

MULTIDETEK2 GC



WITH INTEGRATED MODULE FOR TRACE MOISTURE ANALYSIS

DESIGN REPORT



The Advanced Quartz Crystal Microbalance sensor from Michell Instruments is now integrated inside the MultiDetek2 GC to provide reliable, fast and accurate measurement of trace moisture content in a variety of applications where keeping moisture to a minimum is of critical importance.

The analyzer provides consistently accurate measurements of trace moisture. This consistency is achieved using a self-calibration system, which adjusts the sensor with reference to an internal moisture generator. The moisture generator is supplied with a calibration traceable to NPL and NIST, so long term stability of its measurements is guaranteed.

Having such module inside a GC allows to combine multiple impurities analysis with trace moisture inside the same instrument.

DETECTION PRINCIPLE

Sensor: Quartz crystal microbalance

A quartz crystal is sensitised with a thin film of hygroscopic material.

- ▶ Water molecules are adsorbed into the hygroscopic layer deposited on the surface.
- ▶ The change in mass modifies, in a very precise and repeatable manner, the oscillation frequency.
- ▶ The moisture concentration is measured as a change in the oscillation.
- ▶ The change in the oscillation is evaluated by switching the gas inside the sensor and by measuring the response delta. For sampling response, the sensor is balanced between a dry gas source and the sample source. The difference is then calculated. The same principle is applied for the span gas calibration. However, this time the comparison is between a dry gas and a source of wet gas. A cycling time of 30 seconds on each gas is used to compare the response delta.

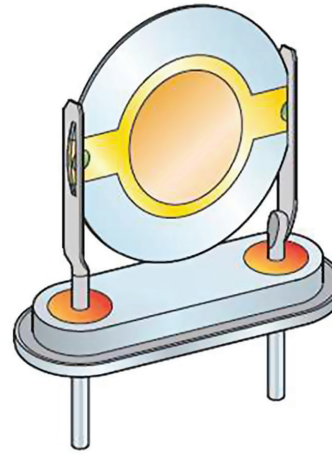
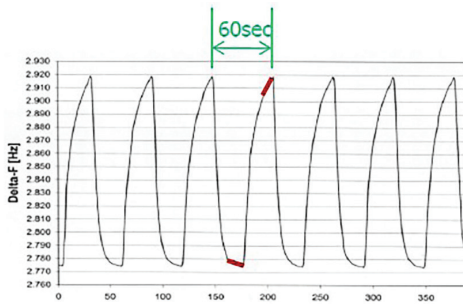


Figure 1:
Quartz crystal sensor

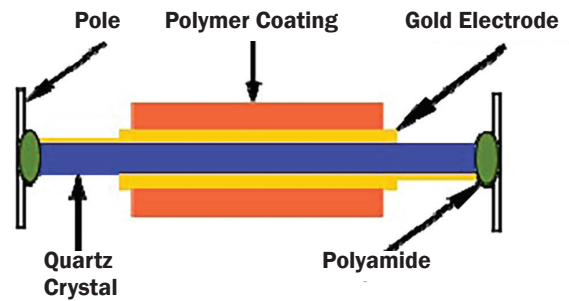


Figure 2: Quartz crystal principle

$$\text{Measurement } \Delta f = \text{primary } \Delta f_{\text{sample}} - \text{primary } \Delta f_{\text{dry}}$$

- 30/30 sec measurement cycle
- Non-equilibrium for fast response
- Self-cancelling effect for contamination

- ▶ The dry gas comes from the carrier gas source of the GC. The sensor is supplied by a known Helium or Argon or Nitrogen grade 5.0 carrier source going through a heated gas purifier model LDP1000 series. This combination generates a gas purity of 8N. By using this technique, the dry gas source contains less than 10ppb H₂O what is ideal as zero gas reference.
- ▶ The wet gas comes from a certified water filled permeation tube heated at a controlled temperature of 45 Celsius. It generates

