

NEXT-GENERATION ONLINE TRACE MOISTURE MEASUREMENT

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ABSTRACT

We present the performance of a novel trace moisture measurement technology that bridges the performance gap between traditional thin-film impedance-based moisture sensors and more accurate, fast and expensive moisture analyzers based on Quartz Crystal Microbalance (QCM) and Tunable Diode Laser Absorption Spectroscopy (TDLAS). This real-time trace moisture analyzer provides the speed and accuracy needed for reliable dryer breakthrough detection in the 0 – 10 PPMv H₂O concentration range at an economical price point and minimal operational cost. The HygroSense analyzer includes an online moisture verification and field calibration module that provides excellent accuracy in a wide variety of gases including hydrocarbons, hydrogen, nitrogen and CO₂.

INTRODUCTION

The liquefaction of natural gas in liquified natural gas (LNG) applications requires drying it to sub-ppm moisture levels for safe storage and transportation. This is typically accomplished using a molecular sieve dehydration system (Figure 1) to meet the stringent specifications (<0.1 PPMv) for H₂O concentration in LNG feed gas [1]. As shown in Figure 1, multiple dryer trains are deployed in parallel, allowing for uninterrupted operation while a saturated adsorbent bed can be taken offline for regeneration. Online monitoring of the H₂O concentration in the gas exiting each molecular sieve dryer vessel is necessary to detect moisture breakthrough. The moisture analyzer results can then be used by operations to prevent gas with elevated moisture levels from entering and freezing in

downstream heat exchangers and cryogenic equipment. A small (PPM-level) increase in the H₂O level that is undetected can have disastrous consequences that would require expensive repairs and downtime. Hence, as shown in Figure 1, a redundant set of moisture measurements is used to maximize availability. Speed of response, accuracy and precision are the most critical metrics of this online moisture measurement system. Keeping these measurements within an analyzer budget without sacrificing plant efficiency is vital to success.

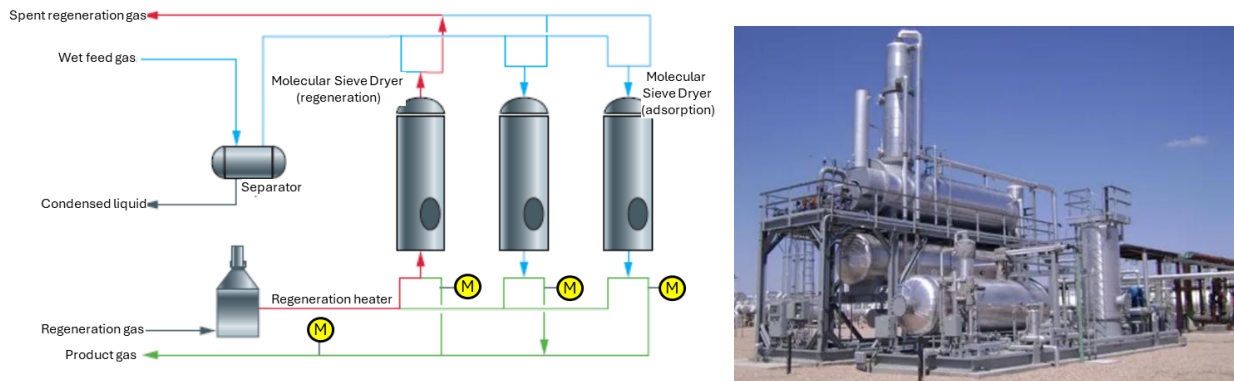


FIGURE 1: MOLECULAR SIEVE DEHYDRATION SYSTEM USED FOR DRYING NATURAL GAS IN LNG PLANTS TO < 0.1 PPMV H₂O – SCHEMATIC SHOWING MOISTURE ANALYZERS (M) (LEFT), ACTUAL INSTALLATION (RIGHT)

To solve this challenging process monitoring problem, an online trace moisture analyzer that can reliably detect sub-ppm moisture upsets in dehydration systems for CH₄, C₂H₆, C₂H₄, C₃H₈, C₃H₆, H₂, CO₂, N₂ and other mixes is required. An ideal solution will have the following specifications:

- Range: 0–10 PPMv
- Accuracy: ±0.05 PPMv or 10% of reading
- Repeatability: ±5% of reading
- Speed of response: <5 minutes for dry down and wet-up

Ideally, the analyzer response and accuracy should be agnostic to significant variations in the feed gas composition, which are driven by reservoir depletion, activation of new wells, or blending from different sources in LNG processing. Any process variations and/or changes in the analyzer response (e.g. drift, temperature dependence, etc.) should be compensated online, thus requiring minimal user intervention and no periodic factory calibration. Immunity to process variation and sensor drift can be accomplished by using an in situ field calibration system that is designed to calibrate and/or verify the sensor response in the process gas at two points – a Zero point that is close to the sensor’s lower detection limit (LDL) and a pre-defined Span point in the 1 – 2 PPMv range. The number of span points required to calibrate the sensor is determined by the non-linearity in its response over the desired 0 – 10 PPMv range of operation. To work as reliably as the factory calibration system, this online calibration/verification system should be capable of repeatably generating Zero and Span points in any process gas with ±5% accuracy.

The design and performance of HygroSense, a novel trace moisture measurement technology that can meet all the above requirements for reliable dryer breakthrough detection in the 0 – 10 PPMv range, is presented. By providing the speed, accuracy and repeatability of more complex moisture analyzers like the QCM and TDLAS in a simpler, lower cost and lower maintenance package, HygroSense enables a more robust and cost-effective deployment of dryer breakthrough monitors at each desiccant tower output. To achieve excellent accuracy and long-term stability in a wide variety of process gases like hydrocarbons, H₂, N₂, O₂ and CO₂, it utilizes an online field calibration and verification module upstream of the sensor probe as shown in Figure 2.

