Proper Mechanisms for Field Validating Moisture Analyzers

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Contents

ABSTRACT

The properties of the water molecule present significant challenges in the on-line measurement of trace humidity levels in process applications. These physical attributes present even more significant challenges when field verification and calibration are desired or mandated.

In the past several decades, analyzer companies have introduced newer measurement technologies. These address the speed of response and accuracy requirements of these high value applications. The same level of innovation has not yet occurred in calibration and verification techniques, which would need to be more accurate than the measurement technique they are validating. Some of these measurement technologies will not change in calibration, removing the necessity for regular calibration or verification.

Process operators and plant engineers have an expectation that some mechanism be available to validate the performance of these analyzers, as moisture plays too vital a role in their process. This paper investigates how a plant can implement and maintain proper field validation techniques that allow for the highest level of accuracy for on-line trace moisture measurement.

1 The water molecule

1.1 Properties causing measurement challenges

Water vapor is a contaminant in hydrocarbon gas and liquid process streams and in industrial gas applications. The measurement of water vapor in these fluids offers challenges not typically found in the measurement of other process contaminants.

Water molecules adsorb onto metallic surfaces and accumulate in dead legs, on soft wetted materials such as fittings and gaskets, and in filter elements.

Changes in process conditions such as an increase in temperature will lead to off-gassing of these water molecules into the process gas or will impact the measurement at the sensor.

This adsorption will also lead to long system dry-down times. In many applications, a manufacturing process may not begin until the moisture content is below a critical level.

Water vapor can permeate through soft materials such as plastic tubing, tapes, and can leak into the process through improperly made fittings, even against high process pressures.

1.2 The need to measure moisture

Moisture measurement is critical to indicate process upsets, trends, and end-use product quality specification. When the moisture analyzer is indicating moisture content different than expected, the challenge is to verify the integrity of the measurement and validity of the reading.

1.3 The challenges in measuring moisture

When bringing a moisture analyzer on line, the sensor, the measurement system, the process, or all of the above are at a moisture content at or near ambient conditions (+10 °C dew point or 10,000 ppmv). For many process applications, the moisture content needs to be below 1 ppmv and the equilibrium moisture content is below 0.1 ppmv.

Process operators would ideally like to see the moisture content of the system dry down from ambient, through the alarm point, to the equilibrium moisture content. For example, the process may be shown as having 0 to 5 ppmv moisture with an alarm at 2 ppmv and a typical moisture content of 0.5 ppmv, but the operators may still want accurate readings from 10,000 ppmv down to the typical moisture content of 0.5 ppmv and below.

A source of frustration for process operators is that they want to believe that once the process fluid begins to flow and/or the moisture analyzer is brought on-line, the water content will quickly drop from ambient to the equilibrium moisture content as soon as the system is swept with fresh process fluid. The properties mentioned in section 1.1 often preclude this from happening. Those who have had experience with moisture measurement have gained an appreciation for the time the process takes to sweep ambient moisture of the process piping, the sample system, and the analyzer. Over time, analyzer manufacturers have introduced faster moisture measurement technologies. Although this has shortened the time to get to the desired moisture content from ambient conditions, it has also shown that the system takes time to shed the water molecules.

2 Moisture measurement technology

2.1 Typical technologies, their calibration, and their implementation

To address the challenges with field-validating the moisture analyzers, one must first understand the typical technologies used to measure moisture, their calibration scheme, and how these analyzers are implemented in the field. Each of these factors will impact the verification system chosen in various applications.

The below assumes the reader has at least a cursory understanding of the technologies, and provides a high-level overview to point out the challenges and factors involved in field-verification.

2.2 Aluminum Oxide

Oxide sensors remain among the most common technologies for process and industrial moisture measurement. Key attributes:

- Measure moisture from ambient moisture down to sub-ppm levels.
- Can be installed at process pressures, allowing for equilibrium process dew/frost point measurement.
- Calibration is independent of process fluid.
- Can measure moisture in hydrocarbon process fluids in the liquid phase without the need for vaporization.

Factory calibration of these sensors is better described as characterization. The sensor is exposed to varying levels of water vapor, with nitrogen or air as the carrier gas. At each moisture content, the raw signal (capacitance, impedance, etc.) is recorded. This table of moisture content with its corresponding signal is programmed into the analyzer or transmitter. The typical recalibration cycle is once a year.

