Experimental evaluation of the performance of humidity analyzers in natural gas under industrial conditions

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Abstract

The work reported is the continuation of the extensive comparison performed in controlled laboratory conditions using natural gas from the Spanish grid, as part of the EMRP project “Characterisation of Energy Gases" (ENG01-Gas, 2009). One of the outcomes of the project was the good performance of a laser absorption spectrometer with respect to other measurement technologies, in particular the absence of appreciable drift effects, exhibited by aluminium oxide (Al_2O_3) sensors that are traditionally used in the natural gas industry. The investigation was performed over a wide range of humidity content (13 ppm_v to 250 ppm_v) in ambient conditions of 23 ºC ± 3 ºC, at Enagás central laboratory in Zaragoza, Spain (Gallegos *et al.*, 2015).

We report the subsequent comparison of two laser absorption analyzers, from different manufacturers, and a conventional Al_2O_3 installed on site in real industrial conditions, at an underground gas storage (UGS) and drying plant of Enagás in Serrablo, Spain. The performance of sensors is reported and discussed for all production stages of the plant (extraction, injection and shutdown), during which extreme ambient temperature values took place (from -15 ºC up to 40 ºC) and at line pressures up to 6.7 MPa. A purpose built sampling system was designed and constructed to accommodate the different instrumentation requirements and ensure an optimum metrological analysis.

The new results are compared with those previously obtained under more ideal laboratory conditions in the context assuring compliance with the contractual specifications that are to be met in Europe (EASEE-gas, 2005).

Keywords: hygrometer, natural gas, humidity, comparison, absorption spectroscopy, drying plant.

1. Introduction

The condensation behaviour of natural gas (NG) is one of the most important features to ensure its efficient and safe use. When water molecules are present together with other trace contaminants in the gas stream, namely H_2S and CO_2, these may react resulting in potentially corrosive acids in the pipeline (Carroll, J., 2009). On the other hand, one of the biggest problems in handling high pressure NG with a high water molar fraction is that, when the gas temperature decreases too, it can lead to hydrate formation. This phenomenon must be avoided not just for safety reasons, but also to minimise pumping costs due to load losses in the national supply grids and to avoid drastic decreases in the life expectancy of the pipeline and its components (Sloan, 1998; Ávila, 1999; Mychajliw, 2002; Sloan *et al*., 2011).

There are not only compelling safety or economic efficiency reasons to consider humidity as a key parameter that must be monitored with high accuracy during the most important operations in any gas processing activity (Mokhatab *et al*, 2006). In addition to this there is the need to fulfil contractual obligations to control gas quality in international exchanges. In the case of the water dew point (WDP), the current specifications that have to be met in the transmission of natural gas within Europe have been defined by the European Association for the Streamlining of Energy Exchange - gas (EASEE-gas). According to this regulating body, WDP must be lower than -8 ºC at 7 MPa or at the maximum pressure line (EASEE-gas, 2005).

Therefore, accurate humidity measurement and control is essential in the NG industry in order to maximise the performance of its processes. Metrology can play an important role in providing the necessary support to overcome some of the current challenges of this energy sector. Indeed, the need for research in this field has been identified by some of the most important international bodies in the field of metrology (BIPM, 2007; EURAMET, 2008). The target to offer support to achieve the actual industrial challenges is also outlined in the European roadmap for humidity and moisture measurement (Bell *et al*., 2008). This is further justification for the cooperation between INTA and Enagás, as the Designated Institute (DI) responsible for the Spanish National Humidity Standards and as the technical manager of the Spanish gas system and main transmission system operator of natural gas in Spain, respectively. This collaboration initially was focused in an extensive comparative performance of diverse humidity sensing techniques in natural gas under semi industrial conditions (Gallegos *et al.*, 2015). This activity was part of JRP “Characterisation of Energy Gases" running under the EMRP (ENG01-Gas, 2009). The comparative study of humidity sensing techniques has shown to be of increasing relevance to the NG community, as shown by the work of other researchers, such as Løkken. (Løkken, 2012a).

The work reported in this paper, extends the results obtained in laboratory conditions to those obtained in the field, consistent with the further work suggested at the conclusion of the EMRP project in view of to the good results shown by one of the newest humidity sensing techniques, absorption spectroscopy (Gallegos *et al.*, 2015). Nowadays, this technology is acquiring great importance not only as a laboratory analytical technique, but also at industrial level, including the energy sector. Thus, it is of interest to the gas industry and manufacturers of humidity analyzers to have direct independent information on the behaviour of the technique under real industrial conditions compared to the most commonly used, Al_2O_3 sensors. The main differences between the initial investigation carried out in the EMRP project and the new work described in this paper have been summarized in Table 1.

Table 1 Differences between the comparative study previously performed under semi-industrial conditions (Gallegos *et al.*, 2015) and the study reported here in industrial conditions.

	Semi-industrial conditions*	**Industrial conditions**
Ambient temperature, ºC	23 ± 3	-15 to 40
Relative humidity, %rh	25 to 63	Up to saturation
Pressure line, MPa	0.45 ; 2.8 ; 5.8	3.4 to 6.7
Water vapour fraction, ppm_v	13 to 250 (at 11 levels)	4 to 350
Instruments compared	11	3
Humidity sensing techniques studied	Absorption spectroscopy, polymeric, electrolytic P_2O_5, Al_2O_3 capacitive, condensation, microwave resonator †	Absorption spectroscopy Al_2O_3 capacitive
Properties analyzed	Response time, drift, hysteresis, response to pressure changes.	Relative dynamic performance, drift and influence of contaminants.

*Process variables controlled using humidity generator and heated pressure regulator. † Reported by Gavioso *et al*. (Gavioso *et al*., 2014).

The dehydration method used by the facility chosen uses triethylene glycol (TEG). This system has gained nearly universal acceptance as the most cost effective of the glycols due to many factors as: superior dew point depression, lower operating cost and reliability (Polák, 2009); TEG exhibits ease of regeneration and operation, minimal losses of drying agent during operation, high affinity for water, chemical stability, high hygroscopicity and low vapour pressure at the contact temperature (Anyadiegwu, 2014). For these and many other reasons, TEG dehydration is the most used worldwide compared to other methods such as activated alumina, silica gel or zeolite molecular sieves (Mohamad, 2009).

In this paper we present the results obtained using two humidity sensing technologies in NG and compare their performance in all production phases of the drying plant in an annual production cycle.

2. Experimental set-up

Natural gas dehydration is closely linked to storage of natural gas for two basic reasons: firstly, from a point of view national strategic reserves to reduce dependency on NG supply; and secondly, to deal with increases in energy demand in short-term peaks, and during winter periods to satisfy heating demand. The most advantageous option for storing large volumes of gas are the underground gas storages (UGS) (Netušil, 2012). In this particular case, the industrial facility chosen to carry out this work was the Enagás UGS of Serrablo (Spain), close to the Pyrenees, which is equipped with a TEG NG dehydration plant with a daily treatment capacity up to 7.5 $NMm^3 \cdot d^{-1}$. It was the first gas field reconverted to UGS in Spain at the end of its exploitation in 1989, so the ground properties were ideal for reuse as a strategic storage. Today, there are six productive wells connected to two independent reservoirs with a total volume of 1.1 NGm^3. (ENAGÁS, 2010).

The pressure inside the storages increases as the gas is being injected, up to values close to 19 MPa, however the usual pressure values inside of the main lines of the national distribution grid are around to 7 MPa. Thus, this kind of facility must be also associated with a pumping system to inject the gas into the wells, and an expansion system when the gas is extracted. Another reason for which this facility was chosen, was that it receives NG from regasification plants of liquefied natural gas (LNG) and from international exchanges via the national grid. Thus, the water content of NG must be very closely controlled at these points to be able to reduce the humidity locally in the case of a problem being detected at any point of the grid.

2.1. Description of measurement instruments

As it has been already mentioned, in this work we compare the performance of instruments using two humidity sensing techniques: Al_2O_3 capacitive transmitters and absorption spectroscopy analyzers. The first is used globally in the natural gas industry, and the latter is an extension of a promising technology used in countless sensing applications in other gases.

The Al_2O_3 capacitive sensor evaluated was a Panametrics-MIS II, manufactured and sold by General Electric (GE), already installed in the plant and used by Enagás after the last drying stage before delivery of NG to the national grid when the facility is operating in extraction mode. This sensor has a heated sampling box that includes the simple glass bead filter. The sensor has a built-in pressure sensor to express the

humidity in WDP directly. Enagás' logging system records one value every hour throughout the year. Another instrument, provided by GE, was an Aurora spectroscopic analyzer, of the same model as an instrument whose performance had already been evaluated successfully in semi-industrial conditions in the previous work (Gallegos *et al.*, 2015). The third instrument was a spectroscopic analyzer, OptiPEAK TDL600, manufactured and provided by Michell Instruments Ltd. When we started the study, this sensor was still in a pre-commercial phase, and the tests were important to the manufacturer to check the performance of its instrument under real industrial conditions after the in-house tests performed at their premises. Currently, this analyzer is already being commercialized with an improved version with respect to the instrument evaluated.