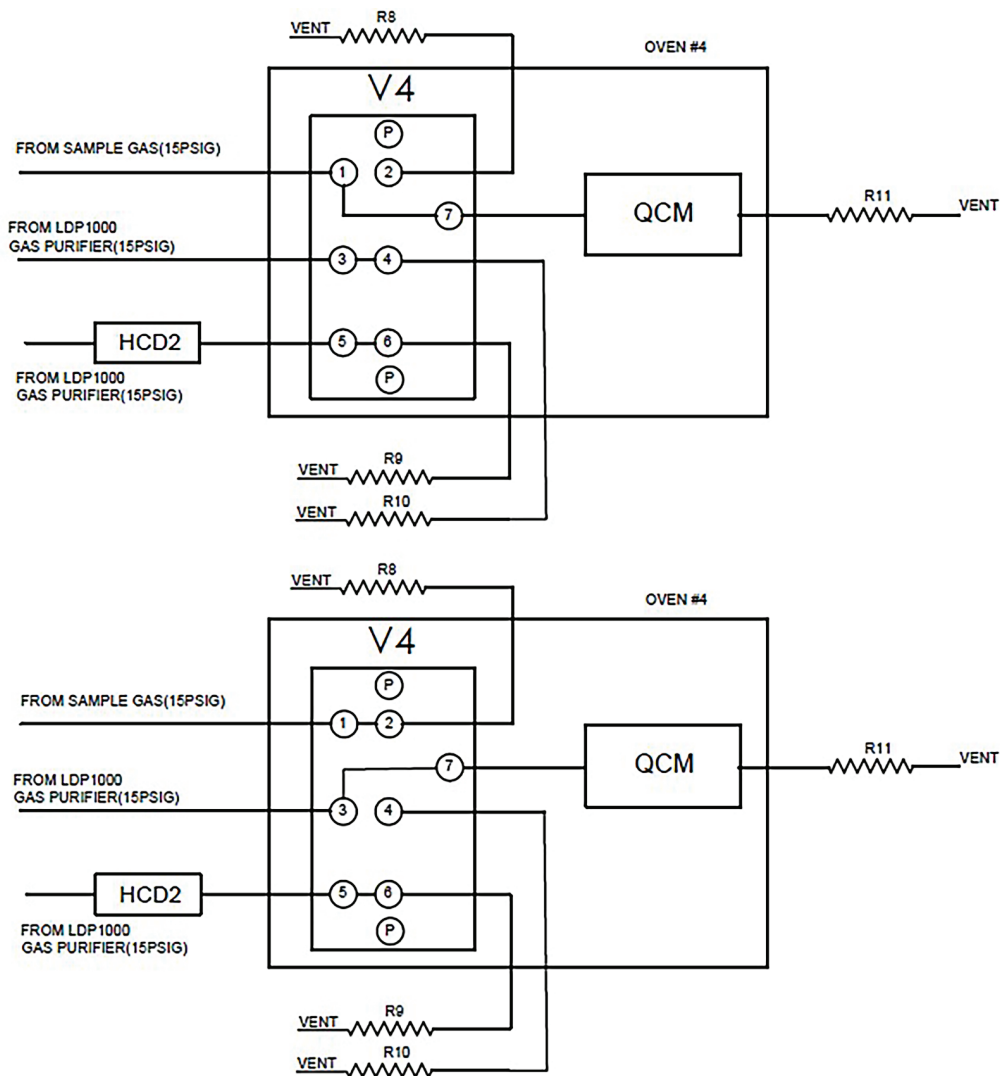
a stable amount of moisture used for span calibration. The moisture generator is made of coated stainless steel to reduce the surface absorption of water molecules and then keep the moisture rate very stable and accurate.

- ▶ The flow inside the module is controlled and maintained by a network of calibrated orifices. All flow passageways upfront the sensor are less than 0.030"ID, all coated with an inert coating to accelerate the response/purge time and improve the performances of the system.