Figure 1: Aluminum oxide sensor with temperature sensor on a process mount with Viton O-ring

2.3 Quartz Crystal Microbalance

This technology is chosen when the process operator places a higher value on speed and has a need for higher accuracy. The model is chosen by application and range. Key attributes:

- Upper range of the analyzer is limited by the range of interest. A sub-ppm analyzer may not provide accurate readings near ambient.
- Measurement is made at near-atmospheric pressure. Sample system needs to account for this pressure drop.
- Measurements are made in the vapor phase. The sample system will need to vaporize hydrocarbon liquids.

The analyzer has an on-board zero-gas (getter/purifier) and moisture generation system (permeation tube). Given this measurement scheme, the sensors are regularly tuned with a calibration gas. Field maintenance of the zero-gas system and the permeation tube can preclude the need for this analyzer to go back to the factory for calibration.

2.4 Tunable Diode Laser

This is the latest widely used technology to offer the process operator the highest speed of response and high levels of accuracy. Key attributes:

- The laser emitter and receiver are not in contact with the process fluid. Only the measurement cell is wetted, minimizing dry-down and wet-up times.

- As long as the zero reference remains unchanged, this technology requires no factory calibration.
- Measurement is made at near-atmospheric pressure. Sample system needs to account for this pressure drop.
- Measurements are made in the vapor phase. The sample system will need to vaporize hydrocarbon liquids.

Factory calibration depends on the measurement range, the process gas, and the exact measurement scheme. As an example, Panametrics first calibrates their analyzer in nitrogen as the background gas. Then the analyzer is characterized for response to moisture in one or more gas mixtures that emulate the customer's application. Each manufacturer makes their own claims on the amount of variation in background gas the analyzer can accept to maintain the stated accuracy specification of the analyzer.

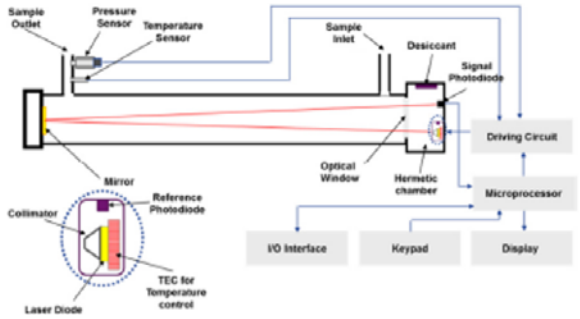

Figure 2: Schematic of TDLAS spectrometer for trace moisture measurement

3 The desire for field validation

3.1 The value of the measurement

The investment in a moisture analyzer is made based on the value in knowing the amount of water vapor in the application. In some cases, the measurement is protecting an expensive catalyst in a reformer. In ethylene production, it is ensuring that the ethylene is free of water vapor that would lead to undesired by-products in polyethylene production.

Purchasing the appropriate analyzer fit for purpose is the first step. Installing it in a fashion that will allow it to perform to its specification is also vitally important.

3.2 Regular calibration

Each of the technologies listed above have different needs when it comes to regular calibration.

Aluminum oxide is best calibrated at the factory, as it is calibrated at moisture contents ranging from as low as -100 °C frost to as wet as +10 °C dew point, if not wetter. There is significant investment required to build and maintain these calibration systems. Field calibration in this range is not practical. Calibration tends to be relatively inexpensive, as is the cost of carrying spare sensors.

Quartz crystal microbalance and tunable diode laser analyzers either self-calibrate or change insignificantly in calibration, negating the need for return to the factory for calibration. There is significant expense in sending these back to the factory, should repair or calibration be required.

Unlike oxide sensors, it may not be practical to maintain a spare QCM or TDL analyzer on-site.

3.3 Reasons for field validation

When the moisture readings are in the expected range and are changing with process conditions, the operator is less likely to want or need to validate the measurement.

If an analyzer is reporting a constant moisture content, with little or no change, the operator may question if the analyzer is responding to changes in moisture content.

Abnormally low moisture readings may indicate that the analyzer has changed in calibration.

The largest driving force for field validation is when the moisture readings are approaching or are above the alarm point. If the moisture readings are correct, the plant personnel would need to take the appropriate action, based on the high levels of moisture to protect their product, process, or safety. If the analyzer is incorrect, the instrument engineers will need to take corrective action to bring this analyzer, or another, back on-line to ensure measurement integrity.