Both spectroscopic analyzers were supplied ready for on-site outdoor use, regardless of weather conditions. The geographical location of the Serrablo facility enabled the instruments to be subjected to extreme weather conditions during the tests reported (see Table 1). Each analyzer had its own integral sampling system, designed by each manufacturer and located in the same enclosure as the laser cell.

The sampling systems of the three instruments have small differences, but follow the same overall scheme: isolating valves in all input and output lines, filtering system, pressure reducing system, flowmeters positioned downstream of the measuring cell and a heating system to avoid water condensation effects in any point. All analyzers had the applicable ATEX certification for use in hazardous areas. Table 2 summarizes the principal features of the humidity instruments. Details of the systems can be seen in Fig. 1.

Table 2 Summary of the most important properties of all humidity instruments evaluated.

	INSTRUMENT 1	**INSTRUMENT 2**	**INSTRUMENT 3**
MANUFACTURER	General Electric	General Electric	Michell Instruments
MODEL	MIS-II	Aurora	OptiPEAK TDL 600
PRINCIPLE OF MEASURING	Al_2O_3 capacitive sensor	Absorption Spectroscopy	Absorption Spectroscopy
RANGE	-65 ºC to 10 ºC	5 to 5000ppm -65 ºC to 2 ºC	5 to 5000ppm -65 ºC to 2 ºC
ACCURACY	± 2 ºC	± 4 ppm_v or 2% of reading ± 2 ºC	± 1-2 ppm_v ± 2 ºC

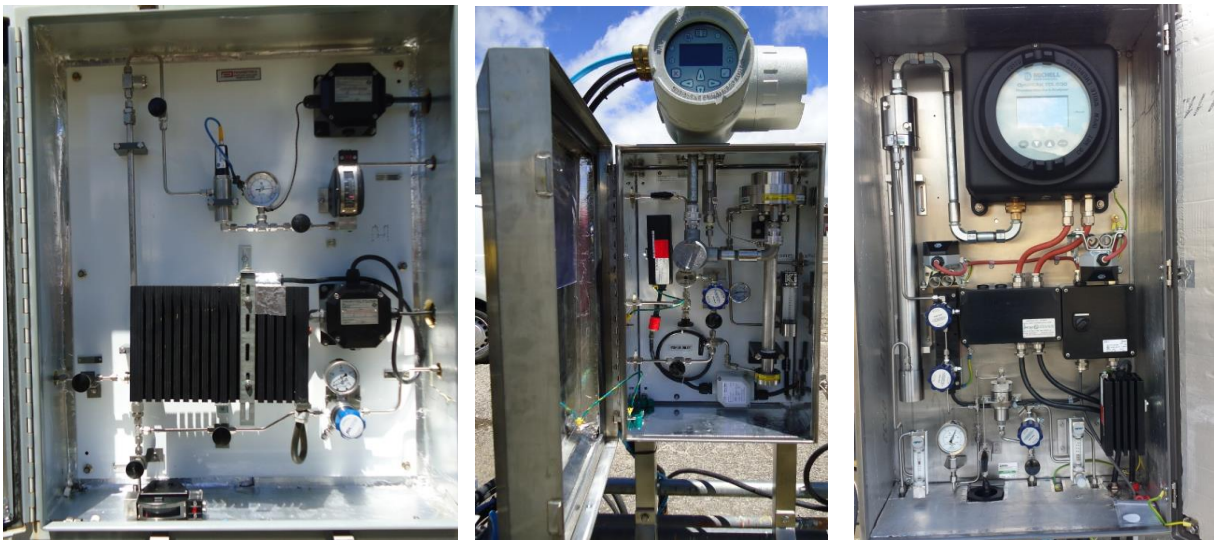


Fig. 1 Pictures OF the three systems evaluated taken in the UGS of Serrablo. From L to R: MIS-II, Aurora and TDL600.

2.2. Description of the experimental setup

The two spectroscopic analyzers were set up in the same position as the MIS-II, owned by Enagás, that is fixed to the wall of the cabin housing the gas chromatograph used to give daily composition of NG. All of these instruments shared the same sampling point through from the main process pipe supplying the national grid. This point was chosen to ensure the maximum reliability and accuracy in the measurements recorded in this study, avoiding condensation phenomena or hydrate formation upstream of the humidity analyzers, consistent with the recommendations of international standard ISO 10715:1997 (ISO, 1997a).

The whole experimental section could be isolated from the rest of plant by means of a valve located just after the sampling point. A three-way valve was added to be used in cleaning or maintenance tasks, for example, purging with dry N_2 to dry this section and remove oxygen prior to filling with NG. All pipe sections, from the isolating valve up to the inlets of all sample boxes of the three instruments analyzed, were heated by means of self-regulating insulated trace-heated lines to keep the sampled gas above the water and hydrocarbon dew-point. The experimental set-up is depicted in Fig. 2 and the photographs can be seen in Fig. 4.

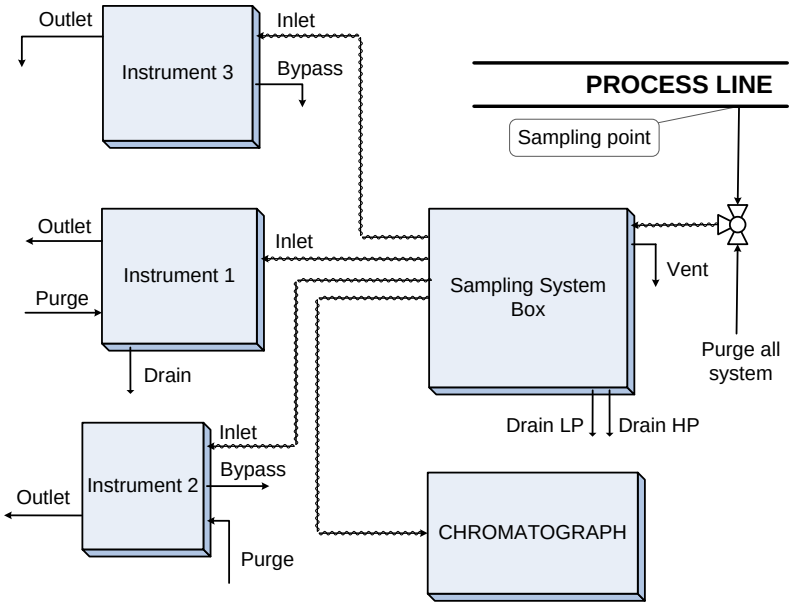


Fig. 2 Block diagram of the whole humidity measurement setup used in this work. Note that all pipe sections upstream of the analyzers are trace heated.

A sampling box was designed and purpose built for the instrument comparison, including supply to the chromatography system, essential to express humidity as water dew point (WDP) in ºC, instead of water content (WC) in ppm_v (ISO, 2005). Although the latter quantity is absolute, it is less used in the NG industry and in this work humidity values have also been expressed in WDP, for which not only the gas composition is required, but also the line pressure.

The new sampling system box was designed to give service to any humidity meter with which it was connected, regardless of the kind of sensing technology used, and taking into consideration the guidelines (ISO, 1997b). This box has the typical sampling scheme with a first filtering stage, followed by a heated pressure regulation system, and finally venting and drainage lines. This system has the peculiarity that there were two pressure levels: high (HP) and low pressure (LP), before and after the heated pressure regulator. This is required because the Aurora and the chromatograph could not measure at the maximum pressure line (around 7 MPa), as the maximum working pressures at the inlets of both instruments are limited to 1.3 MPa and 0.3 MPa, respectively. The output pressure of the LP line was set to 0.14 MPa, and a safety relief valve set to 0.2 MPa, was fitted for added protection.

The schematic diagram of the new sampling system is depicted in Fig. 3. All inputs and outputs had an isolating valve and two pressure gauges were fitted in both sides of the regulator so that any operator can handle the panel safely.

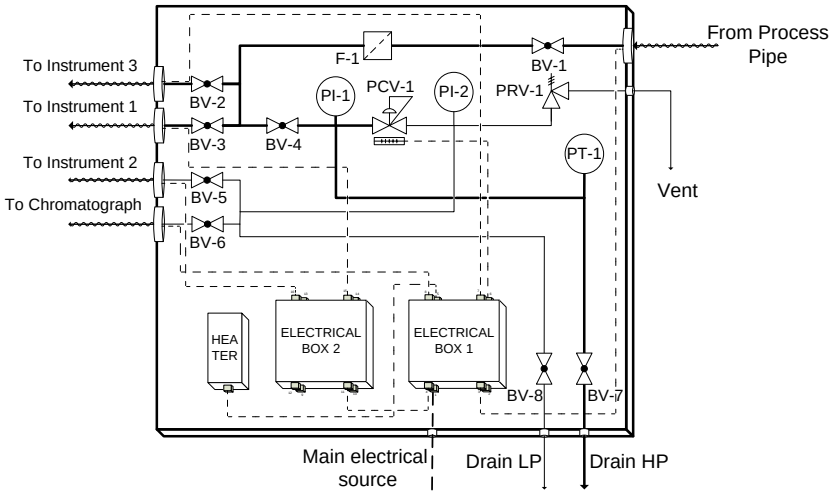


Fig. 3 Pipe and Instrument (P&I) diagram showing the new sampling system box designed and built for these tests. Thick lines denote zones where the NG is at line pressure (HP), whilst the fine lines indicate the areas where the NG had undergone the pressure reduction step (LP).