FLOW DIAGRAM

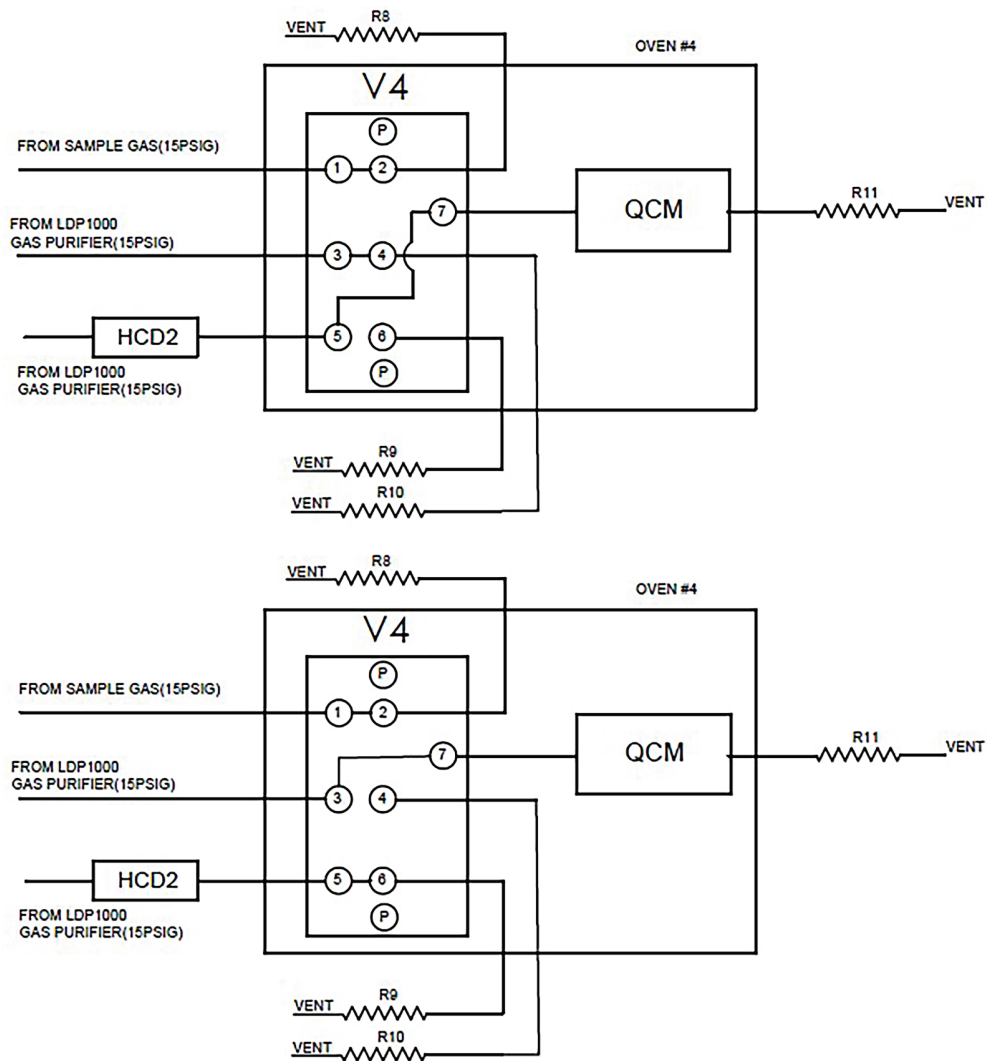
Sampling position:

The V4 diaphragm valve switch between sample gas and dry gas every 30 seconds. The response delta between the dry gas and the sample gas is then measured at the quartz crystal module (QCM). The flow rate inside the sensor is stabilized at 100sccm by the flow orifice R11 mounted at the outlet of the sensor. Two balanced flow orifices R8-R10 keep an equal flow rate of 100sccm to be sure the system stays in equilibrium even with valve switching. This technique ensures the system stabilizes very quickly. This sampling position is the normal running mode and a refreshed reading is proceeding every 60 seconds.