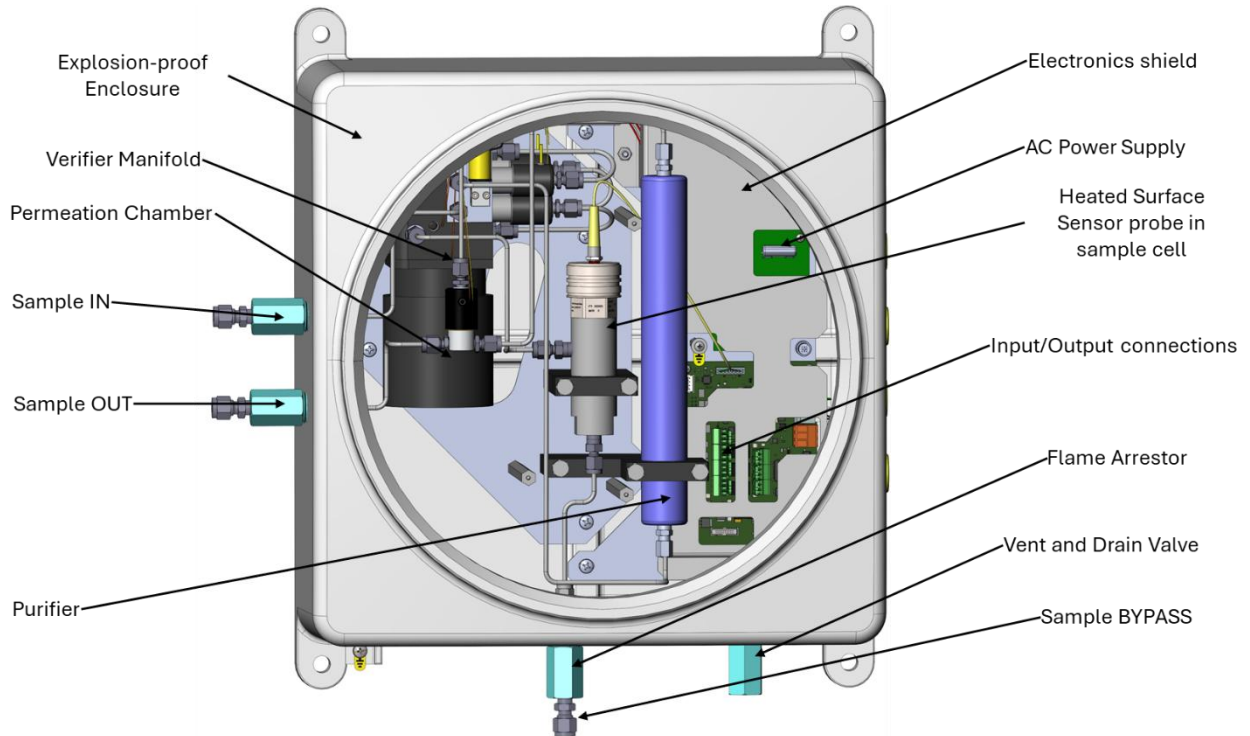


FIGURE 2: HYGROSENSE MOISTURE ANALYZER SYSTEM DESIGN SHOWING MAJOR SUB-SYSTEMS LIKE THE HEATED SURFACE SENSOR PROBE AND THE ONLINE MOISTURE VERIFIER.

DESIGN OF NEW MOISTURE MEASUREMENT SCHEME

Impedance-based thin film Aluminum Oxide (AlO_x) has been the most widely deployed workhorse sensor for numerous industrial applications of online moisture measurement. It is suitable for real-time monitoring of moisture in a wide range of gases and liquids with an industry leading dynamic range of 0.001 – 10000 PPMv, which covers 7 orders of magnitude [2]. Its low cost and simplicity coupled with its unique ability to work at high process pressure (up to 5000 PSI) and availability in both intrinsically safe and explosion-proof packages for hazardous area operation make it ideally suited for rapidly scalable industrial sensing. Since the early 1960's Panametrics has built significant expertise in

developing and deploying this moisture measurement technology in a wide variety of industrial applications globally.

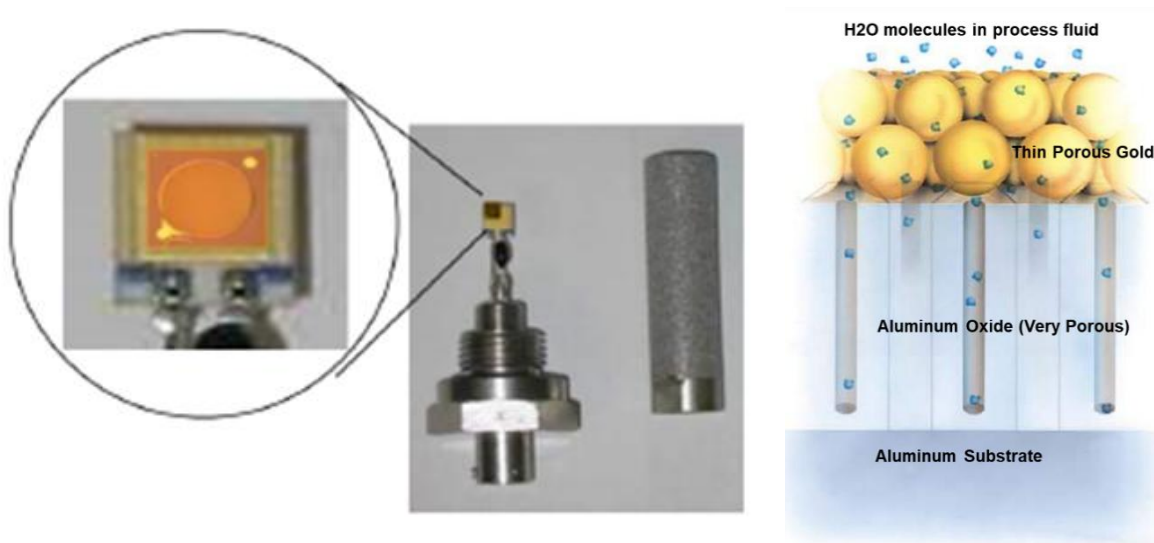


FIGURE 3: THIN FILM ALUMINUM OXIDE MOISTURE SENSORS FABRICATED ON SILICON WAFER SUBSTRATES SHOWING THE TOP ELECTRODE, BACK SIDE CONTACT AND SENSOR SHIELD (LEFT); SENSOR ON ALUMINUM SUBSTRATE SHOWING DIFFUSION OF H₂O MOLECULES THROUGH THE TOP ELECTRODE AND NANOPOROUS HYDROXYLATED DIELECTRIC LAYER (RIGHT)