Fig. 4 View of the whole experimental facility assembled for this project. On the left is the sampling box housing the capacitive sensor. The common sampling system box (with its four trace heated output lines) is positioned between the two spectroscopic analyzers.

2.3. Data logging system

Enagás provided humidity data from its MIS-II together with line pressure and gas temperature data. These values were recorded each hour throughout the whole test period. However, for the new spectroscopic instruments set up in Serrablo a new recording system was implemented. Digital and analogue signals from both spectroscopic analyzers were measured. Digital outputs were recorded using the specific data recording software supplied by the manufacturers. All software was installed in a laptop located in a control room 100 meters from the hazardous area where the analyzers were installed. Standard MODBUS communication protocol on RS-485, was used because of its effectiveness over long distances and in electrically noisy environments.

The analogue outputs from both spectroscopic instruments were measured using a data acquisition and recording unit, model 34970A, manufactured by Agilent. The 4 mA to 20 mA current outputs for WC and

WDP from the Aurora and WC from TDL600, were transformed to voltage by means of precision film resistors with a low temperature coefficient (1 ppm/°C) and high long-term stability that were installed at the input to the scanner.

INTA staff had remote access at this laptop to overview and download the data files generated daily. The laptop was connected to an uninterruptible power supply (UPS), to protect it from small micro-cuts in the power supply during storms.

2.4. Tests performed

The behaviour of sensors was studied during the three production stages of the plant: extraction, injection and shutdown. During extraction the NG withdrawn from the wells is completely saturated, so it must be dried. The two-step drying process in Serrablo consists of physical separation in large horizontal clarifiers followed by a chemical stage which takes place in a drying tower where the chemical equilibrium is modified, decreasing the WC using TEG. Extraction occurs during the winter season or at specific moments in which the peak energetic demand is high. During the summer months, NG from the national grid is injected into the wells. In this case, the drying section of the plant is not operational because the NG will be saturated inside the wells. Between these two production times, there are periods in which the plant is shut-down and maintenance is performed with no NG flowing through the plant, except for small outputs to the measuring instruments and their vents.

The most critical moment for the UGS it during extraction when the saturated NG from the wells is being dried and condensation effects and hydrate formation need to be avoided in the extreme low temperatures commonly experienced in the winter months.

2.4.1. Online recording of all readings

In this study, we have evaluated the evolution of humidity in NG in these three states from the data collected by the three instruments compared. Besides, the transition states produced when one production stage of the plant is changed to another have also been analyzed. This way, the behaviour of these humidity analyzers against these changes was also checked. It was recorded throughout the production year. In this paper we have shown the most representative cases for the three production stages and the intermediate periods.

2.4.2. Checks of analyzer performance

The initial conditions of the three instruments were different. The MIS-II had been in use using the current manufacturer´s calibration coefficients obtained using N_2 as the carrier gas. The Aurora had been supplied for use in a previous research project (Gallegos *et al.*, 2015) and had been in the field for over a year. The OptiPEAK TDL600, was a pre-commercial prototype calibrated by the manufacturer in methane.

The manufacturers checked their analyzers on site at least twice during the test period and included improvements to hardware and software to ensure reliable measurements. The relative performance of the instruments was evaluated. To evaluate possible drift, a simple test with a standard mixture sample of N_2 and H_2O with a water concentration equal to 24 ppm_v ± 4 ppm_v, was used.

The tests commenced with a critical humidity condition of WDP higher than 15 ºC to simulate a high risk situation in which great amounts of condensate may appear inside the sample lines. Initially the whole experimental section e (Fig. 2) was isolated from the NG supply and was dried by flushing dry N_2 at 5 MPa and performing the intermediate check described in section 3.3.

2.4.3. Evaluation of the influence of methanol on the analyzer readings

Hydrate inhibition is typically implemented when it is not cost effective to install a full dehydration unit, or when an operating dehydration unit cannot obtain the desired dew point depressions. A known hydrate inhibitor is introduced upstream of the location where solids formation is predicted to occur. The inhibitor provides some dehydration, but its primary function is to act as an "antifreeze" agent in suppressing the formation of solid hydrates (Kohl, 1985). Methanol injection, proposed by Hammerschmidt in 1939 (Hammerschmidt, 1939), is one of the options available to control hydrate formation but is not regenerated in the gas processing systems, unlike other choices such as TEG (Kohl, 1985).

However, it has been reported that Al_2O_3 capacitive probes, commonly used by the NG industry, trend to drift downwards in the presence of glycols and methanol, giving humidity readings that are drier than the real values (Løkken, 2012b and 2013). A basic methanol injection test was performed during the Summer injection phase by adding 25 L of methanol to the test section during 45 minutes in order to ascertain whether the new spectroscopic sensors are also influenced by this inhibitor. During the test, the mean NG flowrate was 46 $Nkm^3 \cdot h^{-1}$. This yields an approximate methanol concentration of 7 µmol/mol at the temperature and pressure at the time the test was performed.

3. Results and discussion

In this section, we report the most relevant raw data obtained from the instruments compared during the three production stages. The data processing methodology used to simplify the great data volume analyzed and facilitate the evaluation of the properties pertinent to the objectives of the work is discussed. The criteria applied to the comparison and the influence of correlation between the measurements is presented and discussed.

3.1. Raw data processing

Readings from the three instruments were taken during 250 days, during which several extraction and injection processes occurred with intermediate plant shutdowns. During those months, there were situations in which one or more instruments did not provide correct readings or lost communications caused by: updating of pre-commercial communications firmware, failures or resets in digital communications due to micro-cuts in the power supply, instruments or probes sent for calibration, etc. For that, the study of the relative behaviour of the three instruments simultaneously was not always possible, however enough data was collected to represent all process phases.

The analogue and digital signals of each spectroscopic analyzer were in good agreement throughout the tests (see Fig. 6) and no adverse influence on either could be detected. One feature observed in the data was that the readings of the humidity levels of NG of all instruments, in any daily period, exhibited a cyclic behaviour in the three production states. An example of this can be seen in Fig. 5 which shows 10 days during one of the plant shutdowns. It must be pointed out that the oscillations during injection and extraction are highly attenuated with respect to the values obtained in the shutdown modes, as explained in later sections.

As can be seen in Fig. 5, the daily maxima and minima have approximately the same period, with the normal time constant effects. This shows that absorption and desorption takes place between the matrix of NG and the inner walls of any component of plant in contact with the gas. When the outside temperature rises, especially a couple of hours after noon, all components which constitute the drying plant of Serrablo are heated, so finally this temperature increase will raise the temperature of the NG leading to increased water vapour adsorption and the consequent increase in WC and WDP. By contrast, when the outside temperature falls, especially during the last hours of the night, the NG humidity will be lower for the same reason. Evidences of this effect were also reported by Løkken in laboratory environments in which all hygrometers followed a daily cycle synchronized with the ambient temperature variations during the day and night (Løkken, 2012b).

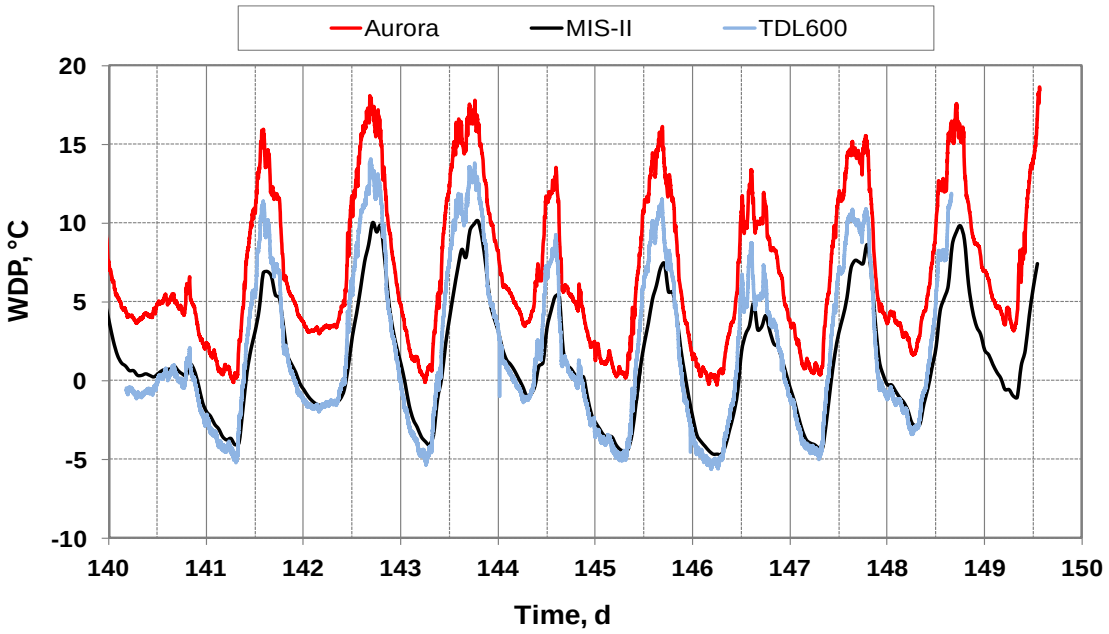


Fig. 5 Representative example of oscillatory readings collected by the three hygrometers during the whole tests time, regardless of the plant process stage. In this particular case, the plant was shut down.

