

FROM SOUL TO HEAVY METAL - THE ROLE OF ANALYTICAL INSTRUMENTATION AND SPECIALTY GASES IN ENVIRONMENTAL WATER ANALYSIS

Water, water everywhere, nor any drop to drink...

... was the soulful lament in the 'Rime of the ancient mariner'. The idea of water that we can neither consume nor enjoy is a perishing thought and, as Claudine Schneider, former US representative wrote 'A healthy ecology is the basis for a healthy economy'. So, whilst we seek to maintain our industrialised way of life and enjoy the pleasure of pure, clean water in our environment, the use of advanced instrumentation and modern specialty gases has become essential. From purified air in the analysis of TOC in treated sewage to Argon in ICP instrumentation for drinking water purity assay, a sophisticated combination of specialty gases and instrumentation are in use daily to ensure that the quality of water in our environment is kept at the highest standards.

The Magic of Making Dirty Water Clean

When we have finished with our bathwater or when factories discharge waste water there are many process steps involved before it is returned to the rivers or oceans as clean water. A common final step in the process is to accurately determine the water quality before final discharge. To do this, many waste water treatment facilities are equipped with an online TOC (Total Organic Carbon) analyser.

Waste water flow rates to the sewage works are unpredictable because they come from uncontrolled sources and are influenced by diurnal cycles, sudden rainfall or rapid changes in operating conditions of factories feeding the treatment works. Despite these tough process parameters, the discharge consent levels must be met consistently. For this reason, the online instrumentation measuring TOC levels in the treated waste water needs to react quickly. Modern instrumentation using oxidation reactions and measuring the TOC as oxidised CO_2 in the gaseous phase with an NDIR detector are currently able to provide a response signal from a sample within 2 to 3 minutes. If problems are detected, mitigating actions can quickly be put into place. This means that discharge consent levels can be maintained and the environment will be protected.

There are several oxidation technologies in use in online TOC analysers. Some rely on a reaction between air and the organics in the water over a catalyst operating at high temperatures between 680 and 1200°C. Others use the addition of Persulphate and air in the presence of UV light as the main oxidation process. A

third process relies on the extreme oxidation potential hydroxide radicals. All three of these technologies rely on the supply of air as the primary source of Oxygen. Martin Schlögl, Country Manager for the Czech Republic and Slovakia at Hach commented, "We use the patented TSAO oxidation process in the Hach BioTector TOC analysers. This process takes in air to create ozone and combines that with the sample in a highly alkaline environment." The hydroxide ions present at pH 12 create hydroxyl radicals in the presence of ozone and these radicals rapidly convert all available TOC to CO_2 . Shlögl continues, "the main advantage that we see with this low temperature technology is that it is extremely robust and highly reliable. For example, it is highly resilient to typical operating parameters that may adversely impact other systems, such as elevated levels of Calcium and other salts, algae build up and occasional oils / fats / grease carry over from the water treatment plant. Therefore, it results in maximum up-time and minimum maintenance for the user".

Clean Water Measurement Requires Clean Air

Since these TOC analysers are used at the back end of the water treatment process to ensure that the job has been done, this analysis must be done without introducing any contaminants to the system that could falsely inflate the TOC readings. For this reason, a high purity air supply to all of the three systems above is essential. The key attributes of the air fed to these systems are



pic credit: Linde AG



Hach B3500c



Biodetector TOC Analyser

that it should contain approximately 21% oxygen and it should be free of hydrocarbons. If the air were to introduce additional hydrocarbons, for example lubricant oils from an air compressor which are in themselves organic compounds, that would add to the TOC count and would falsely inflate the TOC reading.

Supply of high purity air to the online TOC analyser can be achieved using a variety of options. Firstly, site instrumentation compressed air can be used. This does call for additional purification steps and, if the system is not frequently monitored, hydrocarbon or CO₂ breakthrough can occur. So, to avoid these problems it is now common to use specialised high purity air generators. Gerard Catchpole, European Sales Manager - Gas Division of VICI AG International explains, "Our GT Ultra Zero air generator is ideal for online TOC analysers. This device uses an oil free compressor and has various types of gas scrubbers to remove acid gases such as CO₂, SO₂ and NOx from the air stream. This is important because sulphurs such as SO₂ can poison the oxidation catalysts used in some TOC instrumentation. There is also a platinum based catalyser in our air generator that is super effective for the removal of hydrocarbons to minimise interference with the TOC analyser".

As a result of the final catalytic oxidation stage in these generators there can be a small level of CO₂ contained in the purified air. For the majority of online TOC analysers this represents no problem, because the baseline CO₂ level in the air can be analysed by the TOC instrument's NDIR detector and subtracted from the final TOC reading. However, if end users are not comfortable with this mode of operation there is still one more weapon left in the armoury. For the highest levels of purity and confidence, it is possible to source specialty gases cylinders of Zero grade synthetic air from gas companies such as Linde.

Dr Roberto Parola, Global Specialty Gases Product Manager with Linde Gas commented, "our HiQ grades of Zero air can be supplied free of hydrocarbons and CO₂ and can, on request, be accompanied by a certificate to prove this. Small portable cylinders are available for remotely located instrumentation, larger cylinders and bundles are also available for convenient continuous supply to laboratories using online TOC analysers".

Heavy Metal – Purity Analysis in Drinking Water

Another measurement that is critical for environmental management and public health is the assay of heavy metals in water. The levels of toxic elements which as Mercury, Cadmium and Strontium in drinking water can be analysed using ICP AES or the more sensitive ICP MS instruments. These technologies are generally replacing the previous alternative of flame photometry atomic adsorption spectroscopy (AAS). The operating principle of ICP is to create a high energy plasma from Argon gas. This plasma is the modern alternative to the high temperature AAS flame which is typically generated by the combustion of acetylene and nitrous oxide gases.

Matt Onions, CRYOSPEED® product manager at BOC UK & Ireland says, "we have been supplying Acetylene and Nitrous Oxide for AAS applications for many years and we continue to do so, but the trend in trace metals analysis in water is an increasing demand for Argon to be used in ICP instruments. The quantities of Argon consumed to create the plasma are quite high and an analytical run will often consume more than the contents of a traditional gas cylinder. In response to this, we have developed modern gas



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supply solutions such as CRYOSPEED®. With this service, our operators deliver liquid argon directly into a 200 litre tank which resides at the customer site." The liquid argon vaporises slowly to give the required gaseous argon flow that the ICP instrument requires. It allows the safe storage of vast quantities of argon in a very limited space, so the lab technician can be certain that they will experience hassle free supply of Argon throughout multiple analyses over many days and weeks. Onions added, "In addition to continuous supply over extended periods, the benefits of the CRYOSPEED® liquified gas delivery service are fewer changes of cylinders for the lab team, resulting in improved productivity in the laboratory and a reduction the associated support processes such as ordering and stock management."

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