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Dissolved gases analysis in water

The problem:

Some companies are using water cooler systems where oxygen concentration needs to be controlled in continuous: to avoid corrosion problems. Less oxygen in water involve less corrosion problems in the pipes. A detection limit of 5 ppb of oxygen in water is required.

Solution offers by Chromatotec®:

Chromatotec® has developed a specific system for the measurement of oxygen, nitrogen and hydrogen in water. The analyzer is coupled with a degasing system allowing the extraction of dissolved gases.

Technical information and results:

A continuous degasing system using an inert gas was used to extract oxygen, nitrogen and hydrogen from water. A constant controlled flow of water is passing through a small pipe and is sucking an inert gas. Then, the extracted gases will go to the analyzer to be analyzed. The amount of inert gas is precisely controlled by a Mass Flow Controller (MFC).

A calibration system with a Faraday cell is used to validate the results. This cell is producing a known quantity of oxygen and hydrogen allowing calibration of the instrument.

A/ Degasing system

The different parts of the degasing system are:

- Online water input (accurate flow control)
- A complete glassware especially designed for dissolved gas extraction
- Inert gas inlet precisely controlled with Mass Flow Controller
- Faraday cell piloted by Vistachrom for online calibration
- Sample output through Vigreux column directed to the analyzer
- Water event

The system is designed to extract most of the components dissolved in water: permanent gases and/or VOC.

B/ Calibration

To be able to ensure good quality of results, Chromatotec® has used an online Faraday cell to produce a known quantity of oxygen and hydrogen based on water electrolysis. Therefore, if the water flow and the applied current are stable and well known parameters, it is possible to have an accurate control of oxygen concentration produced. Then, a comparison of experimental and theoretical values is made to validate the whole system.

Here below is an example of calibration made with the Faraday cell:

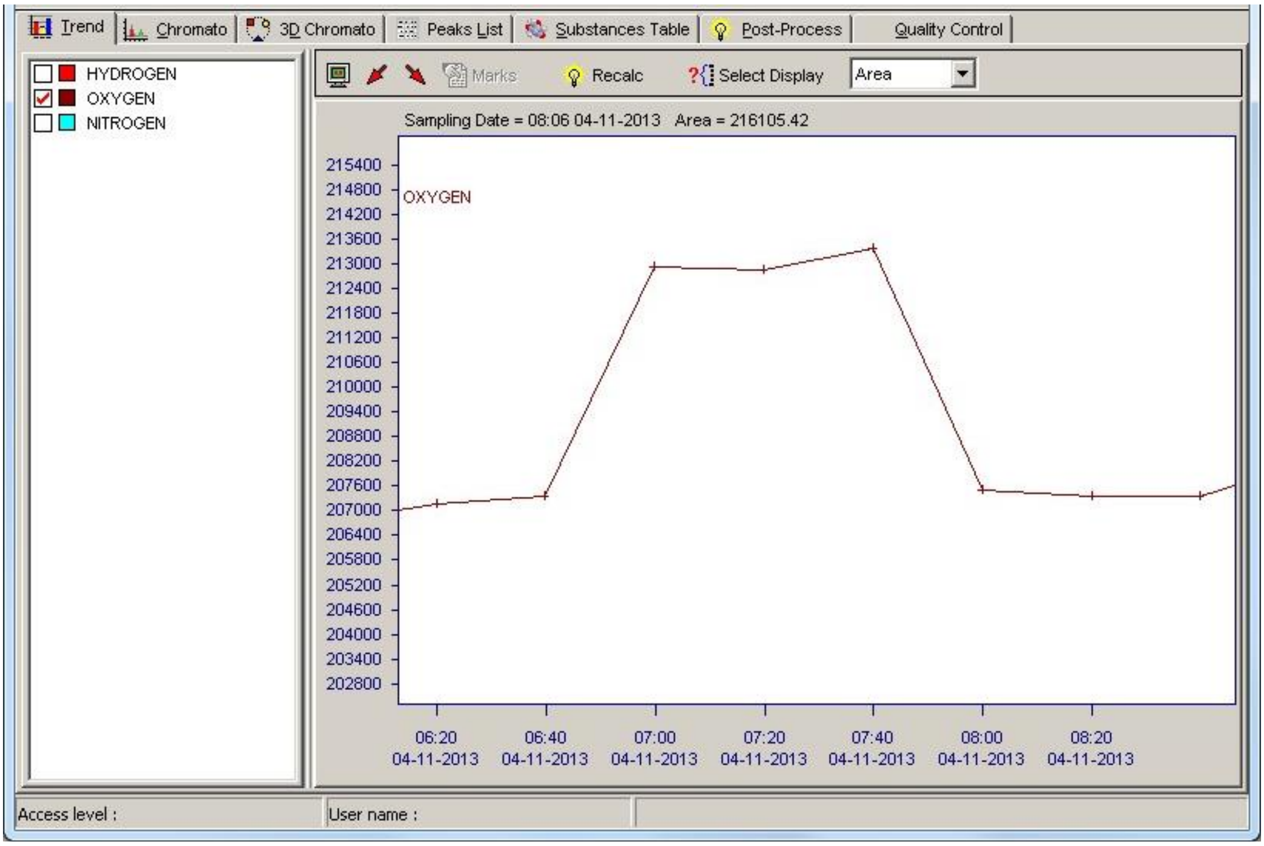

Fig 1: evolution of oxygen peak area during calibration

In this example, the peak area variations before and after the use of the calibration device (calculated with the following formula) are used to calibrate the system.

$$C = (K \times I) / Q$$

With K: known constant (depending on Faraday cell)
C : produced oxygen concentration in $\mu g.kg^{-1}$
I : current applied through Faraday cell in mA
Q : water flow passing through the degasing system in $mL.mn^{-1}$

To study the linearity and the behavior of the whole system, several calibration points were made at different oxygen concentrations (different Faraday cell intensities). The first point corresponds to the dissolved oxygen concentration in standard conditions. The different results were obtained at fixed water flow and temperature conditions. Several analyses were made for each conditions and the average with the error bars are plot here below:

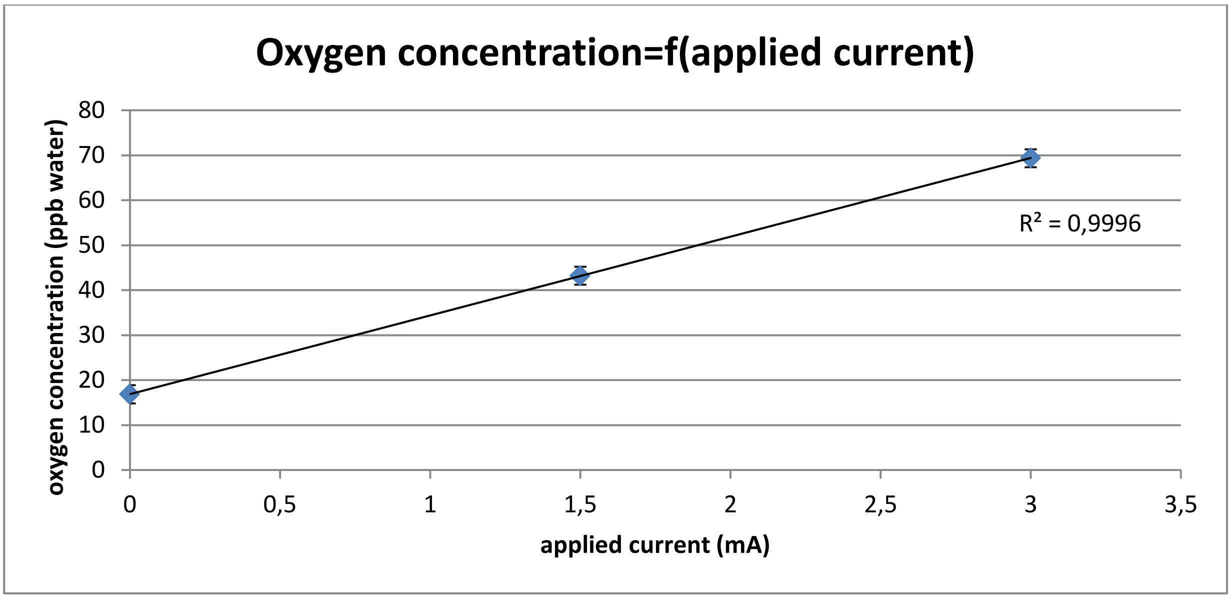

Fig 2: oxygen concentration=f(applied current)