Why not just trust the analyzer?

Contaminants and other environmental factors may result in any technology to read in error.

- Filters could be plugged and may now be off-gassing moisture into the process with changes in pressure or temperature.
- Moisture from a leak in the sample system may result in false high readings.
- Unexpected changes in background gas may be impacting a TDL analyzer.
- A spent purifier in the QCM analyzer is resulting in an artificially low moisture reading.

The consequence in an incorrect reading is inversely proportional to the amount of trust in the readings, especially when they deviate from the expected moisture content or approach/exceed the alarm point.

4 Methods for field validation

4.1 Gas cylinders

Instrument technicians are very familiar with the use of calibration cylinders. These are used extensively to calibrate other analyzers such as oxygen analyzers. There are several manufacturers of calibration cylinders for water vapor. Typically, these are provided with nitrogen as a background gas. Although available down to 1 ppmv, practically, 10 ppmv has been the lower limit.

Figure 3: Gas cylinders

Nitrogen as a background gas tends not to be the problem, especially if the analyzer is insensitive to background gas or if the analyzer has a nitrogen mode. In the case of a TDL analyzer, if the analyzer reads properly in nitrogen, but the readings are questionable in the process gas, has the operator fully validated the analyzer?

Cylinders are only as accurate as the analyzer that checked the moisture content of the gas entering the cylinder. The accuracy of this standard may not match the accuracy of the analyzer the cylinder is validating.

If the analyzer is reporting 0.5 ppmv in natural gas, and this measurement is in question, would checking the moisture content in nitrogen at 10 ppmv validate the analyzer?

Improvements have been made in the quality of gas cylinders today, but one must still be careful of the following:

- Water will adsorb to the inner surface of the cylinder. The moisture content of the gas may increase as the cylinder empties and the adsorbed water molecules desorb into the exiting gas.
- Regulators need to be properly purged so as not to introduce excess moisture into the analyzer when performing the validation.
- Aged cylinders need to be replaced.

4.2 Moisture generators

The most common technology used today to generate known amounts of moisture in a background gas is a permeation tube. These are used in conjunction with a purifier to attain a zero-gas. This zero-gas is then passed by the permeation tube, allowing water vapor to permeate from the wet side to the dry side. The amount of water vapor exiting with the carrier gas is a function of temperature, flow rate, and the permeation tube chosen.

The difficulty with implementing this in the field is that temperature and flow are difficult to control accurately in a field environment. The carrier gas is often the process gas. The purifier must be sized to remove water from this gas. There is no reasonable means to know if the purifier is still able to remove water vapor from the background gas.

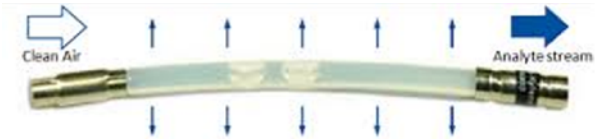

Figure 4: Permeation tubes

4.3 Uncertainty in the validation

In both cases above, the uncertainty of the validation technique can raise more questions, when the validation was intended to retire concerns. When using either technique, great care must be taken to implement the technique, to minimize external factors.

The above techniques can validate the analyzer at a specific moisture content, but may not be able to validate the analyzer at the measurement it is currently reading or meant to read.

- The process operator may believe the liquid butane to be at a moisture content of 1 ppmw. Will a calibration cylinder of 1 ppmv in nitrogen validate his moisture analyzer?
- Another operator believes the moisture content of his hydrogen/nitrogen mixture to be 1 ppmv and his analyzer is reporting 5 ppmv. Should he order a 1 ppmv standard or a 5 ppmv standard (or both)?
- An aluminum oxide sensor is reporting 10 ppmv at 1000 psig. The sensor is calibrated in dew point, thus converting -24 °C frost point to 10 ppmv. Will a 10 ppmv cylinder at 100 psig validate the analyzer (-44 °C frost point), or should he use a 500 ppmv cylinder at atmospheric pressure (-27 °C frost point)?

4.4 Redundant sensors

The value of the moisture measurement might be such that redundant sensors are a practical method for validating a moisture measurement. If multiple sensors are implemented, there is a strong argument that these are exposed to the same fluid continuously, ensuring that they are exposed to the same process conditions and upsets. These should be on the same calibration cycle, to mitigate differences in reading based on off-cycle rate of change in calibration.