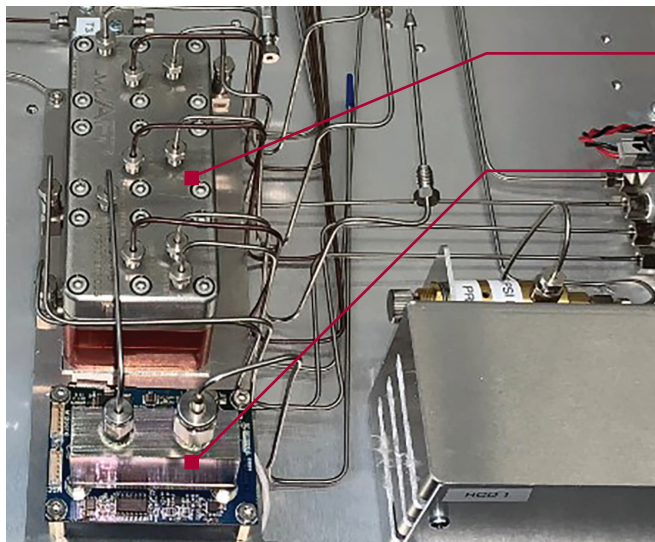


Span calibration position:

The V4 diaphragm valve switch between the dry gas and the wet gas every 30 seconds. This position is used for span calibration purpose. The span calibration frequency depends of the requirements but is generally required every 2 weeks for check spot. During this process, the V4 valve switches every 30 seconds to compare the response delta between the wet gas to the dry gas. Again, the flow is balanced with R9-R10 flow orifices. The humidity control device (HCD2) is continuously kept under a fix flow rate at a stable temperature of 45 Celsius in an inert coated controlled environment. The permeation device generates a certified and known moisture ppb rate. The concentration generated depends on the application, but is generally 500ppb. Having the calibration performed at the bottom of the scale increase the accuracy of the system in the most critical reading region.



MODULE ASSEMBLY



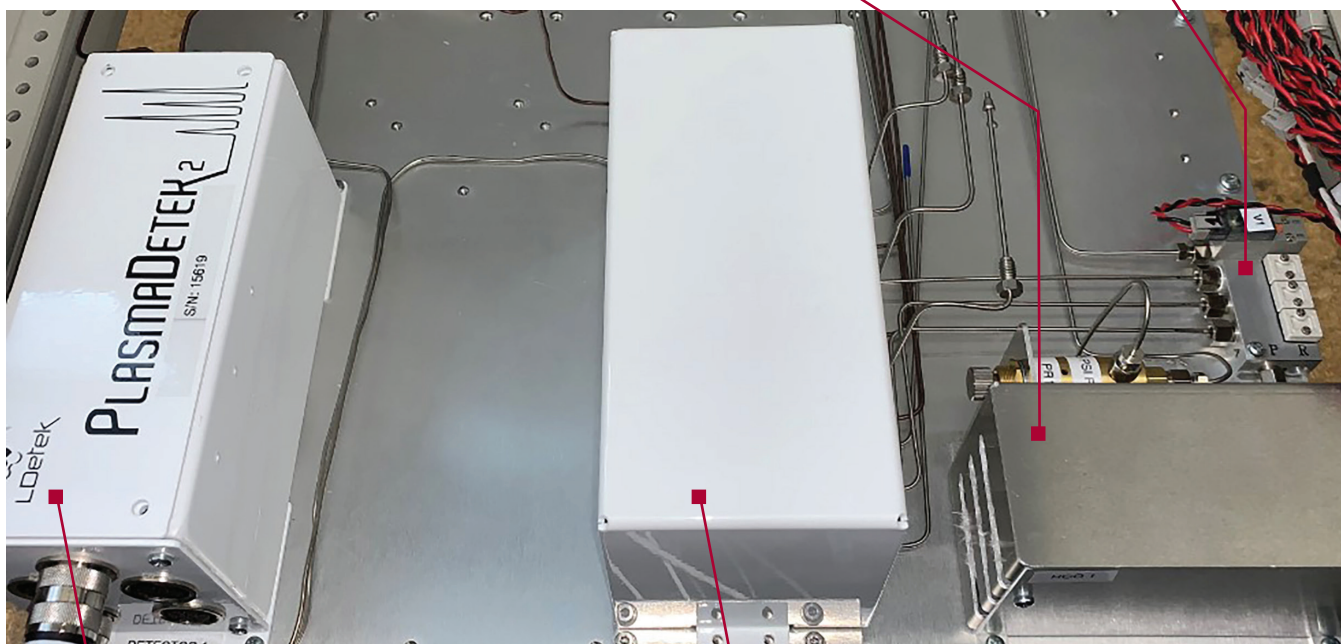
■ 3 streams, inert coated stainless-steel diaphragm valve with sample bypass and purge