This plot is an evidence of the good linearity and stability of the whole system.

C/ Analyzer: chromaTCD

The fully automatic analyzer dedicated to this application is our new high performance chroma using special µTCD detector. The Limit of Quantification (LOQ) for this system is around 3 ppb of dissolved oxygen in water. The analyzer needs high purity helium cylinder (6.0) as carrier gas coupled to a gas purifier.

chromaTCD: front face view

This analyzer is used to continuously monitor dissolved gases (oxygen, hydrogen, nitrogen) in water.

Here below is an oxygen concentration trend for more than 5 hours of measurement and two calibrations made with the same conditions.

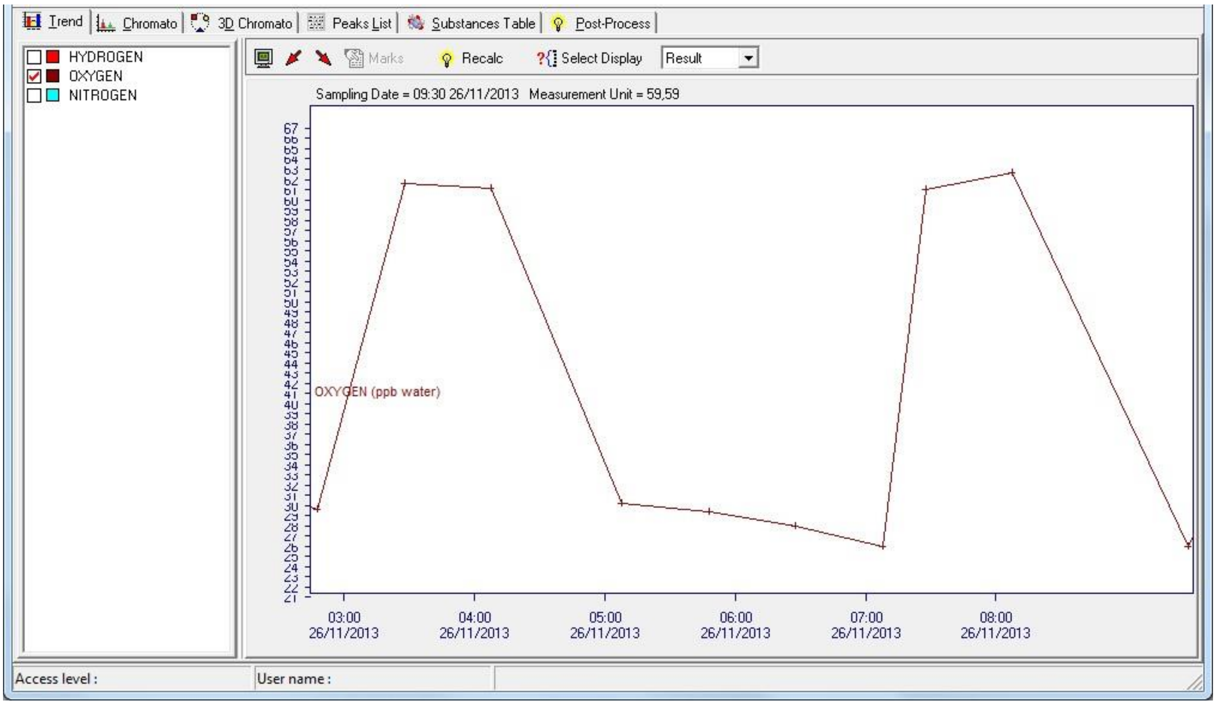

Fig 3: oxygen concentration evolution along the time (6 hours)