These absorption/desorption cycles were analysed in terms of their amplitude and period by means of a study of daily maxima and minima. A data processing tool was developed to identify such daily points and calculate the time between consecutive maxima (or minima). From this study the dynamic behaviour of the industrial facility as well as the humidity measuring instruments can be known.

3.2. Performance of humidity sensors under stable conditions

Due to the effect of the oscillatory readings previously explained and in order to simplify the comparison of the instrument readings, we evaluated the behaviour of the humidity instruments at specific points of the cycles. The options were to use the points of inflexion (maxima or minima) or the intermediate section between these (rising or falling). The minima were considered the best choice for three reasons: firstly, they coincide with the minimum ambient temperature, when the potential risk of condensation effects appearing is highest; secondly, differences among instrument readings are larger at their minima, so if they reach good correlations in the most adverse conditions, better results are ensured during the rest of day; and finally, the minimum values of WC and WDP are more relevant to the optimal operating range found in the NG industry.

The three instruments were evaluated in two situations: the first, under stable conditions, considering these periods when the humidity levels of NG were relatively stable during several days, which occurred at the end of a production stage of the plant; and the second under transitional conditions that happened when the production mode of the plant was changed.

The choice of analysing the minima without potential problems of "aliasing" of signals by the instruments is corroborated by analysing the evolution of the differences between instruments as a function of time. A horizontal baseline (constant difference) even in a slowly varying signal as found in the "stable" condition is indicative of high correlation between instrument readings and the fact that they are in equilibrium with the measured quantity. This can be seen from the instrument readings during the third injection process depicted in Fig. 6.

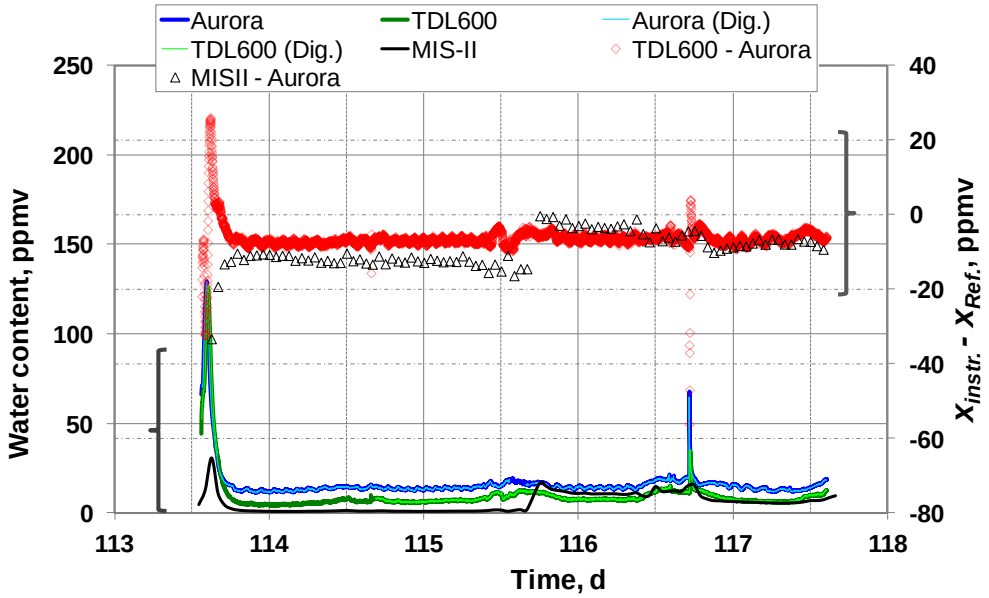


Fig. 6 Instrument readings (bottom) during the third injection process, expressed as WC, and difference (top) between instrument and reference (Aurora), on the left and right ordinate axes, respectively.

The graph shows the whole process over four days, starting with the change-over from a 10-week shut-down to the injection mode. Within approximately five hours the instruments are seen to be stable with respect to each other. Note the MIS-II probe substitution at 4 pm of day 115. The peak around 5 pm the following day, detected by all instruments is due to maintenance on the Aurora.

To compare the instruments and determine their correlation level to each other, the acceptance criterion given by Eq. 1 was used. It is based on calculating the maximum permissible limits from the readings given by the instrument used as reference. After that, if the readings from the instrument under study are within the range given by these limits, both instrument readings are considered to be correlated. The accepted range was calculated from the expanded uncertainty of measurement, obtained from the root square summation of the individual standard uncertainties, multiplied by a coverage factor, k=2, for a confidence level of approximately 95 %.

$$x_{instr} = x_{ref} \pm 2 \cdot \sqrt{u_{instr}^2 + u_{ref}^2 + u_{calc}^2} \qquad \text{(Eq. 1)}$$

where x is the measured quantity (WC or WDP), u, is the standard uncertainty of x, and the subscripts *ref* and *instr* refer to the instrument used as reference or the instrument under study, respectively. Finally, the last term inside the root, u_{calc}, is the standard uncertainty due to the equivalence between WC and WDP and vice versa, calculated according to the model used by GERG (Oellrich and Althaus, 2001) which is summarized from Eq. 2 to Eq. 4. In their work, the uncertainty estimation of WC was expressed in $mg \cdot Nm^{-3}$ units, however in this paper the WC is always referred as ppm_v units. Thus, to apply this unit conversion the original simplified equations were divided by 1.245, which is the conversion factor at standard conditions (101325 Pa and 273.15 K). For WC, this component of the expanded uncertainty (U_{calc}), is given by the following two equations depending on the range:

- If WC is lower than 100 $mg \cdot Nm^{-3}$ (80.3 ppm_v) then:

$$U_{Calc}(ppm_v) = \pm (2.7 + 0.134 \cdot WC) \quad \text{(Eq. 2)}$$

- By contrast, if WC $\geq$ 100 $mg \cdot Nm^{-3}$

$$U_{Calc}(ppm_v) = \pm (14.4 + 0.017 \cdot WC) \quad \text{(Eq. 3)}$$

For the case of the uncertainty calculation of WDP the following was applied:

$$U_{Calc}(°C) = \pm 2.0 \quad \text{(Eq. 4)}$$

The study was performed for both WC and WDP readings given by the humidity analyzers. The instruments used as reference were two: the MIS-II, because it was the only Al_2O_3 capacitive sensor; and the TDL600 because it was the latest instrument added at this work. Table 3 summarizes the acceptance criteria obtained for WC in each process phase. In the case of WDP the acceptance criterion is 3.5 ºC across the range.

Table 3 Summary of acceptance criteria for the comparison of the readings of WC in the production stages reported.

WC, ppm_v	Extraction 1st time	Extraction 2nd time	Extraction 3rd time	Shut down 1st time	Shut down 2nd time	Injection 1st time	Injection 2nd time
$\lvert x_{Aurora} - x_{MIS\text{-}II} \rvert \leq$	9	8	8	9 up to 16	14 up to 18	10	10
$\lvert x_{TDL600} - x_{MIS\text{-}II} \rvert \leq$	---	---	6	7 up to 15	11 up to 18	---	---
$\lvert x_{Aurora} - x_{TDL600} \rvert \leq$	---	---	6	15	11 up to 18	---	---

As mentioned previously, the TDL600 was only compared in the last extraction period as it coincided with its installation. It was not evaluated during the two injection stages as it had been returned to the factory to implement the latest development updates. An example of the results obtained for the three stages, extraction, shutdown and injection is reported in the following sections:

3.2.1. Extraction

The detailed analysis of the last of the three extraction stages is provided as it was the only one in which all three instruments were installed. Fig. 7 depicts all the daily minima of WC with their assigned expanded uncertainty. Table shows the summary of the mean and standard deviation of all readings.

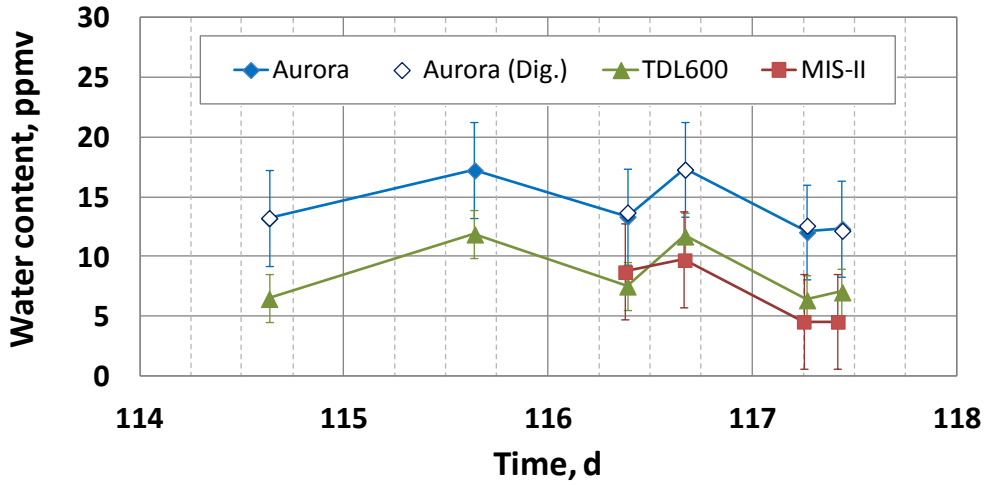


Fig. 7 Daily minima of WC and assigned expanded uncertainty for the three instruments.

