10.2 Connect the electrical systems (power and signals) per the manufacturer's recommendations. The method for taking concentration readings may vary by manufacturer or user; in most cases, the analyzer will have a display. Additionally, the analyzer may have a digital or analog interface.

10.3 Start the analyzer according to the manufacturer's instructions. Observe recommendations regarding electronics startup and sample system startup and allow for warm-up and purge times. Follow the manufacturer's recommendations, if any, regarding external influences such as electromagnetic interference (for example, high-power radios) or heavy vibration (for example, compressor pump).

10.4 *Analyzer Validation*—Once the analyzer has been initialized, the analyzer performance can be evaluated by comparing to a known reference. The TDL analyzer's measurement is inherently more reliable than a moisture reference gas because of the difficulty in handling, transporting, storing, and sampling such mixtures. After a successful validation, it is assumed that the analyzer's original factory certification remains valid. Generally, it is not necessary to adjust the readings of the analyzer to match the reference gas unless dictated by policy or by the manufacturer.

10.4.1 *Reference Standard: Using Binary Gas Mixture*—A binary gas mixture (for example, moisture in methane) can be used to validate a TDL moisture analyzer. In Fig. 6, a simple setup is shown. The analyzer may have a validation gas inlet built into the sample-conditioning system so the three-way ball valve is optional. Because the moisture in the mixture tends to interact with wetted surfaces such as the cylinder and sample tubing, it is best to maintain them at an elevated and stable temperature. The cylinder shall be used at a temperature above 25°C, and a heated regulator with a set point of 50°C is

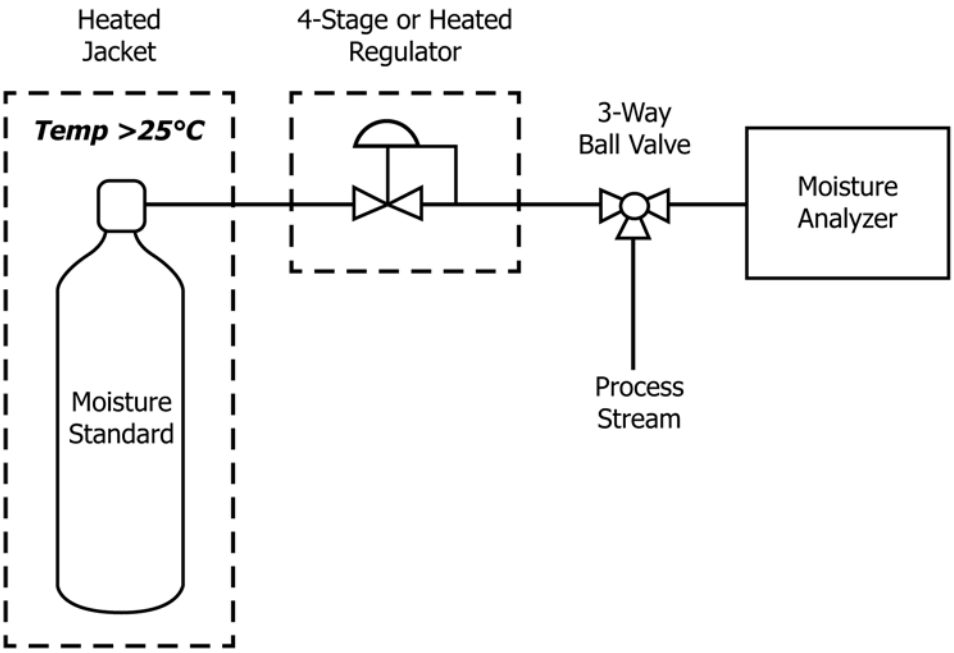

FIG. 6 Example Set Up for Introducing a Binary Gas into the Moisture Analyzer

recommended. Alternatively, a four-stage regulator may be used to drop the pressure gradually while allowing the gas temperature to stabilize. All of the components used should be "instrument grade" and the sample tubing should be electropolished stainless steel.

10.4.1.1 After setting up the validation test equipment, test for leaks by blocking the inlet to the analyzer and pressurizing the sample transport line to around 100 kPa. Do not block the outlet of the analyzer as high pressure may damage the analyzer. Inspect for leaks using soapy water or an electronic leak detector if available. If no leaks are found, the analyzer port can be opened. Monitor the readings for several minutes until they stabilize and then record the concentration reading. If the readings are within the tolerance of the reference standard's stated uncertainty plus the analyzer's stated uncertainty, the analyzer's performance has been validated and the factory certification remains valid.

10.4.2 *Reference Standard: U.S. Bureau of Mines (BOM) Chilled Mirror*—In many instances, the BOM chilled mirror is considered a primary reference for the determination of moisture in natural gas. The proper usage is described in Test Method D1142. An operator skilled at making the measurement is necessary to achieve an optimum level of accuracy. The hydrocarbons, glycol, methanol, and any other components with a dew point in the stream shall be well understood to avoid interferences. For example, if the hydrocarbons have a higher dew point temperature than the moisture, then it will not be possible to get an accurate measurement because of the interference of the hydrocarbons on the mirror. If moisture is the first condensate, chilled mirror readings may be compared with the TDL analyzer reading on a gas sample taken from approximately the same sample point and same time of day. Again, if the readings are within the uncertainty of the chilled mirror method, including the uncertainty of conversion from dew point concentration, plus the uncertainty of the TDL, then the analyzer passes validation.

10.5 *Concentration*—The concentration for performing validation may be recommended by the manufacturer. Alternatively, a value close to the normal operating concentration or the concentration at which critical decisions are made may be used. A single-point verification is sufficient for most applications to verify that the analyzer is working properly (given the inherent linearity of TDLAS response). However, a two-point verification may be required for some applications and instruments.

10.6 *Validation Frequency*—Validation should be performed when the analyzer is first commissioned. After that, the initial frequency should be defined by company policy or regulatory requirement as applicable. As a standard recommendation, the analyzer should be validated once per year.

10.7 *Validation Blends*—As explained in Section 6, the TDL analyzer's readings can be affected by the background gas. For example, if the analyzer was calibrated at the factory for moisture in methane, the analyzer may not be accurate when measuring moisture in pure carbon dioxide. For this reason, it is important to validate the analyzer using the appropriate background gas that the manufacturer should prescribe. Do not attempt to validate the analyzer using an alternative background gas without manufacturer confirmation that it is appropriate.

10.8 *Field Calibration*—The analyzer may be adjusted to match a reference standard calibration gas, if necessary. Refer to the manufacturer's instruction manual as procedures vary.

## 11. Precision and Bias

11.1 *Precision*[4]—The repeatability standard deviation from a single operator has been determined to be approximately ±0.1 to ±1.0 ppmv on an average reading of 100 ppmv.

11.1.1 Precision will vary depending on the model and manufacturer of the TDLAS analyzer. The operator must refer to the manufacturer's published repeatability specifications as well as the associated repeatability conditions for the apparatus under test.

## 12. Keywords

12.1 moisture concentration; natural gas; TDL; TDLAS; turnable diode laser absorption spectroscopy

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[4] An interlaboratory study of this test method is being conducted and a complete precision statement is expected to be available on or before 2018.

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