■ Trace moisture sensor (quartz crystal microbalance)

*All flow passageways to the sensor are made of stainless steel inert coated with an ID of 0.030"

■ Moisture generator (certified permeation tube) maintained at a fixed temperature

■ Solenoid valve and manifold to actuate the diaphragm valve



■ PED sensor integrated inside the same GC unit to allow other impurities analysis in one GC

■ Temperature controlled module for the sensor and the valve assembly

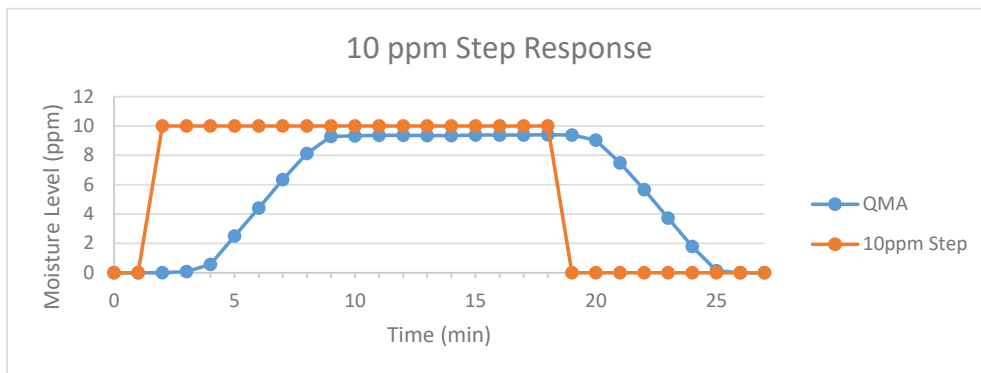
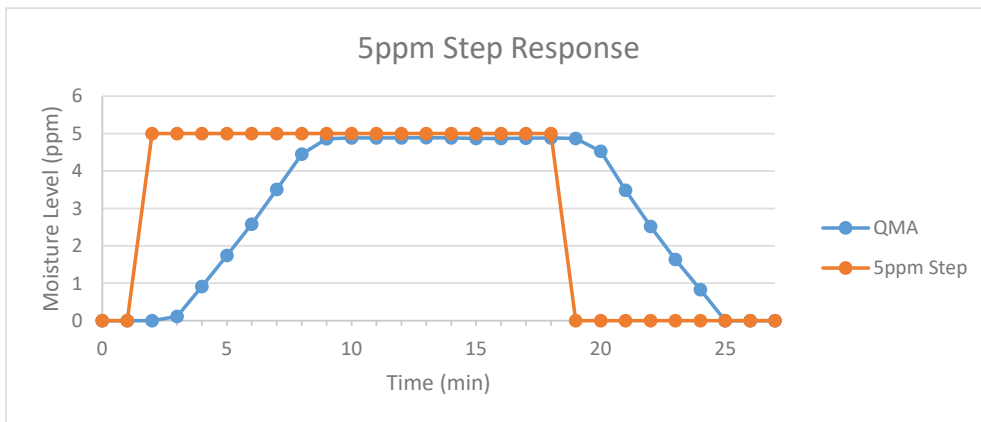
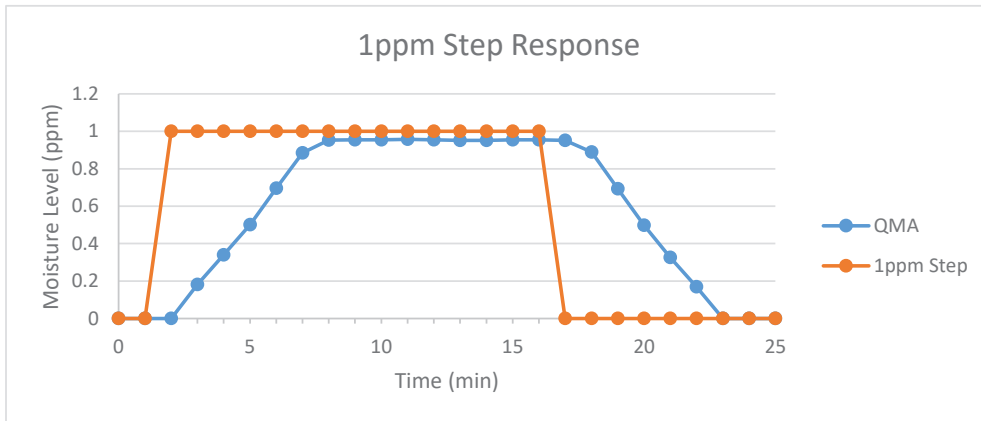
MAINTENANCE