AlOx sensors measure moisture by detecting changes in the electrical impedance (capacitance and resistance) of a thin film of aluminum oxide as it adsorbs water molecules, hence acting like a variable capacitor/resistor. As shown in Figure 3, when an AlOx probe is exposed to a process fluid, water molecules in the fluid are rapidly transported across nanometer-sized pores in a metal (Au-Cr) electrode deposited on a nanoporous film of aluminum oxide hydroxide (γ -AlOOH). Driven by diffusion, they are adsorbed on pore walls at a decreasing rate until they equilibrate with the bulk of the AlOx film. Hence, the signal generated is proportional to the change in the dielectric constant of the AlOx layer, which in turn is proportional to the vapor pressure of H₂O in any process fluid. The steady-state impedance value at any given H₂O concentration relies on the equilibrium response of the bulk AlOx film, which in a diffusion-driven process could take several hours to reach. Furthermore, this equilibrium response continues to drift over time as the de-hydroxylation of the bulk AlOx film leads to small changes in its nanoporous structure (referred to as pore widening) and its impedance.

Recently, both the drift and speed of response limitation of AlOx sensors have been resolved by developing a novel heated surface sensor (HSS) that continually flip-flops between two preset temperatures. This moisture sensor utilizes the **non-equilibrium surface response** of a thin film of dielectric material to provide accurate, real-time response to moisture upsets. The new sensing method shown schematically in Figure 4 is based on a flash-heated thin-film sensor fabricated on a Silicon die that is attached with eutectic solder to an electrically heated ceramic substrate. The electronic drive circuit

applies a high temperature pulse to “dry” the sensor, i.e., to drive H₂O molecules away from its surface. Instead of measuring the raw impedance of the dielectric film (Hygro Volts), the rate of re-adsorption of H₂O molecules on the surface m (Hygro Slope) is measured while holding the sensor temperature constant. Measuring the rate of change of impedance is essentially equivalent to a high pass filter, which implies DC (zero frequency) drift is observationally absent.

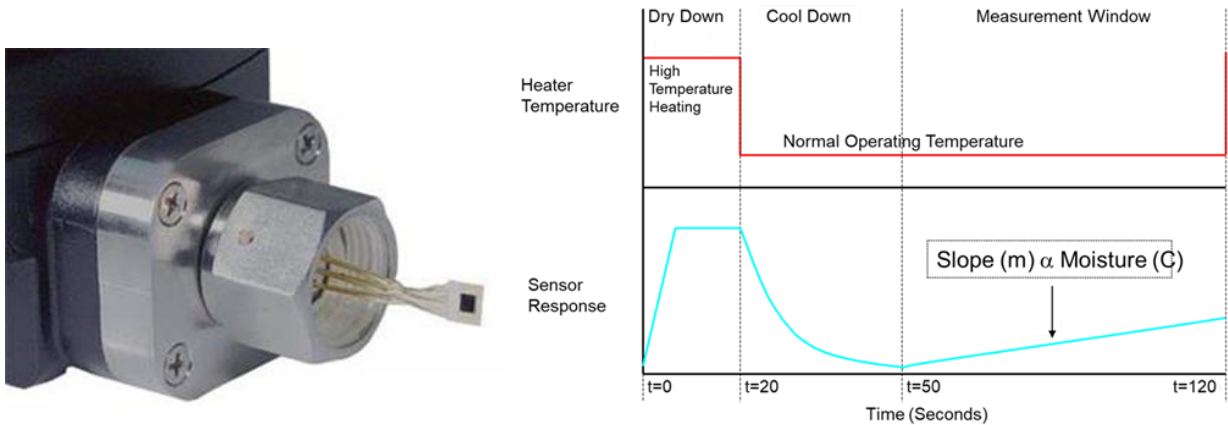


FIGURE 4: NEW HEATED SURFACE SENSOR FABRICATED ON A SILICON DIE BONDED TO A CERAMIC SUBSTRATE (LEFT); NEW MOISTURE MEASUREMENT METHOD THAT CYCLES THE HEATED SURFACE SENSOR BETWEEN TWO PRESET TEMPERATURES AND RECORDS THE HYGRO SLOPE m AS A FUNCTION OF H₂O CONCENTRATION c (RIGHT)

The transient non-equilibrium response of the flip-flop heated surface sensor is shown by the blue curve on the right of Figure 4. A typical measurement cycle is about 2 minutes (120 seconds) long and consists of the following 4 steps that can be individually programmed and are repeated sequentially:

1. Heat time (<5 seconds)
2. Dry down time (~20 seconds)
3. Cool down time (~30 seconds)
4. Measurement window (~70 seconds)

The Hygro Slope is proportional to c , the H₂O vapor concentration (PPMv) in the gas, and being dominated by the surface response, i.e., the rate at which H₂O molecules cross the surface monolayer, the slope changes rapidly with H₂O adsorption/desorption. Thus, every two minutes the H₂O PPMv reading is updated. This implies this sensor is much faster than a conventional thin-film dielectric sensor that relies on bulk response. The HSS concept is based on Langmuir’s assumptions for a uniform surface monolayer response at constant temperature, which assumes less than a monolayer of adsorbed moisture, and is valid ONLY at trace (<10 PPMv) moisture concentration [3]. The average Hygro volt output from a sensor over the measurement window is referred to as the Hygro mean b and is representative of the slower bulk sensor response. Unlike the surface response, it does not saturate until very high levels (>5000 PPMv) of moisture and can

therefore be used to provide an additional lower accuracy output trending beyond the 0 – 10 PPMv HSS calibration range.