As can be seen, the digital and analogue signals of the Aurora are identical. The constant offset between the instruments is indicative of the high level of correlation within the measurement uncertainty. The readings of the MIS-II for the first two days have not been shown as the instrument had been substituted for a spare sensor and only the readings of the new sensor have been reported. Also, the instrument standard deviations shown in Table 4 are almost identical, within the instrument specifications and hence the condition during the period studied can be considered stable. It should be noted that at this point in the tests, the WDP values for the spectroscopic analyzers had been obtained using a simulated line pressure as the pressure transducers had not yet been fitted.

Table 4 Summary of the mean and standard deviations of the readings of WC and WDP of the three instruments in the third extraction stage.

PARAMETER		INSTRUMENT		
		AURORA	TDL600	MIS-II
WATER CONTENT ppm_v	Mean	14.3	8.5	6.9
	Std. Dev	2.4	2.6	2.7
WATER DEW POINT ^oC	Mean	-24.9	-23.9	-25.5
	Std. Dev	1.9	3.0	3.3

3.2.2. Shutdown

The results for the three instruments obtained during the shutdown period in terms of WDP are represented in Fig. 8 and in Table 5 for WC and WDP.

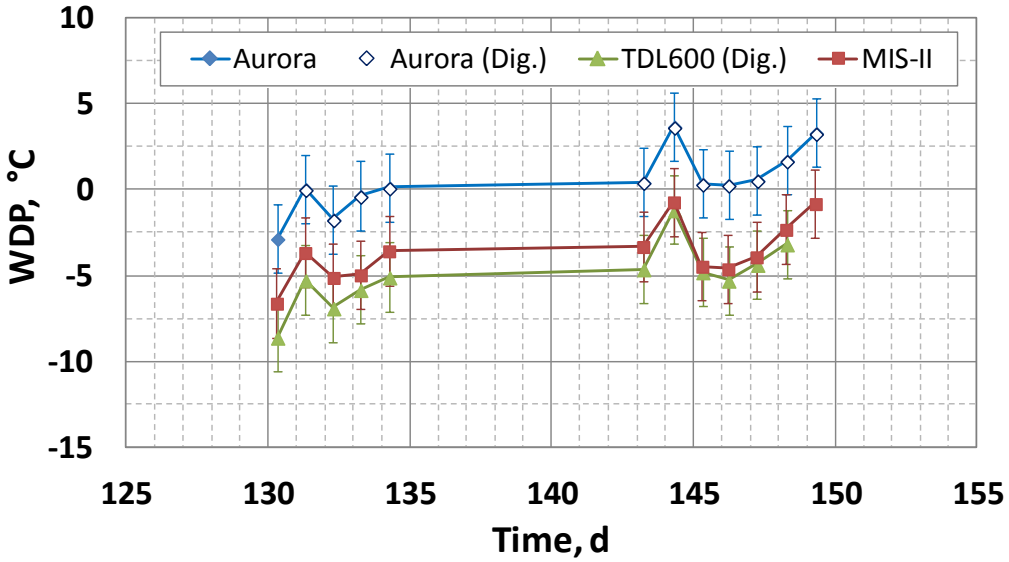


Fig. 8 Daily minima of WDP and assigned uncertainty for shutdown period.

As can be seen the measured WDP for the three instruments exhibit and identical behaviour with a constant offset, and identical standard deviations during the shutdown period. The trend in the series shows a gradual increase in humidity at a rate of approximately 0.3 °C/day. Although the assigned expanded uncertainty of the instruments is larger than the increase, it can be seen clearly that they all are capable of detecting the change in a similar fashion. This is indicative of an improved performance with respect to the manufacturers' specification for WDP above -10 ºC. Table 5 shows the mean and standard deviations obtained for the three instruments.

Table 5 Mean and standard deviation of the readings of WC and WDP for the three instruments during the shutdown period.

PARAMETER		INSTRUMENT		
		AURORA	TDL600	MIS-II
WATER CONTENT ppm_v	Mean	102.9	77.5	61.3
	Std. Dev	13.8	14.6	10.3
WATER DEW POINT ºC	Mean	0.5	-5.0	-3.7
	Std. Dev	1.8	1.9	1.7

3.2.3. Injection

The results obtained during the second injection phase for the MIS-II and Aurora are shown in Fig. 9 in terms of WDP. As before, the instruments have a virtually constant offset, with the MIS-II reading drier. When the WC readings are analyzed, the same behaviour is found with an offset of 13 ppm_v, consistent with the WDP values.

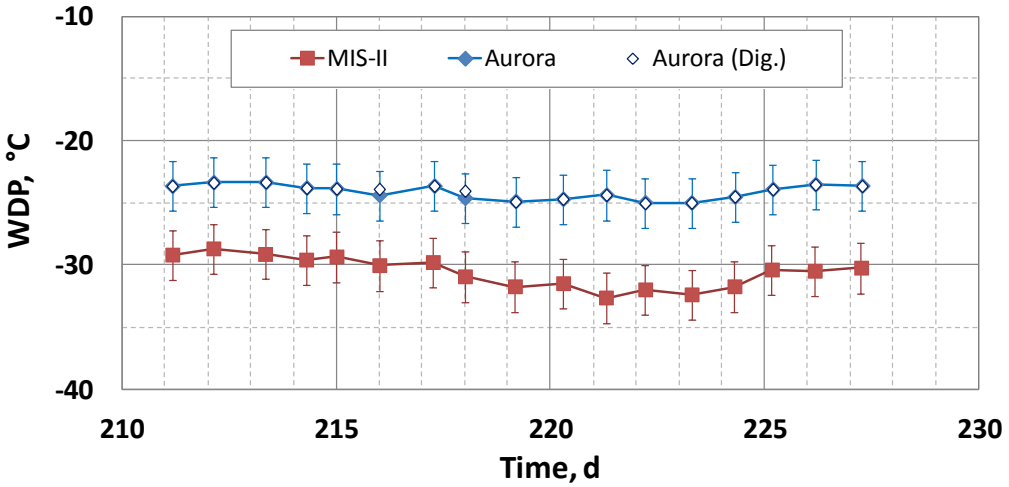


Fig. 9 Daily minima of WDP and assigned uncertainty for the MIS-II (analogue signal) and Aurora (digital and analogue) during the first injection phase.

Table 6 shows the mean and standard deviations of the WDP readings for the MIS-II and Aurora analogue outputs during the second injection phase.

Table 6 Summary of the mean and standard deviations of the WDP readings for the MIS-II and Aurora analogue outputs during the second injection phase.

PARAMETER		INSTRUMENT		
		AURORA	TDL600	MIS-II
WATER CONTENT ppm_v	Mean	15.7	---	3.8
	Std. Dev	0.5	---	0.4
WATER DEW POINT ^oC	Mean	-24.1	---	-30.6
	Std. Dev	0.6	---	1.2

3.2.4. Discussion

The previous sections have illustrated examples of results obtained during the three phases (extraction, shutdown and injection). The summary of the complete results in terms of the percentage of the acceptance criteria for the complete sample is given in Table 7 for WC and WDP.

Table 7 Summary of the comparison of WC and WDP for the three instruments during the extraction, shutdown and injection phases.

	WATER CONTENT, ppm_v			WATER DEW POINT, ºC		
	Mean	Std. Dev.	% accepted values	Mean	Std. Dev.	% accepted values
EXTRACTION						
First time						
$x_{Aurora} - x_{MIS\text{-}II}$	5.6	0.6	100	5.7	0.6	0
Second time						
$x_{Aurora} - x_{MIS\text{-}II}$	3.7	0.8	100	6.0	0.7	0
Third time						
$x_{Aurora} - x_{MIS\text{-}II}$	6.9	1.5	25	0.2	1.5	100
$x_{TDL600} - x_{MIS\text{-}II}$	1.3	1.7	100	1.8	2.3	100
$x_{Aurora} - x_{TDL600}$	5.7	0.5	100	1.1	1.7	100
SHUTDOWN						
First time						
$x_{Aurora} - x_{MIS\text{-}II}$	34.8	5.9	0	18.1	1.2	0
$x_{TDL600} - x_{MIS\text{-}II}$	30.1	7.1	0	16.3	0.7	0
$x_{Aurora} - x_{TDL600}$	5.7	0.4	100	0.9	2.1	0
Second time						
$x_{Aurora} - x_{MIS\text{-}II}$	41.6	4.6	0	4.1	0.5	8
$x_{TDL600} - x_{MIS\text{-}II}$	16.2	5.0	25	-0.9	0.8	100
$x_{Aurora} - x_{TDL600}$	25.4	1.4	0	5.2	0.3	0
INJECTION						
First time						
$x_{Aurora} - x_{MIS\text{-}II}$	13.3	1.2	0	8.0	1.7	0
Second time						
$x_{Aurora} - x_{MIS\text{-}II}$	11.9	0.4	0	6.5	0.8	0

The results obtained are discussed as follows for the three phases.

3.2.4.1. Extraction

During the first and second extraction periods, 100 % of the WC readings of the Aurora and MIS-II meet the established acceptance criterion. However none of the WDP readings meet the criterion.