One may choose to display the readings from all sensors at the same time, implementing an averaging and/or polling routine. Another method may be to display the readings of one sensor, and then only display the redundant sensor(s) when questions arise.

Mixing technologies may also offer an advantage. One example might be to have an aluminum oxide sensor mounted on the outlet of a TDL analyzer. Although there may be an accuracy mismatch, most often, it is large differences in moisture reading expectation that drive questions. Employing multiple technologies may point to the source of the reading discrepancy, as an upset condition or a contaminant that may impact one technology and not the other.

Having multiple sensors on-line can help identify if the sensor is performing well, especially if the redundant sensors agree with it. Should these readings disagree with the operator's expectations, this method doesn't point to the discrepancy.

4.5 Validating measurements with a portable moisture analyzer

A portable moisture analyzer can answer questions that a calibration standard is unable to. It can report the moisture of the process, and if this is different than what the dedicated moisture analyzer is reporting, it can be used as a troubleshooting tool to determine the cause of the discrepancy.

The operator should start by placing the portable at the outlet of the dedicated analyzer. This ensures that the identical fluid passes through both analyzers in the same state.

- If the readings agree, the dedicated analyzer is working.
- If the analyzers disagree, and the portable is reporting the expected moisture content, the dedicated analyzer requires calibration or repair.
- If the readings agree and they do not agree with the expected readings, the portable can be moved to the sample take-off point, bypassing the entire sampling system and analyzer. If the portable reports the expected moisture content, not the moisture content of the dedicated analyzer, the issue is between the sample take-off and the sensor itself.

Should the portable analyzer report a different moisture content than both the dedicated analyzer and the expected moisture content, then coupling this method with the techniques in 4.1 and 4.2 may help.

The operator should choose a moisture analyzer that will best validate the sensor in place. In most cases, an aluminum oxide moisture analyzer provides the greatest flexibility, as it can operate at elevated pressures, report dew point and ppmv, and can measure moisture in the state of the fluid. This technology is lightweight, making it truly portable, and one should choose a brand that allows for the use of interchangeable sensors, should one or more be contaminated or questioned in the field.

The portable analyzer should have a recent calibration and ideally not have been used prior to the validation of this point of measurement.

A portable TDL moisture analyzer can also be used when validating another TDL analyzer, especially if speed of response is an issue.

Regardless of brand or technology, the operator should be proficient in the use of that specific portable analyzer, to remove the operator as a source of uncertainty. If there is not such a comfort level with the portable analyzer, the plant may choose to hire a field service engineer to come in with a portable and validate the readings. The field service engineer would also be able to audit the entire measurement system, and would be best able to identify the source of the discrepancies. (1)

5 The proper mechanism for field validating moisture analyzers

5.1 The perfect portable moisture generator

In the past several decades, analyzer companies have introduced newer measurement technologies into the process marketplace. These address the speed of response and accuracy requirements of their high value applications. The same level of innovation has not yet occurred in calibration and verification techniques, which by their very nature, would be more accurate than the measurement technique they are validating.

The ideal moisture generator could use any background gas as the carrier, one would dial in the moisture content they would want to generate, and within minutes the generator would be able to provide the gas at the desired temperature and pressure. The accuracy would match the accuracy requirements of the analyzer in question.

5.2 In the absence of perfection

This paper promised the proper mechanism for field validating moisture analyzers.

First we discussed the key attributes of several moisture measurement technologies. I have shown that calibration cylinders and moisture generators can be used to validate measurements and laid out the concerns and impediments in their use.

Redundant or multiple sensors offer benefit, but they too do not answer all concerns. Mixing technologies may provide insight as to the reason for a measurement discrepancy.

Use of a portable moisture analyzer, in the absence of a perfect moisture generator, appears to be the best available method for validating a dedicated moisture analyzer in the field. It can validate the moisture content of the process and point out where in the measurement system there may be a discrepancy.

The proper mechanism is that one that performs the validation quickly and with the highest level of confidence. This may utilize any one of the above techniques or several in conjunction.

6 Notes

(1) Portions taken from *Advantages of Using Portable Analyzers to Field-Verify Moisture Analyzers*, N. Sparages, as presented at the American Gas Association (AGA) 2016 Operations Conference, Phoenix, AZ, USA.

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