PERFORMANCE OF NEW HSS PROBES

Two key metrics of HSS probe performance that have been extensively investigated are its speed of response to sub-PPM moisture upsets and drift over extended time periods. Figure 5 shows on the top a comparison of response time to sub-ppm steps in moisture level in N₂ between the new HygroSense, TDLAS and QCM analyzers. This includes both wet up (top left) and dry down (top right) transients. The refresh rate of HygroSense is 2 minutes, that of the QCM analyzer is 1 minute, and the TDLAS analyzer generates 1 Hz data with a 2-minute moving average.

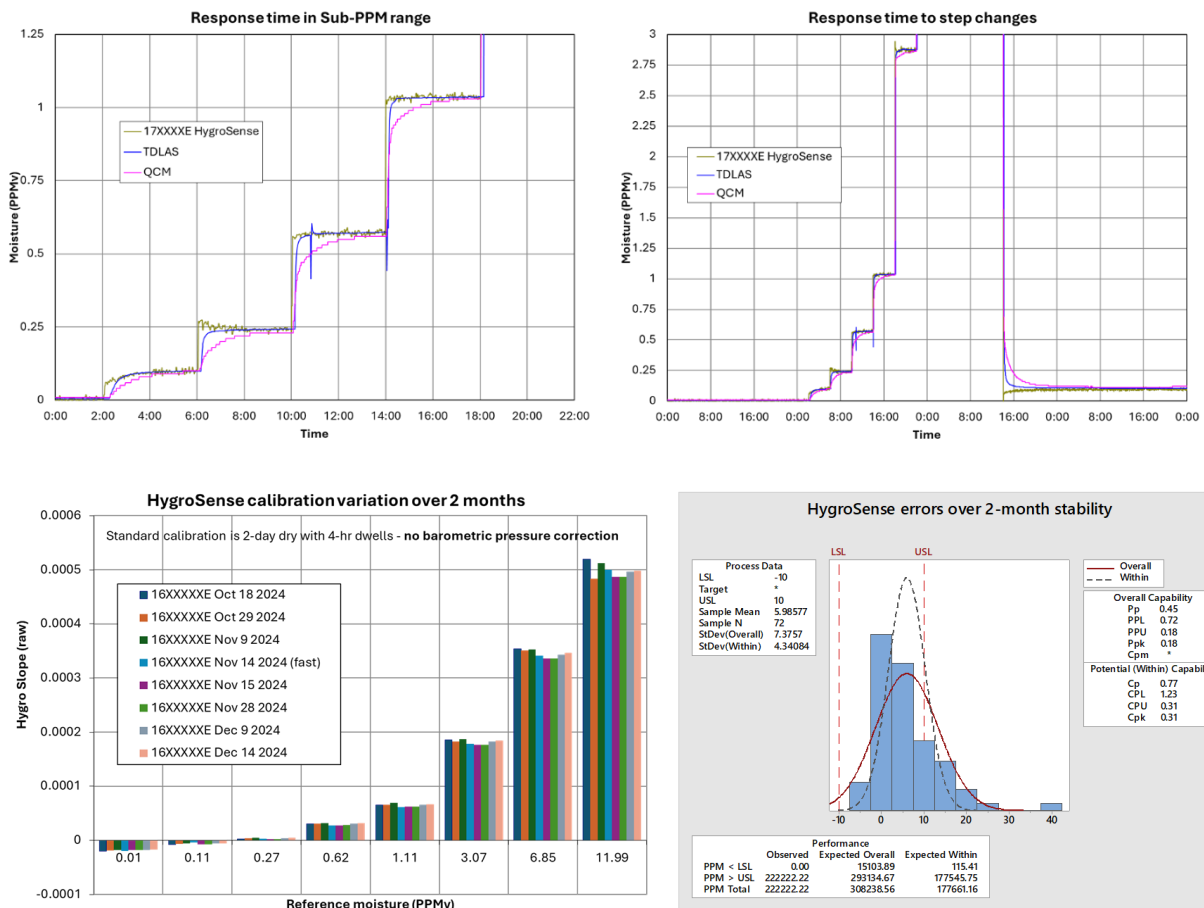


FIGURE 5: SPEED OF RESPONSE COMPARISON BETWEEN THE NEW HSS PROBE, TDLAS AND QCM ANALYZERS TO SUB- PPMV MOISTURE STEPS IN N₂ (TOP); DRIFT BETWEEN SUCCESSIVE MULTI-POINT MOISTURE CALIBRATIONS OVER THE 0 – 12 PPMV RANGE FOR HSS PROBE OVER A TWO-MONTH PERIOD (BOTTOM).

A reliable indicator of accuracy is the drift between successive multi-point moisture calibrations in the 0 – 15 PPMv range as shown on the bottom in Figure 5. The variation in the Hygro slope response of a HSS probe measured in N₂ against a TDLAS reference over 2 months. The raw (unfiltered) slope data are based on a standard 5-day calibration cycle in N₂ with 2 days of dry down and 4 hours of dwell time at each moisture level. The process capability curve shown on the bottom right indicates a mean error of 6% with Std Dev of 4.3%, which is well within the lower/upper accuracy specification limits of $\pm 10\%$. Although the overall drift in 2 months in the HSS response over the 0 – 10 PPMv range is $\sim 3X$ lower than that in the standard AlOx response, the slope response will take 3 – 4 months of repeated calibration to stabilize. This is due to the fact that the Hygro slope (surface response) depends on the value of the Hygro mean (bulk response), and the first derivative high-pass filter is not perfect. It should be noted that storage conditions between successive calibrations were deliberately changed (i.e., sensors moved from desiccated storage to room air multiple times) to identify the impact of any residual hysteresis on drift and accuracy.