During the third extraction period, 100 % of the readings of WC and WDP, of the spectroscopic instruments meet the criterion when compared against each other and with respect to the MIS-II, providing a high degree of confidence in the humidity level of the gas supplied to the grid.

The performance of the spectroscopic analyzers at the humidity levels achieved during the extraction phase (4 ppm_v to 20 ppm_v, and -29 ºC to -19 ºC) exhibit an identical behaviour and agreement for 100 % of values. The fact that during the first two periods there is no agreement between the readings of WDP can be attributed to two factors: (a) the line pressure input to the Aurora was a fixed simulated value that differed

from the actual value measured by the MIS-II installed at the full line pressure. This was corroborated by recalculating the data using the MIS-II pressure values. In subsequent measurements this was not a problem as each manufacturer provided pressure sensors and the instruments were configured to use the actual pressure readings. The MIS-II sensor installed at the beginning had undergone excessive drift (to a drier reading); (b) the WC in the third period was approximately double that of the two previous periods that can be easily covered by the acceptance criterion for WC but not in the case of WDP where it implied larger differences that could not be covered by the expanded uncertainty of measurement.

3.2.4.2. Shutdown

During the first period the percentage of acceptable values was very variable, due mainly to the large fluctuations of the readings with correspondingly high standard deviations of the differences between the three instruments.

Before the updating of the firmware of the TDL600, the level of agreement the Aurora was excellent, fulfilling the criterion in 100 % of cases. However, in the second period it only had a very good level of agreement with the MIS-II as it was reading very low both in terms of WC and WDP. Subsequent recalibration by the manufacturer confirmed the large drift of the first MIS-II probe.

3.2.4.3. Injection

During the first period of injection the percentage of acceptable values was very variable, due mainly to the large fluctuations of the readings with correspondingly high standard deviations of the differences between the MIS-II and Aurora, as observed during shutdown in the previous section. None of the values met the acceptance criterion due to the large drift of the initial MIS-II that is reading too dry and the slightly high reading of the Aurora, leading to a large relative difference well above the acceptance criterion during the injection period.

3.2.5. Transitional conditions

Knowledge of the dynamic behaviour of humidity measurement instruments upon resuming operation of the facility after shutdowns of the plant, or vice versa, is essential to detect potential differences such as signal delays or even drift effects. As it has already been commented and, because of the oscillating behaviour of the NG humidity associated with daily thermal cycles, this part of the work was performed by assessing the variation of the cumulative average of the amplitude and period of the daily minima during the time required to achieve a new stable condition, as a function of time (see Fig. 10 and Fig. 11, respectively).

The study the cumulative average of both quantities allows to know the overall behaviour of the instruments during the different periods studied, minimizing individual daily perturbations that any hygrometer can

suffer on any given day during the study. Two representative examples have been chosen: (a) when the plant was extracting and then shutdown, and (b) when it was shutdown and was started to inject gas into the wells.

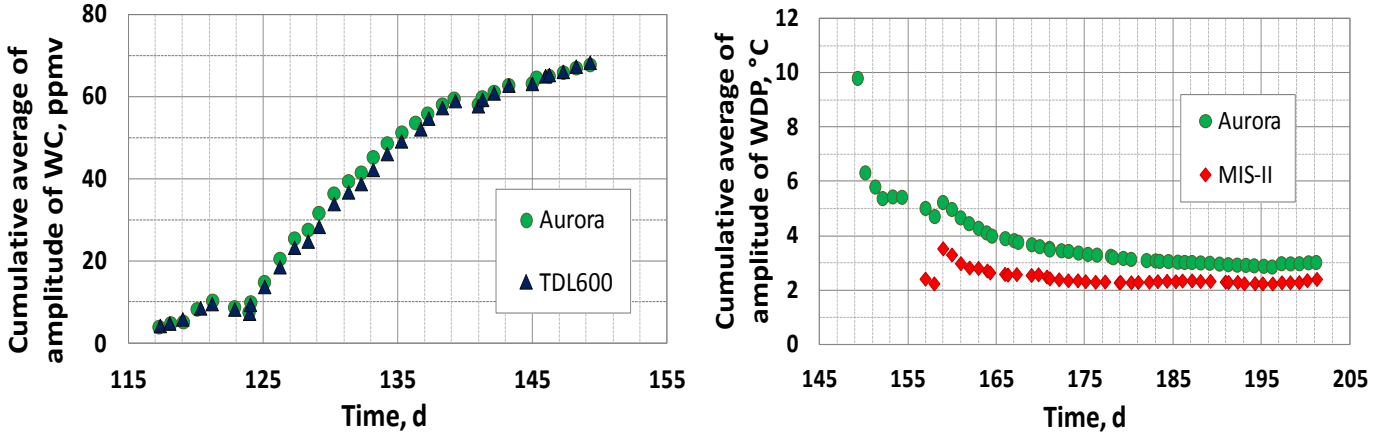


Fig. 10 Cumulative average of the amplitude: Left) from extraction to shut-down for WC. Right) from shutdown to injection for WDP.

The study of the amplitude of the cycles of the analyzer readings shows that the spectroscopic instruments behaved equally both at the start of the change in production mode and when achieving process stability. From Fig. 10 we can see that the Al_2O_3 sensor gives smaller amplitudes in all the periods studied. This can be justified by the fact that the maxima recorded by this sensor were always lower than the other instruments, especially at higher humidity values.

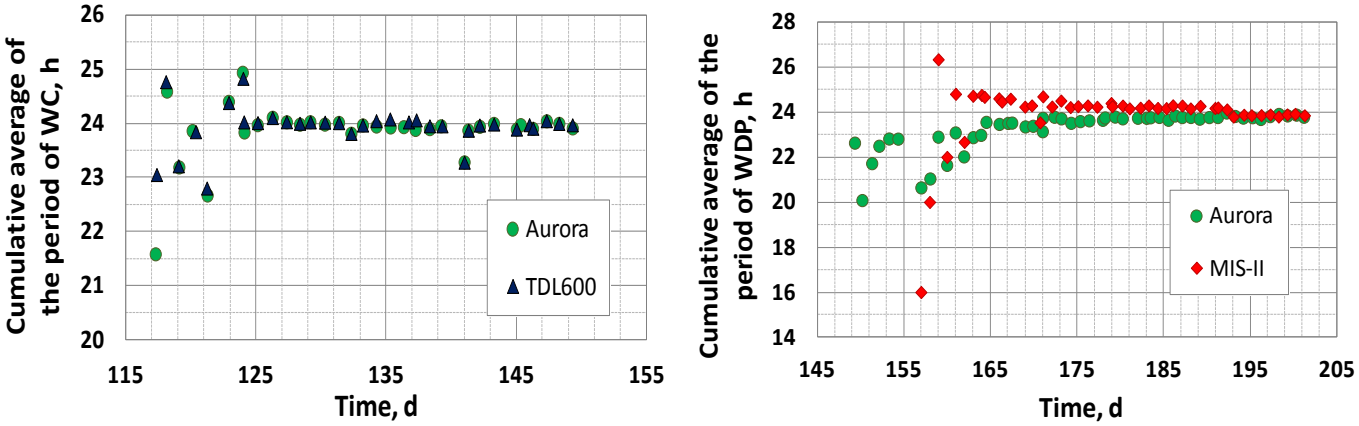


Fig. 11 Cumulative average of the period: Left) from extraction to shut down for WC. Right) from shutdown to injection for WDP.

The results shown in Fig. 11 show that in all cases and regardless of the process change, a 24 hour cyclic behaviour can be observed, coinciding with the day/night thermal cycles and corroborate the capability of both sensing technologies to detect increases and decreases of the gas humidity.

3.3. Intermediate check with standard sample

The process of purging and check performed on the instruments is represented in Fig. 12.

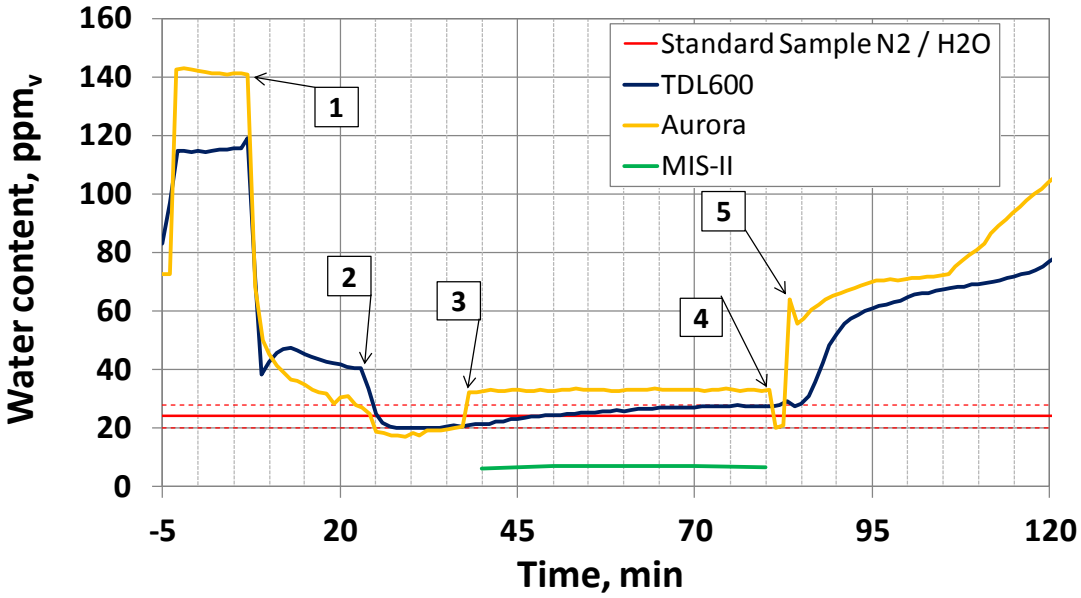


Fig. 12 Readings given by the three instruments studied before and during the intermediate check of their performance, flowing N_2/H_2O with a concentration of 24 ppm_v.

