



# Fast and Reliable Online Monitoring Optimises Industrial Processes

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Water is an important component in many production processes and industries. It is raw material, consumable and supply – all in one. Hence, optimisation of processes that use water is significant and hence, its fast and reliable analysis is of importance. Water reuse and therefore its treatment is common practice. Either the operators discharge process water into rivers and lakes or reuse it in processes, the organic contamination of the water in question needs to be determined. In order to describe organic contamination of water the TOC (Total Organic Carbon) is used as a parameter. It is a sum parameter that shows the total content of organic carbon of a sample.

The process oriented construction of a chemical plant is one of the most important aspects to ensure high quality production. Malfunctions may lead to extensive and expensive consequences. In the worst case, the whole production and/or plant may be shut down. To prevent this, the TOC content is detected in the immediate proximity of the processes using an online analyser. In contrast to laboratory methods, these analysers work automatically and detect contamination quickly so that in the case of an alarm, any counter measures or trouble shootings are possible almost immediately.

Modern production sites use online measurement systems to optimise their processes, monitor their waste water effluent and consequently reduce costs. The following article describes two case studies showing how specific online techniques help to optimise economical and economic aspects of industrial processes.

## Monitoring Cooling Water and Waste Water with One Analyser

During the production processes at an international chemical group located in western Germany, cooling water is used. It may be discharged into the river Rhein provided that the local regulations are respected. If the threshold value is exceeded, the discharge will be prohibited. Hence, the operators are requested to monitor the water quality prior to its discharge. However, as organic solvents may enter the cooling water during production processes, it is of importance to monitor these purgeable organic carbons as well. However, they are a real challenge for analyses. In addition to the cooling water monitoring the plant's operators have to monitor the waste water produced which is characterised by a high salt concentration of about 100 g/l.

In order to monitor both water qualities two TOC online analysers were installed that worked with the catalytic high temperature method below 700°C. Despite the use of a dilution unit, the analysers could not reach a higher reactor service life than 7 days. Additionally, these installed analysers worked with the TOC direct method determining the parameter only insufficiently. In the first step of this method, the total inorganic carbon (TIC) is stripped out by adding acid. However, during this process also the volatile and purgeable organic carbons are removed resulting in the so called non purgeable organic carbon (NPOC). This is why this method is also known as the NPOC method. This NPOC value however is claimed to be the TOC. Especially when there was a break through of organic solvents into the cooling water, this method did not correspond to the operators' requirements. In consequence further methods were planned to be tested in order to find a better solution.

The trial started in spring 2008. Among the tested analysers there was one with a wet chemical method and one with the non-catalytic Ultra High Temperature combustion at 1,200°C. The online system using the wet chemical method, nevertheless, proved itself as inappropriate due to its low recovery rates of several substances as well as its strong dependency on the sample's composition. Furthermore, neither the wet chemical method nor the installed catalytic combustion were able to determine both water qualities without any cross contamination.

The non-catalytic TOC online system, however, convinced through its innovative oxidation method, its robust design as well as the exceptionally long service life of its reactor. This system determines the TOC by difference, whereby in the first step, the inorganic and organic carbon compounds are completely and verifiably combusted. The CO<sub>2</sub> produced is detected by an NDIR detector (non dispersive infrared). The result is shown as total carbon (TC). Afterwards, a separate determination of the TIC takes place. The TIC is deducted from the TC resulting in TOC that includes POC and VOC.

In summer 2010 both analysers installed were replaced by one LAR QuickTOC. This TOC analyser now quickly determines any exceeding of the threshold values ensuring counter measures in time. With the two-stream feature the analyser measures both water qualities, the cooling water

as well as waste water, without any cross contamination. Using only one device the operators saved not only high costs of investment, but also costs for operating and maintenance. Additionally, by use of LAR techniques the availability of measurements has been significantly increased.

## Monitoring the Effluent of an Industrial Waste Water Treatment Plant

A German speciality chemicals company with production sites all over the world, monitors the waste water effluent of its site in western Germany for the total organic carbon (TOC) and nitrogen compounds (TN). The waste water is characterised by a very high salt concentration and strongly varying compositions. After treatment, the waste water may be discharged into the river Rhein. If the effluent's contamination is exceeding the threshold values set by local authorities, the waste water will be led into a reservoir.

As the local regulations require the monitoring of TOC and TN, an analyser that measures TOC in combination with TN<sub>b</sub> (Total Nitrogen bound) is now used. The requirements on such an online system are high accuracy, a robust design as well as exceptional availability. To find out the best analysers suitable for the application, the operators tested several systems and methods.

During the first trial one analyser that used a wet chemical oxidation method showed a high dependency on the sample matrix resulting in a bad recovery rate. Additionally, various substances of the resin production were only recovered by 40% which was not sufficient for the operators. Apart from these analytical shortcomings technical malfunctions occurred. A valve in the inlet of the reactor was blocked very frequently causing an increased need of maintenance. A defect of the delivery pump could hardly be solved even by the manufacturer's technical support. The analyser failed the test. The next system tested, worked with a catalytic thermal combustion below 700°C. The TOC was detected by use of a NDIR detector while the TN was determined by an expensive ChemiLuminescent Detector (CLD). However, also for this analyser the application seemed to be too challenging. The sample taking system was blocked





every few days causing fall outs of measurements and the expensive CLD was defective. The plant operators' requirements were not met.

In spring 2011 another trial started. This time an analyser based on the Ultra High Temperature method at 1,200°C was tested. This system determines the TOC and  $TN_b$  by thermal combustion without the need of any expensive catalysts, as the temperature guarantees complete oxidation of all carbon compounds. This online analyser does not require any sample pre-treatment as it handles particles and salts very well. The innovative

XY-injection system injects a part of the homogenised sample into the reactor where it is combusted completely. The so produced  $CO_2$  is detected by the NDIR detector (non dispersive infrared) resulting as TOC. Finally, the nitrogen shown as  $TN_b$  is determined by use of an electro chemical cell (EC-cell). The complete digestion of the ingredients including particulates ensures the accurate analysis of the requested parameters. In the course of the trial, this analyser proved to be a very reliable measurement system. It considered fluctuations of the sample composition, had no problems with high salt concentrations and provided an exceptionally long service life of the reactor. This online system required only little maintenance resulting in a high availability. This online technique was convincingly suitable and hence, won the test.

Since summer 2012 the LAR online TOC analyser helps to optimally monitor and control the effluent of this industrial waste water treatment plant. By reliably monitoring the organic contamination in combination with nitrogen the discharge of the waste water is optimally controlled observing the local discharge regulations.

Either the monitoring of cooling water within industrial processes or monitoring of effluent waste water of industrial WWTP: The applications challenge the online monitoring systems to be installed with various requirements. Especially high salt concentrations or high particle density may lead to technical problems such as blockages and clogging. The compositions of industrial waste water are often changing causing analytical problems and insufficient recovery rates. Specific configurations such as combination with measurement of further parameters or multi stream monitoring are requested as well. More over, innovative and accurate measurement methods in robust designs are demanded.

In order to solve such special tasks specifically customised analysers need to be installed that reliably monitor the water quality of most complex compositions. Being a specialist for online TOC analyses LAR offers a variety of analysers for the determination of TOC in combination with other parameters. Since their installations, the online analysers mentioned above optimally control the processes meeting the operators high requirements. Using the LAR measurement systems saves high maintenance costs due to technical shortcomings or analytical inaccuracy as well as operational costs due to the purchase of expensive catalysts or chemicals. The LAR TOC online analysers particularly suit difficult applications, where other analysers fail.