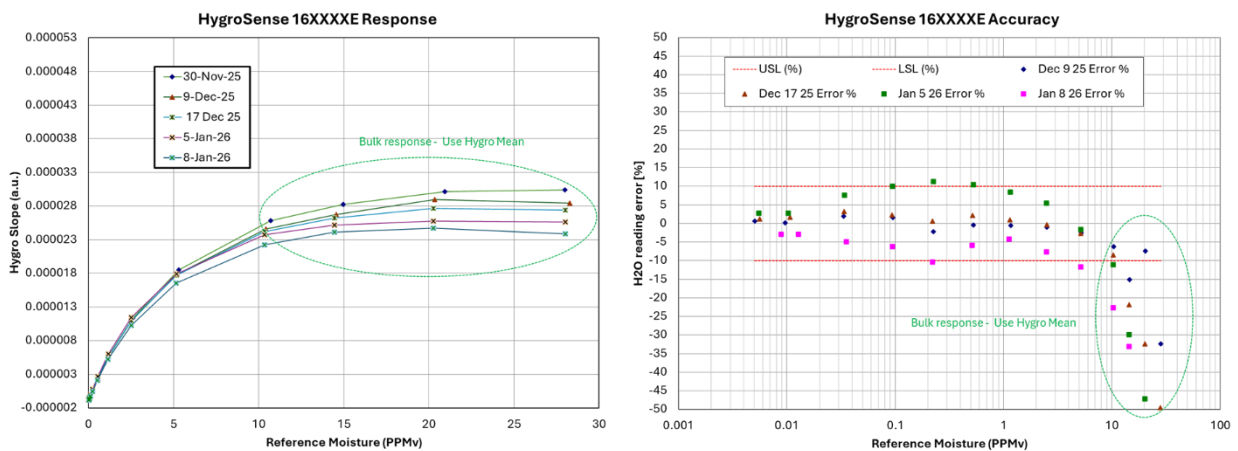


FIGURE 6: ACCURACY OF HSS SENSOR AFTER STABILIZATION – SLOPE RESPONSE OVER 0 – 25 PPMV RANGE (LEFT); ERROR WITH RESPECT TO TDLAS REFERENCE (RIGHT) SHOWING DECREASED ACCURACY DUE TO SATURATION IN SLOPE RESPONSE ABOVE 10 PPMV

Figure 6 shows the calculated accuracy on a stabilized HygroSense probe in N₂ during multiple 4-day validation cycles at 14 Dew Point settings. The primary reference is the low-end Dew Point Generator and a TDLAS analyzer serves as the secondary reference. The first 2 validations were performed 10 and 18 days after original calibration of Nov 30, 2025, and show the best accuracy. For all 4 validations, the accuracy is better than $\pm 10\%$ of reading over 0–10 PPMv range, while it starts to degrade beyond this range as the slope response saturates. The last 2 validation runs were done after extended dry down periods during probe repeatability measurement; hence they exhibit the highest effect of the drifting bulk response.

Figure 7 shows the results of testing short-term repeatability of calibrated HSS probe response in the 1 – 10 PPMv range. Results of repeated cycling of the moisture level at

1, 2 and 4 PPMv levels (as indicated by TDLAS reference) show that both these probes are repeatable within $\pm 5\%$ of their reading. This is determined by calculating the standard deviation of 40-minute average readings from 7 replicate measurements at each level, which works out to 4.6% @ 1 PPMv, 2.1% @ 2 PPMv and 2.1% @ 4 PPMv. Clear contribution of the bulk response in terms of a long-term drift is evident in these results.

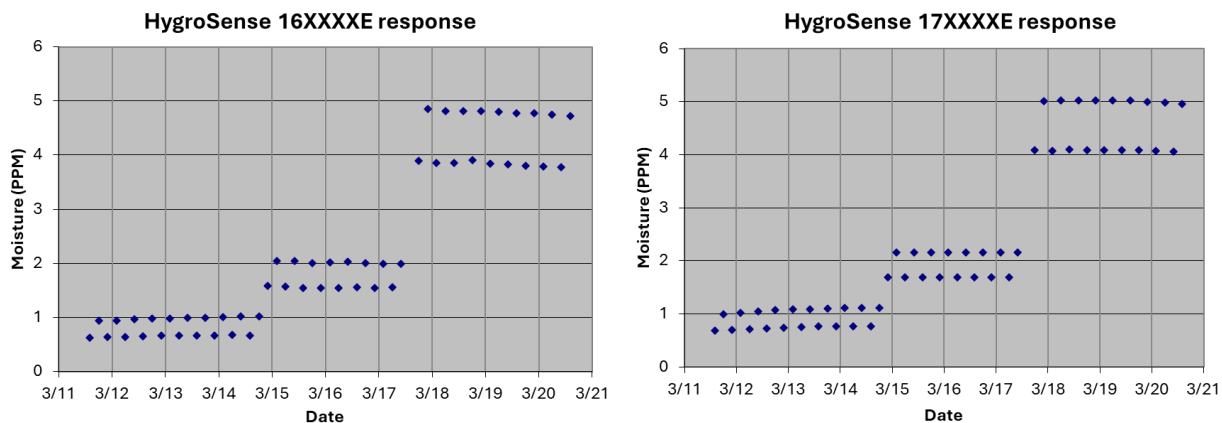


FIGURE 7: REPEATABILITY OF CALIBRATED HSS PROBES AT 1, 2 AND 4 PPMV IN N₂.

Figure 8 shows the repeatability and noise of a calibrated HSS probe in the 10 – 30 PPBv range. Nine consecutive cycles between 10 PPBv and 30 PPBv as measured by a TDLAS analyzer show excellent (<2 PPBv) repeatability and speed of response. The transient response and baseline noise of two calibrated HSS probes with different structural designs are shown on the right of Figure 8. The thin-film sensors on these probes have slightly different structures. The overshoot in sensor response can be tuned as a function of sensor design (probe 17XXXXE has a much lower overshoot than probe 16XXXXE) as well as optimal choice of operating parameters like cycle times (i.e., heat up, dry down, cool down, measurement window) in a given background gas.