Once the gas humidity started to approximate stable values, the dry N_2 gas cylinder was substituted for one containing a nominal water vapour concentration of 24 ppmv. At first the sample was supplied at a pressure of 1.7 MPa [1], but later was increased to 5.9 MPa, the pressure simulated by the spectroscopic instruments was set in [2]. Then the configuration of the Aurora was changed to read N_2, hence the change observed in [3]. Once the period of stable readings was complete, the measurement was switched to NG [5].

Fig. 13 shows the readings of the three instruments during the measurement of the reference concentration in N_2 (points 3 and 4 of the previous figure). The shaded portion represents the expanded uncertainty of the reference mixture (k=2).

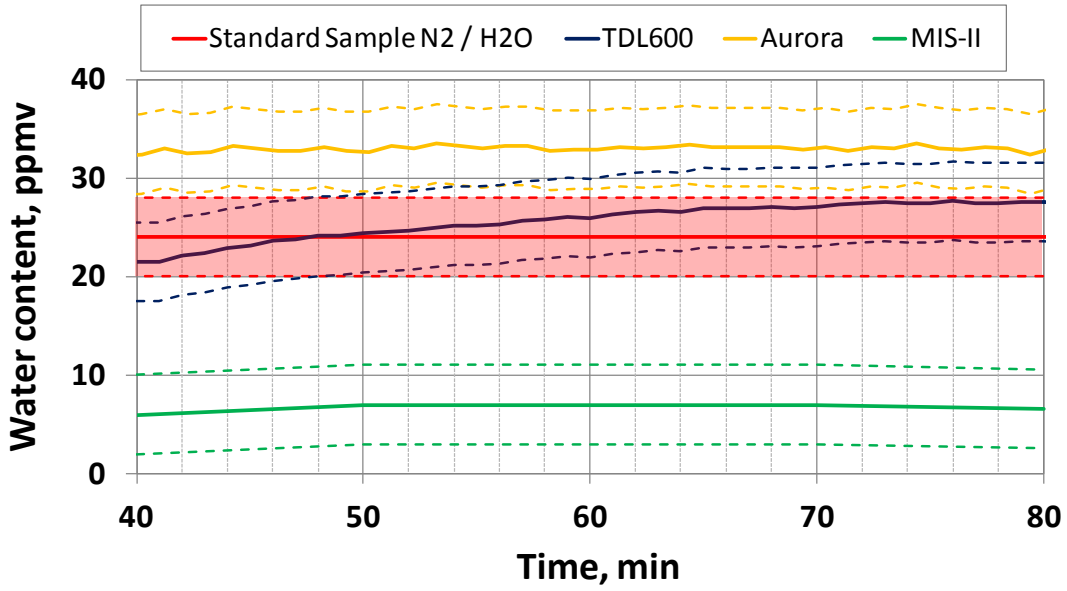


Fig. 13 Readings given by the three instruments studied while the standard sample of N_2/H_2O with a concentration of 24 ppm_v ± 4 ppm_v (shaded region) was applied.

As can be seen, the Aurora and MIS-II instruments already had obtained stable humidity values, whilst the TDL600 took nearly another 30 min to achieve a stable signal. On the one hand we can see that the MIS-II was providing extremely dry values, despite having been recently re-calibrated. On the other hand we can see that the spectroscopic instruments follow each other very closely in relation to the final stable value measured. However, despite needing more time to reach stable values, the TDL600 was the only instrument to meet specifications when compared with the reference gas. It must be noted that the Aurora had not been recalibrated since it was supplied two years before, and the obtained value is consistent with the manufacturer's drift specification for this extended period.

3.4. Long-term stability

The difference between the readings of the MIS-II Al_2O_3 sensor and the Aurora spectroscopic analyzer as a function of time can be seen in Fig. 14 together with the line pressure as indicated by the pressure transducers of both instruments. The Aurora WC measurements in ppm_v were converted to WDP using the equations contained in GERG (Oellrich and Althaus, 2001), based on line pressure measured by the Aurora and the gas composition measured by the chromatograph. The series starts with a transition to injection mode from a shutdown period on day 149 ("INJECTION-first time" in Table 7) and the measurements take approximately 17 days to stabilize that is the normal time for the plant to reach a stable condition after a prolonged shutdown period. This is consistent with the analysis of the cumulative average of the amplitude shown on the right in Fig. 10. On day 196 from the start of the tests, maintenance tasks were performed

upstream of the pipeline in which scrapers were introduced in the line between Serrablo and Zaragoza to remove possible hydrates and this produced an increase in the WDP that was finally detected three days later as the pipeline inner surfaces adsorbed water vapour and finally reached the Serrablo facility. The increased WDP lasted approximately 8 days before the readings stabilized again, ("INJECTION-second time" in Table 7).

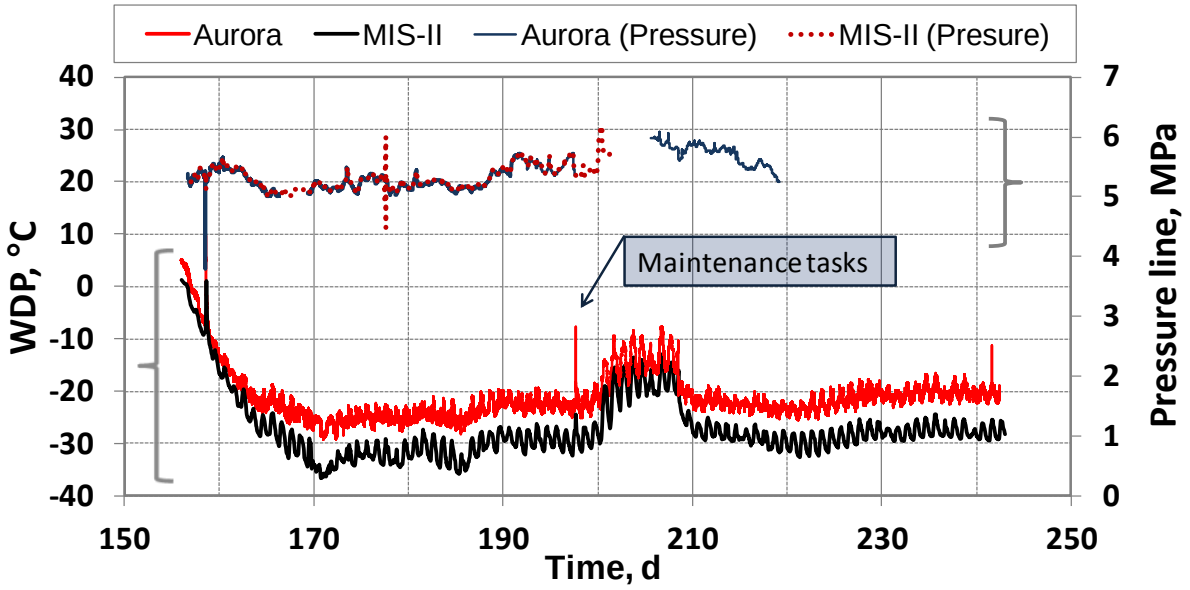


Fig. 14 WDP signals of MIS-II and Aurora as a function of time and line pressure recorded by both instruments.

Fig. 15 shows the results of the difference between the MIS-II and Aurora WDP in °C as a function of time during the same period. The shaded area represents the expected reproducibility of the Al_2O_3 probe (Gallegos *et al.*, 2015). The red triangles are the reading in the first transitional state in which the MIS-II WDP signal converges towards the Aurora readings at a rate of approximately -0.52 °C/day, meeting the expected reproducibility 17 days after the commencement of injection. The yellow rhombi are the readings after the maintenance task and take 8 days to meet the expected reproducibility limit, converging to the Aurora readings at a nominal rate of -0.18 °C/day, much lower than that observed in the first transitional state. The blue squares represent the two stable periods, where, the mean differences between the instrument readings are maintained with a small positive drift over 85 days with a nominal drift rate of 0.14 °C/month, within the manufacturer's specification. However, this is different to what was observed in the tests performed under laboratory conditions on other Al_2O_3 sensors, where in general the drift was to drier readings (Gallegos *et al.*, 2015). If we analyse the data of both the stable periods as one series, the standard deviation of the difference is 0.64 °C. During the first stable region the standard deviation is somewhat higher after the prolonged shutdown with a maximum interval of ± 1.7 °C, whereas in the second stable

period this is reduced to ± 1.1 °C, consistent with the values for the individual instruments given in Table 6 and the expected reproducibility of the Al_2O_3 sensor.

