INTERNATIONAL

Designation: D7904 – 21

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

This standard is issued under the fixed designation D7904; 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.

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 dependent. 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 dew point can be estimated from ppmv and pressure. Refer to Test Method D1142 and ISO 18453.

1.6 *Units*—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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* **Some specific hazards statements are given in Section 8 on Hazards.**

1.8 *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. 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 (Withdrawn 2017)[3]

[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] The last approved version of this historical standard is referenced on www.astm.org.

2.2 *ISO Standards:*[4]
ISO 10715 Natural Gas Sampling Guidelines
ISO 18453 Natural Gas—Correlation Between Water Content and Water Dew Point

3. Terminology

3.1 *Definitions:* For definitions of general terms used in D03 Gaseous Fuels standards, refer to Terminology D4150.

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

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

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. 1. 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. 2 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.

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

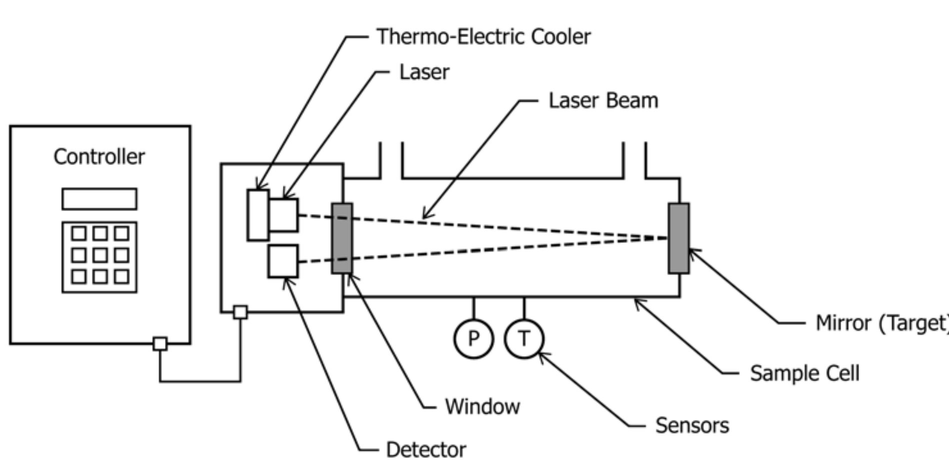

FIG. 1 Main Components of the TDLAS System

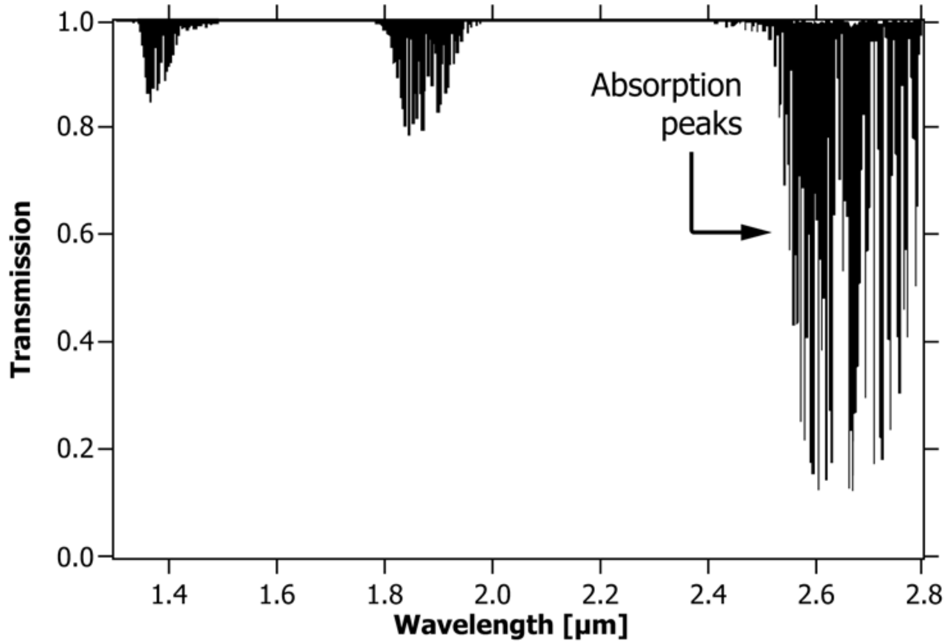

FIG. 2 Water Transmittance in the Near Infrared (NIR) Spectrum
SOURCE: HITRAN

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 ppmv. 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. 3, 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. 4). 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 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.

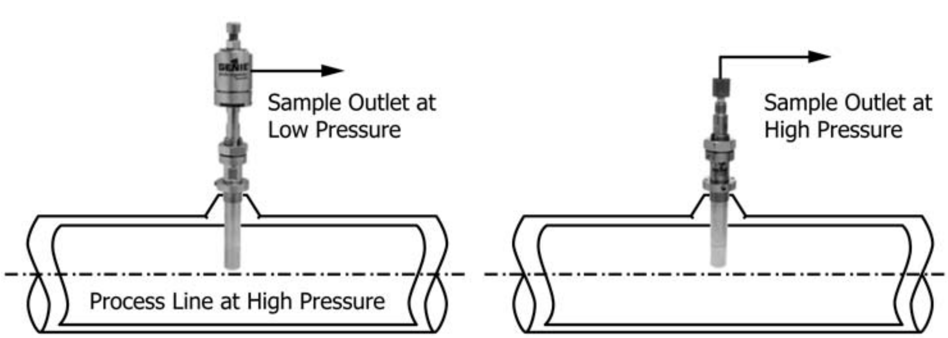

FIG. 3 Heat-Trace Tubing with Self-Regulating Heat Tape Bundled with Insulation and Protective Jacket