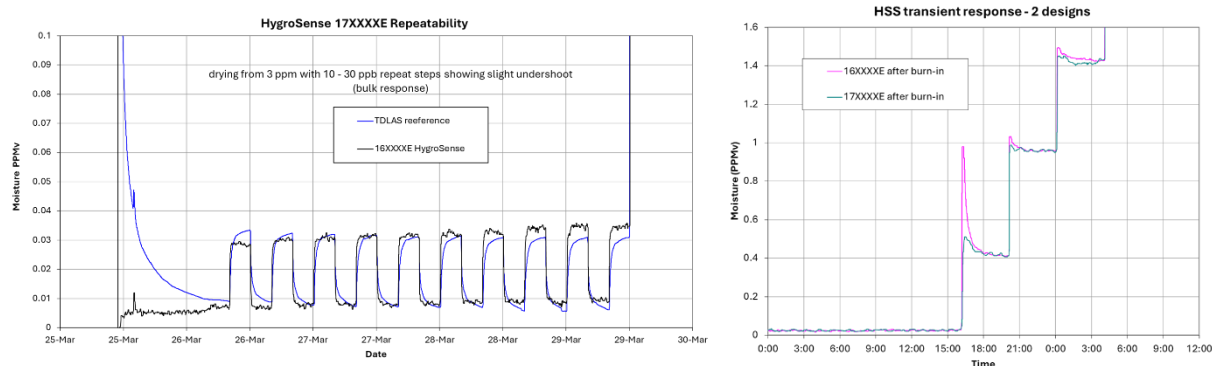


FIGURE 8: REPEATABILITY OF CALIBRATED HSS PROBE IN 10 – 30 PPBV RANGE (LEFT); TYPICAL TRANSIENT RESPONSE SHOWING BASELINE NOISE AND SENSOR-DESIGN DEPENDENT OVERSHOOT (RIGHT).

Two of the best indicators of long-term stability of any HSS probe have been observed are its baseline noise and its upper detection limit (UDL), i.e., the moisture level at which the Hygro slope saturates. As the response of any probe stabilizes over a 4-month burn-in period, its calibration curve, UDL and baseline noise at its LDL become more stable and repeatable. An analysis of the baseline noise distribution of 6 burned in HSS probes calibrated in N₂ over 10 dew point levels from -100°C to -50°C shows that all of them have a noise level in the 1% of reading range. The average noise of all sensors over the 0–10 PPMv range is 0.91% of reading which is significantly lower than the maximum acceptable value of 3%.

ONLINE MOISTURE VERIFICATION SYSTEM

Industrial moisture sensing applications like LNG production, petrochemical processing, and metal heat treatment involve a wide range of gas mixes that vary with time. Hence, a moisture analyzer whose response and accuracy are agnostic to the background gas composition is highly desirable. To achieve this goal, an online field calibration and verification module has been designed and implemented as a critical component of HygroSense. By compensating for sensor drift and process variation online this new module will eliminate the logistical burden of periodic factory calibration. It will be used during system installation to transfer the factory calibration of the HSS probe from N₂ to the process gas that is specified by the user via the HMI (this could be a mix of N₂, H₂, CO₂, CH₄, C₂H₄, C₂H₆, C₃H₆ and C₃H₈). It will also be used for periodic or on-demand verification of the sensor accuracy on a low duty cycle, i.e., the system will be offline no more than 4 hours each time it is run.

Given the largely linear response of the HSS probe in the 0 – 2 PPMv range, any check and subsequent adjustment of this probe's response can be accomplished by generating a Zero and Span point in the process gas. The online verifier module can deliver a Zero sample (moisture below HSS LDL) and 1 or more Span samples (moisture in 1 – 10 PPMv range) in any process gas with ±5% accuracy, which is as good as the factory calibration. As shown in Figure 9, the online calibrator/verifier consists of a combination of a gas purifier, an orifice-based flow controller and temperature-dependent permeation device. It supplies process gas to the downstream HSS probe mounted in an electropolished sample cell, and can operate in one of 4 modes depending on the settings of the four 3-way solenoid valves as shown in Figure 9:

1. Process mode – default condition wherein only the flow of the process gas into the probe sample cell containing the probe is regulated to its pre-set value
2. Zero mode – sample gas is routed through the purifier which reduces its moisture level to under the system LDL (20 PPBv) of the HSS
3. Span mode – dried sample gas from the purifier is routed through the heated permeation tube containing moisture generator that adds a precise (1 PPMv) amount of moisture to it
4. Process + span mode – sample gas is routed directly through the moisture generator without being dried so the moisture level in it is the nominal level plus the 1 PPMv span (to be used as a “bump test” and not intended for any calibration)

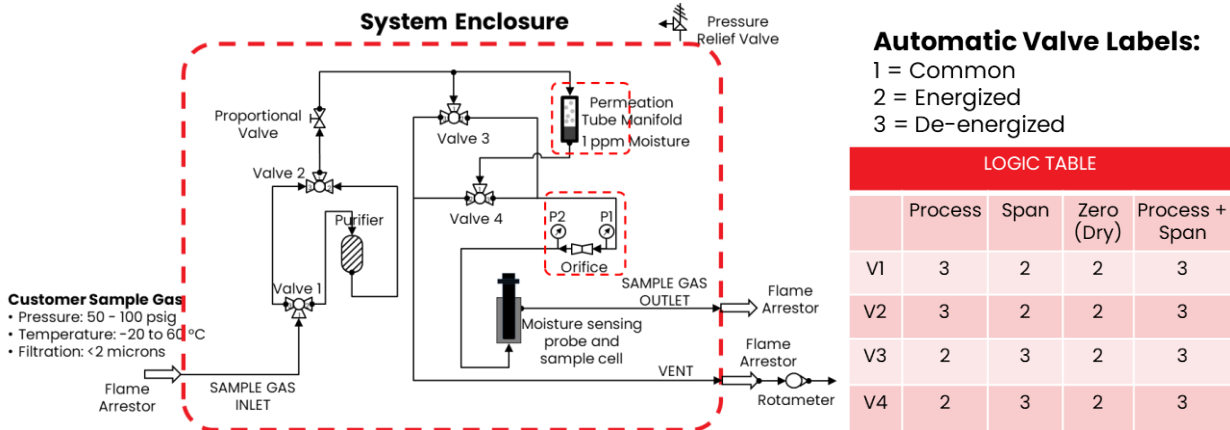


FIGURE 9: PIPING & INSTRUMENTATION DIAGRAM OF HYGROSENSE SHOWING HSS SENSOR MODULE DOWNSTREAM OF THE VERIFIER MODULE (LEFT); TRUTH TABLE FOR 4 VALVE POSITIONS FOR EACH OF THE 4 MODES OF OPERATION OF THE VERIFIER.