The system is built to be resistant and simple of operation with minimum interventions. A routine maintenance interval every 5 years for replacing the moisture generator, the gas purifier and the diaphragm of the valve is required. The unit has been designed in a way that replacing those components is very easy. In addition to that, the 5 years period is very long what is appreciated to keep the maintenance tasks at its minimum. These tasks are at the same frequency that the ones required inside the GC. So, a GC check can be done every 5 years for all modules together.

ANALYSER PERFORMANCES

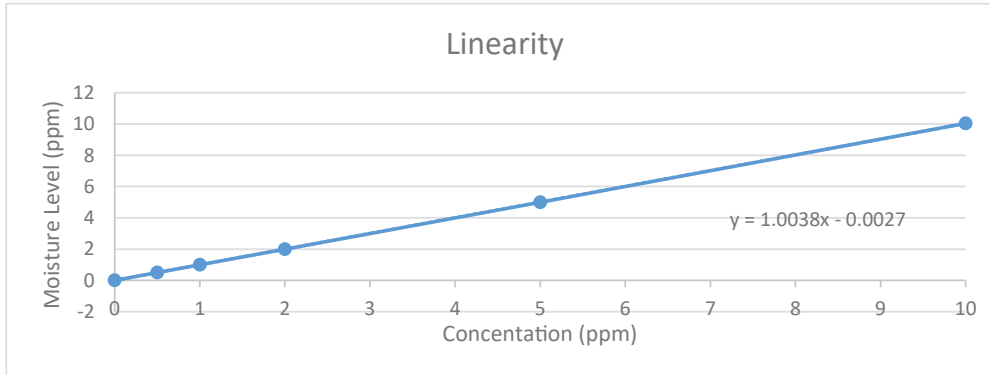
RESPONSE TIME

Different steps of 1ppm, 5ppm and 10ppm have been tested to demonstrate the response time at different concentrations. The response time either on upfront or down end steps remains at about 4-5 minutes. The readings are updated every 60 seconds. The flow path design having low internal volume (0.030"ID) and the purged diaphragm valve makes the system being very quick to respond. On top of that, the system is coated with an inert coating to keep the surface absorption as low as possible to avoid signal drifting. Also, no flow control hardware has been mounted inline with the sensor. The system is configured with bypass flow controller to avoid response delay normally caused by residual dead volume in a standard flow controller.



LINEARITY/ACCURACY/REPEATABILITY

The system is fully linear due to the linearization rectification of every sensor. Each system is characterized in function of its full range to ensure the respond is linear. A series of multiple points at different concentration within the scale are performed.