We have studied the retention time stability over 45 hours for oxygen and nitrogen.
The relative standard deviation on retention time over the selected period is below 0.5%. Some results are displayed below:

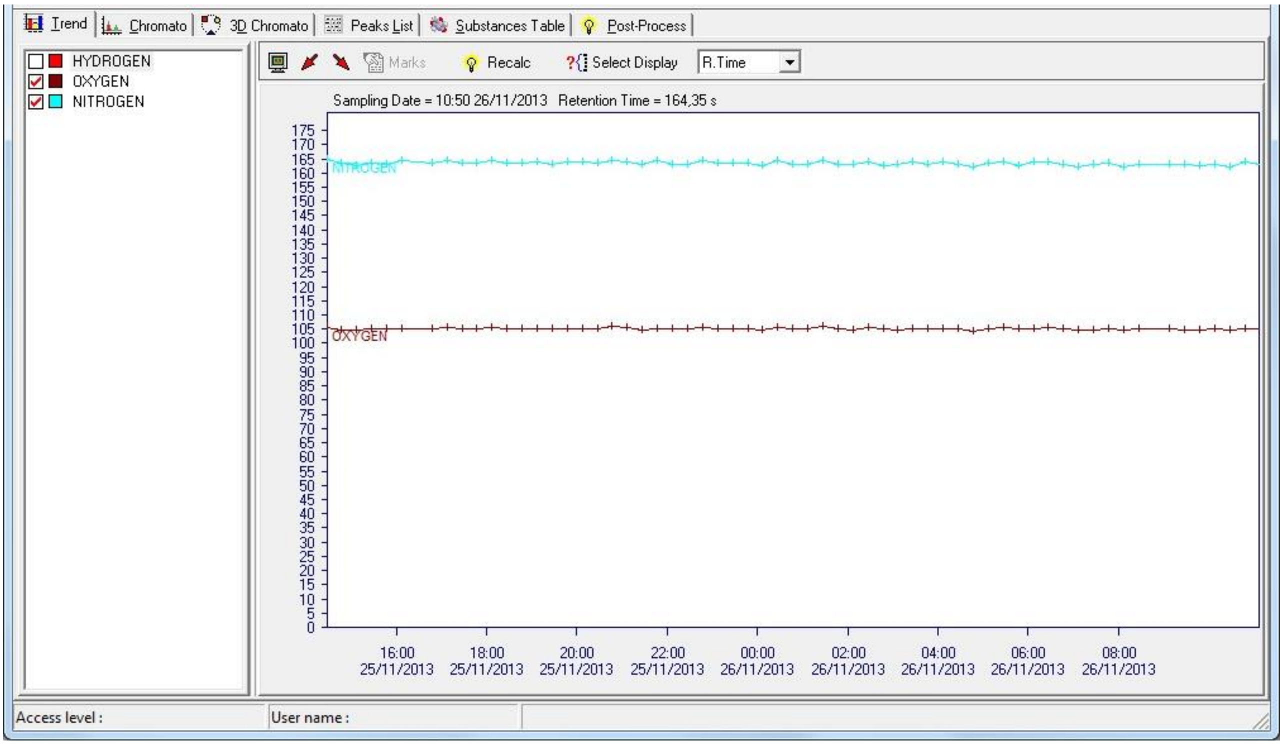

Fig 4: retention time stability along the time (45 hours)

Conclusion:

Chromatotec® has developed a specific system for the measurement of dissolved gases in water. The key point of this system is the on-line validation of results by production and analysis of standard (oxygen and/or hydrogen). Then, Chromatotec® bring is expertise with its new high performance chromaTCD using µTCD detector for higher sensitivity.

The main difficulty was to measure at least 5 ppb of oxygen in water. As the LOQ ≤3ppb is indicating, this difficulty was passed successfully. It will be possible to reach a better sensitivity if requested.

Regarding the system improvement, we are planning to decrease the whole system size.

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