The span point value from a constant emission source such as a heated permeation tube shown in Figure 9 is a function of its operating temperature and is inversely proportional to the mass flow rate of process gas through it. Hence, both the permeation tube chamber temperature and the mass flow of process gas through HygroSense have to be tightly controlled. Furthermore, in order to avoid any dead legs where moisture accumulates over time, the flow manifold includes a vent flow path which constantly flows process gas through portions of the manifold that are not delivering sample gas to the HSS probe (note the flow meter shown on the vent gas line is for test purposes only). One of the key ingredients of this automated moisture verification/calibration system is the high precision closed loop flow control based on critical flow through a constant temperature orifice [4]. Both the orifice manifold and the permeation tube chamber shown in Figure 9 are heated to a constant preset temperature above the highest operating temperature specification of 60°C (since there is no cooling built into the system).

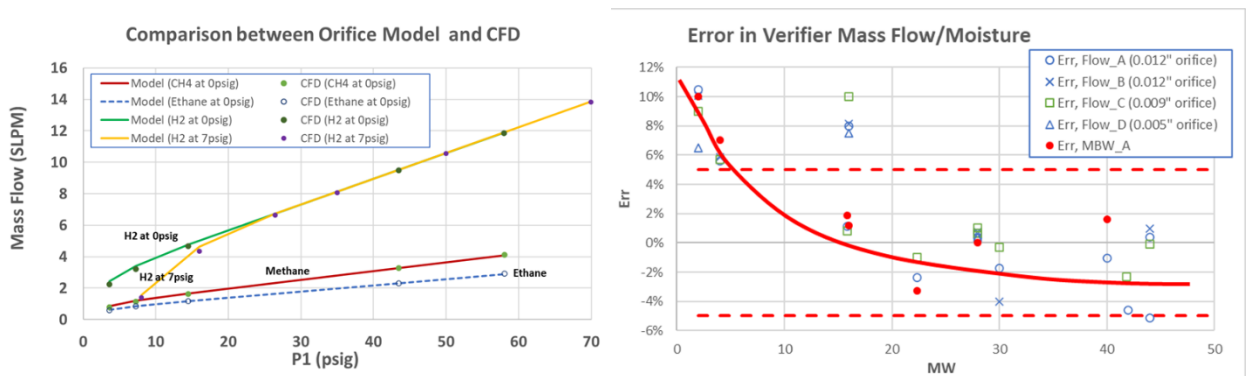


FIGURE 10: COMPARISON OF ACCURACY OF THE ORIFICE MODEL AND A CFD MODEL IN PREDICTING FLOW OF CH₄, C₂H₆ AND H₂ THROUGH AN ORIFICE OVER A RANGE OF VENT PRESSURE P₂ (LEFT); MEASURED ACCURACY OF 1 PPMV

SPAN POINT GENERATED BY THE VERIFIER MODULE OVER A RANGE OF PROCESS GAS MOLECULAR WEIGHT (MW) (RIGHT).

The proportional valve upstream of the two pressure sensors P1 and P2 across the orifice is used to automatically control the flow based on a critical flow model implemented in the control software. This model correlates the measured pressure drop ($P1 - P2$) to the flow of any gas based on its thermodynamic properties. The orifice-based flow control design is significantly less complex, expensive and easier to maintain than a commercially available mass flow controller. Figure 10 shows the performance of the orifice model vis-à-vis a Computational Fluid Dynamics (CFD) model in predicting mass flow of 3 gases – CH_4 , C_2H_6 and H_2 – through a high-precision sapphire orifice over the 0 – 7 PSIG range of manifold outlet pressure P2. In each case, the mass flow was measured using a calibrated off-the-shelf mass flow meter as a function of inlet pressure P1, with the deviation from linear behavior indicating the inlet pressure below which flow is not in the critical regime, i.e., independent of outlet pressure P2.

The gas correction factor (GCF) derived from the critical flow model has been demonstrated to successfully transfer mass flow calibration from N_2 (factory) to CH_4 , C_2H_6 , H_2 , and C_2H_4 over a 0 – 15 PSIG vent pressure (P2) range. The right side of Figure 10 shows the % error (red dots) in a 1 PPMv span point generated by the verifier flow control module equipped with a permeation tube moisture generator measured by a calibrated chilled mirror hygrometer. Two different orifices were used in this test – 0.009” dia orifice for H_2 and 0.012” dia orifice for all other gases. Over a range of process gas molecular weight (MW), the flow errors and span point errors are well within the $\pm 5\%$ accuracy requirement for field calibration of the HSS probe. In some cases (e.g., for CH_4) the GCF derived from the orifice model is more accurate than the correction factor provided by the mass flow meter. The secondary correction of orifice model (red curve) with a MW input derived from the process gas composition and using a smaller orifice (to achieve critical flow) can meet the $\pm 5\%$ accuracy for span points in process gas with high levels of H_2 .