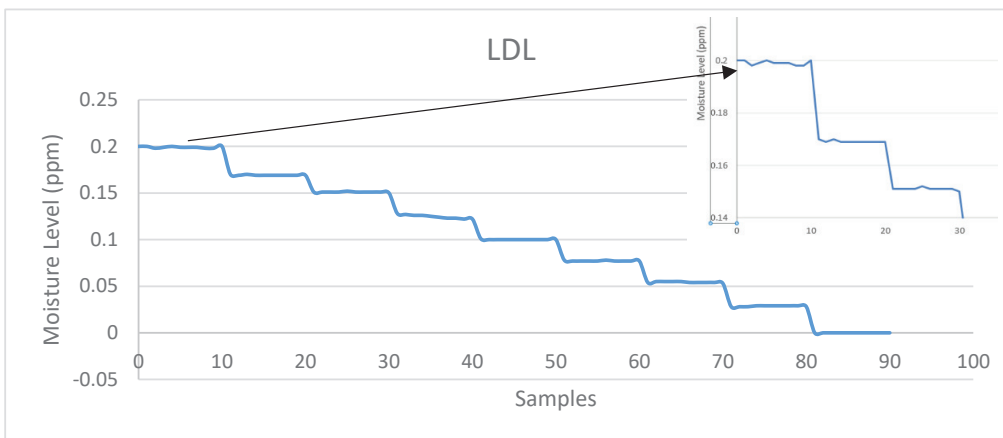
The accuracy error is +/-1% of the scale. In this instance, the table below shows the % error for 6 points diluted at different concentrations between 0-10ppm.

The repeatability error is +/-1% of the scale in a stable environment.

Diluted concentrations *(ppm)	Analyser reading (ppm)	Accuracy (% of scale)
0	0	0
0.5	0.505	+0.05
1	1.004	+0.04
2	1.999	-0.01
5	5.04	+0.04
10	10.042	+0.42

DETECTION LIMIT (LDL) AND NOISE

Multiple steps at a concentration of 25ppb of moisture have been performed to demonstrate the lowest detection limit (LDL). The steps are repeatable and clearly show a good response. Considering a maximum noise level equivalent to 0.002ppm(2ppb) at a concentration of 0.2ppm(200ppb) represented by the zoom section, then 5 times this noise level brings the ldl at a value of 10ppb.



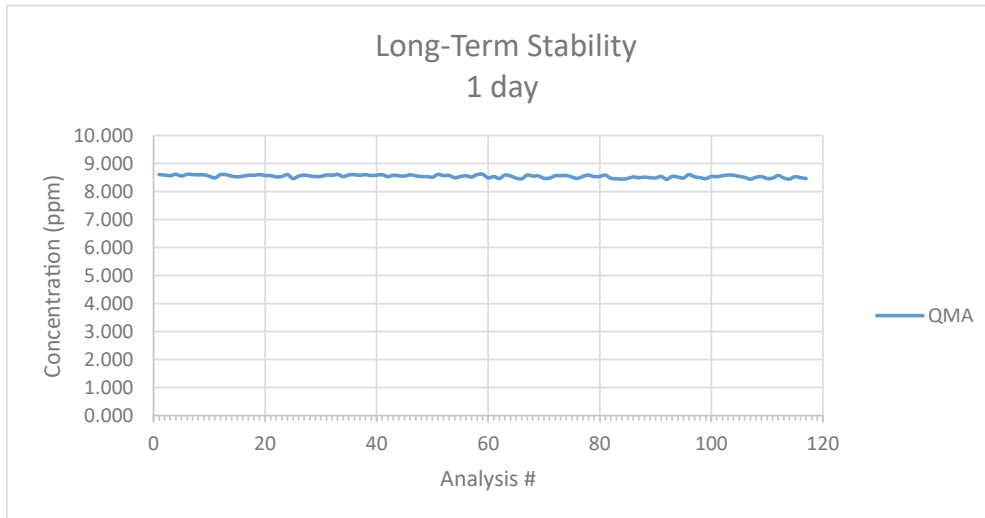
STABILITY/DRIFT

Stability/drift

To evaluate the stability and drift of the unit, a gas bottle containing 8.5ppm moisture in a balance of helium has been connected as sample gas. The system was offering a stability of better than +/- 2% on 24 hours+ period.

8.61ppm (maximum reading) - 8.50ppm (reference value) = 0.11ppm (+1.1%)

8.5ppm (reference value) - 8.44ppm (minimum reading) = 0.06ppm (-0.6%)



CONCLUSION

LDetek is proud to be the first GC manufacturer capable of bringing an innovative and highly performant trace moisture module inside its gas chromatograph MultiDetek2 series. Its presence in LDetek 's portfolio offers the market the possibility to combine many impurities measured by gas chromatography along with an integrated online trace moisture module inside one instrument.