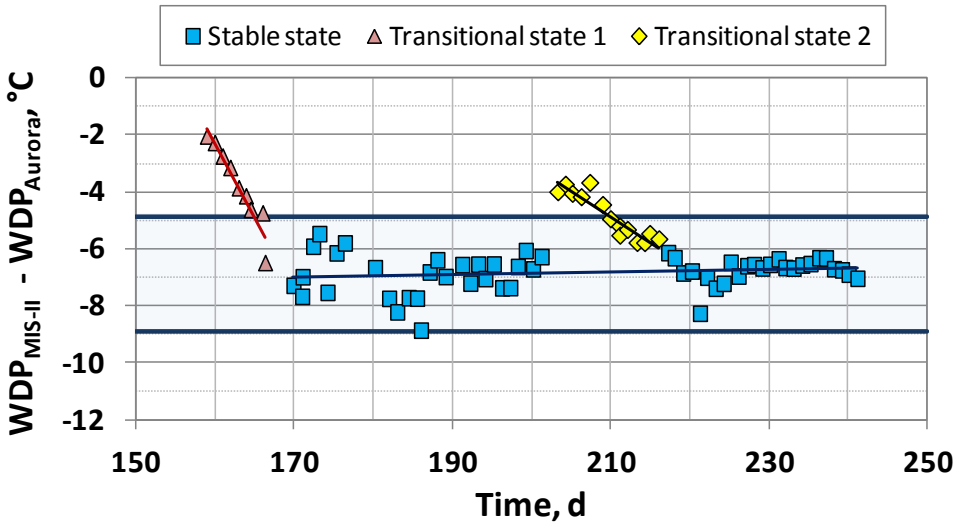


Fig. 15 Long-term stability of difference in WDP of the MIS-II versus Aurora in stable and transition periods. The shaded region represents the expected reproducibility of the MIS-II sensor (± 2 °C).

3.5. Influence of methanol on the analyzer performance

Fig. 16 shows the results obtained on the possible influence of methanol on the humidity indications of the two technologies as described in section 2.4.3. The marker at t = 0 indicates the start of methanol injection.

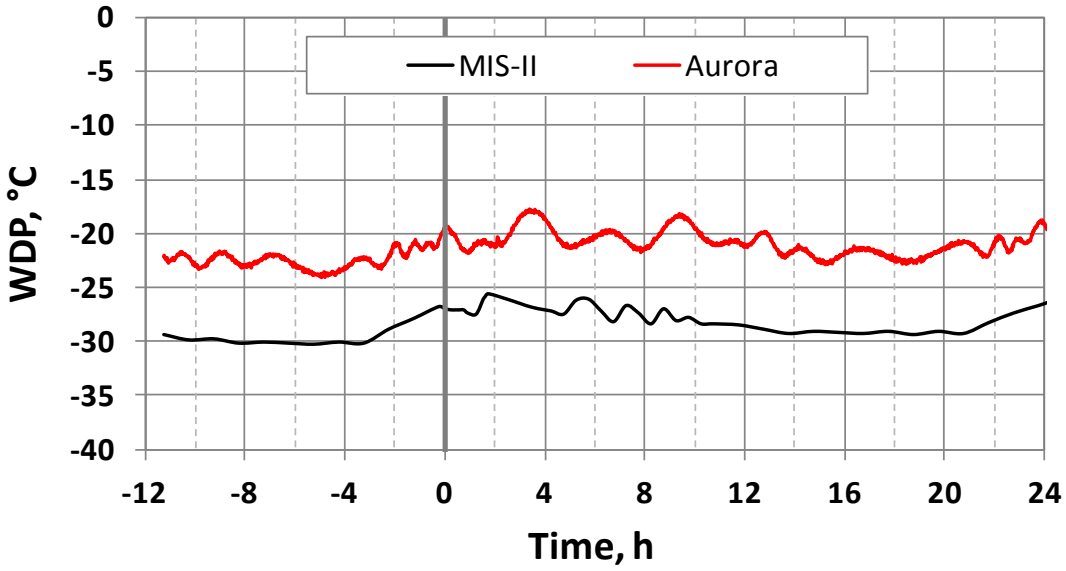


Fig. 16 WDP readings during the test to determine the influence the methanol on the analyzers.

From the results obtained in the previous figure we can interpret that methanol at the concentration applied (7 µmol/mol) does not influence the measurements of the spectroscopic instruments, as claimed by the manufacturers. Similarly, the MIS-II Al_2O_3 sensor was not affected by the presence of methanol.

This value is slightly lower that the studies performed by (Løkken, 2013) where they evaluated more extensively the effects for methanol concentration in the range from 750 µmol/mol to 10 µmol/mol and they detected appreciable changes in only one of the two Al_2O_3 sensors studied.

4. Conclusions

A comparative study of the performance of two spectroscopic humidity analyzers and a capacitive Al_2O_3 humidity transmitter, installed at an Enagás NG UGS facility with associated TEG drying plant was performed using a purpose built sampling system over a period of 250 days. The water vapour fraction range covered the range from 4 ppm_v to 350 ppm_v at line pressures from 3.4 MPa to 6.7 MPa and ambient temperatures from -15 °C to 40 °C. This spans the range of interest to the NG industry in the injection and extraction modes of the UGS facilities plus additional measurements in shutdown mode outside the normal production range but plausible in case of an incident at any point in the national grid. A basic test on the possible influence of methanol on the measurements was also performed at a nominal concentration of 7 µmol/mol without any detectable influence on the two measurement technologies.

During the measurements a cyclic behaviour in the measured WC and WDP, associated to adsorption and desorption effects correlated with the ambient temperature, was observed. The effects of this on the measured humidity were minimized via a differential analysis of the instrument readings at the daily minima in order to reduce the measurement uncertainty of the comparison. Measurements were performed successfully using this method both in stable conditions during extended periods in injection and extraction modes and also in shorter transitional conditions between these and successive shutdowns. The stability acceptance criteria were fixed using the expanded uncertainty obtained from the combination of the standard uncertainty components associated to both measurands, together with a contribution based on the simplified expression of the calculation of the WC used by GERG.

During extraction mode, where the lowest values of WDP were recorded, the degree of correlation between the three instruments was highly satisfactory within their respective specifications. Outside the normal operating range found during shutdown periods, the large drift of the Al_2O_3 sensor produced a difference of up to 18 °C, nearly an order of magnitude higher than expected, whilst the two spectroscopic instruments maintained their excellent level of agreement. In injection mode, after substitution of the Al_2O_3 sensor, it was shown that in the normal operating range of the plant the Al_2O_3 sensors exhibited a good dynamic performance but still could not reach the level of agreement (0 %) with the spectroscopic instruments

expected from their combined specifications and susceptible to excessive drift when used in harsh industrial conditions over extended periods.

The long-term stability of the instruments was controlled using a reference mixture of H_2O/N_2 with a nominal WC of 24 ppm_v ± 4 ppm_v. No appreciable drift of the spectroscopic analyzers was detected. When comparing the readings during the stable periods, the drift of the second capacitive sensor over 85 days was less than +0.4 °C, consistent with the expected reproducibility based on the manufacturer's annual specification of ± 2 °C although in the opposite direction as that observed in previous work in semi-industrial conditions on a number of Al_2O_3 sensors.

The importance of correctly measuring the line pressure in order to calculate the WDP from the WC measurements inherent to the spectroscopic analyzers, rather than using the conventional specified line pressure (simulated value) was highlighted, showing that failure to do so could produce errors of up to 13 ppm_v in normal line pressure fluctuations produced by NG flow variations.

The results reported in this work extend the work performed in NG in semi-industrial conditions at the Enagás R&D facilities in Zaragoza as part of the EMRP JRP ENG01 GAS. The results performed on the absorption spectrometers, one commercially available and the other a in pre-commercial prototype, confirm the adequacy of the spectroscopic analyzers for accurate humidity measurement and control in the demanding industrial conditions essential for the NG industry in order to maximise the performance of its processes.

Acknowledgements

First of all, thanks to the staff of the Enagás Serrablo UGS facility who has allowed us access to its facilities with a great flexibility and confidence, providing us with all the necessary support and means to perform all the tasks carried reported. In particular we are grateful to Alberto Martinez, head of UGS of Serrablo and Pilar Lardies for her supervision and providing us with information of all instruments and promptly reporting any incidents detected.

The authors wish to express their gratitude to Michael Summers (Michell Instruments Ltd.) and Cesar Muñoz (GE Measurement and Control Solutions) for their support in the setting up and operation of their instruments during the project and to Pedro Hernández (INTA) for his assistance in taking the measurements and to Rebeca Larroca (INTA) for developing the necessary data analysis algorithms.

Nomenclature

Abbreviation

DI	Designated Institute
EASEE-gas	European Association for the Streamlining of Energy Exchange - gas
EMRP	European Metrology Research Program
EURAMET	European Association of National Metrology Institutes
GE	General Electric Company
GERG	European Gas Research Group
JRP	Joint Research Project
LNG	Liquefied natural gas
NG	Natural gas
TEG	Triethylene glycol
UGS	Underground gas storage
UPS	Uninterruptible power supply
WC	Water content
WDP	Water dew point

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