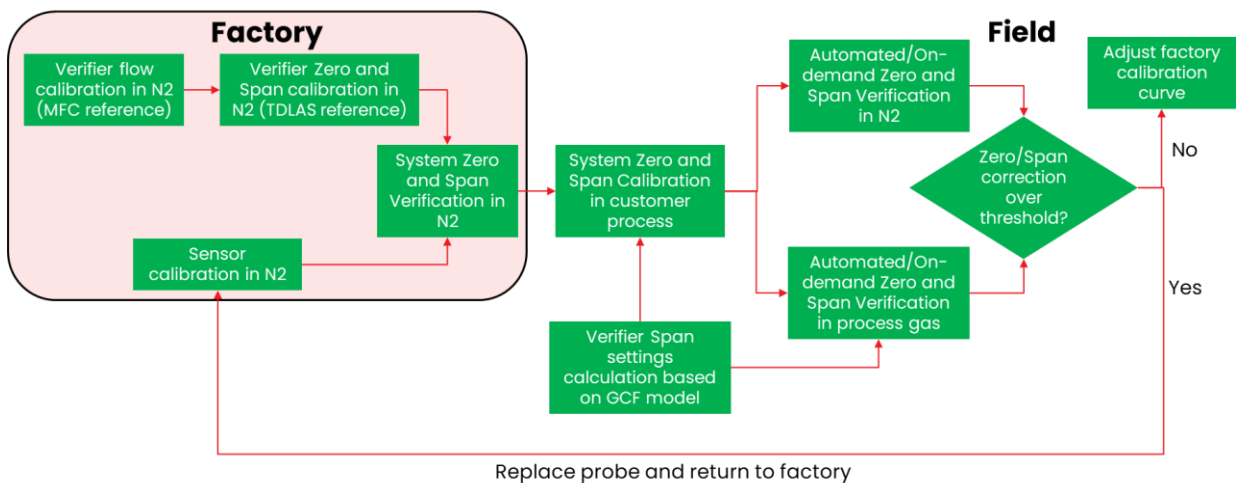


FIGURE 11: SCHEMATIC LOGIC FOR HYGROSENSE FACTORY CALIBRATION, FIELD CALIBRATION AT INSTLLATION, AND AUTOMATED PERIODIC VERIFICATION IN N₂ OR PROCESS GAS.

Figure 11 illustrates the overall logic of how HygroSense achieves the required moisture accuracy specification through a combination of factory calibration in N₂ followed by field calibration in the process gas in which it is installed. Both the HSS probe and the verifier module are independently calibrated in N₂ and validated as an analyzer system in the factory. Upon installation in the field, the user specifies the composition of the process gas, based on which the verifier module calculates the mass flow setting required to generate a 1 PPMv span, runs a Zero + Span calibration cycle, and calculates the HSS calibration curve in the process gas by applying a 2-point (offset and slope) correction to the N₂ factory calibration curve. The system can be corrected for sensor drift and process variation by periodic Zero + Span verification in the process gas. The user can also check and correct its accuracy using the verifier module on demand. There are low and high thresholds defined for both the Zero and Span points in the system that determine when the change in HSS probe response is too high to be corrected in the field, at which point the system prompts the user to return the probe to the factory for calibration. The system monitors the long-term repeatability of the Zero and Span point moisture levels to decide when to warn the end user of the impending end-of-life of the purifier and/or the permeation tubes. For the recommended quarterly verification frequency of the sensor, these components are expected to last for at least 3 years.

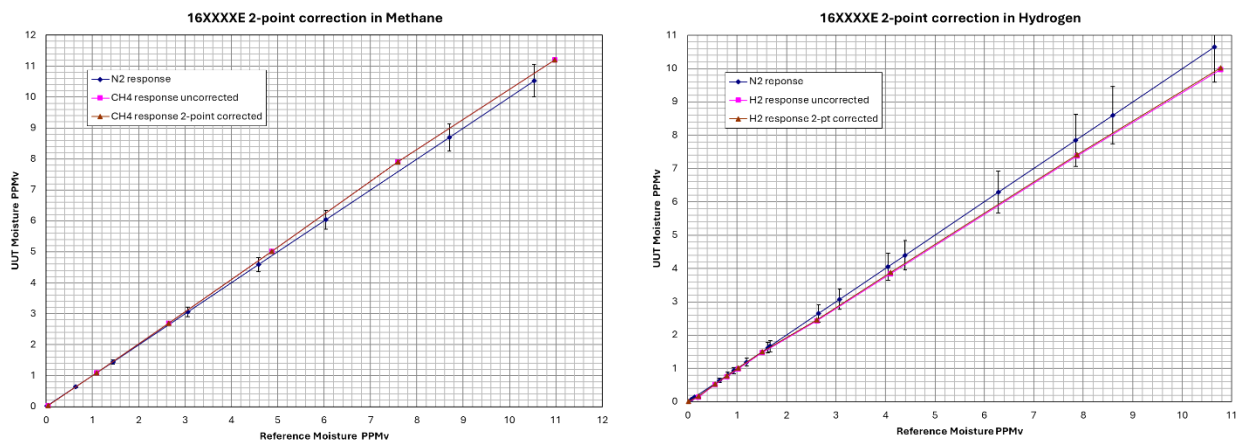


FIGURE 12: RESULTS OF ZERO + SPAN CORRECTION OF THE N₂ CALIBRATION CURVE OF A HSS PROBE IN METHANE (LEFT) AND HYDROGEN (RIGHT).

Figure 12 shows results of applying a 2-point correction to the N₂ calibration curve of a freshly calibrated HSS probe in N₂ over the 0 – 10 PPMv range. By calculating an offset and slope correction in any gas based on the Zero and Span readings of a calibrated HSS probes, the N₂ response can be linearly transformed to the calibrated response in that gas. However, this linear transformation may not be accurate enough to correct any changes in the shape of a nonlinear N₂ response as shown in Figure 6. This limitation in the calculation of a 2-point corrected response in CH₄ and H₂ is evident in Figure 12. In

spite of the small deviation from linearity, the corrected response still falls within the $\pm 10\%$ accuracy specification over the 0 – 10 PPMv range.

CONCLUSION

A new design has been developed for heated surface sensors based on the dielectric thin-film platform that shows >100X improvement in the speed of response to trace moisture upsets vis-à-vis standard AlOx impedance sensors while maintaining $\pm 10\%$ accuracy over the 0.05 – 10 PPMv range. The HSS probe performance shows the repeatability and long-term stability required for sub-PPMv dryer monitoring over the full calibration range in a variety of process gases such as N₂, CH₄, C₂H₆, H₂, etc. The operation of a modular, athermal, leak-free moisture calibrator/verifier module that utilizes critical flow through an orifice and secondary gas correction factors to achieve a 1 PPMv span point with $\pm 5\%$ accuracy has also been demonstrated. Furthermore, the HSS probe has been integrated with the Zero + span verifier module to demonstrate in situ calibration and verification in N₂, CH₄, C₂H₆, H₂, etc. HygroSense is highly cost competitive on both CapEx and OpEx versus other high-precision moisture analyzers based on QCM and TDLAS. This novel in situ moisture monitoring solution is immediately applicable in industrial markets like LNG, petrochemical, and hydrogen quality, where analyzer speed, long-term stability and accuracy are critical to performance.

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