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PROCESS-WATER CHARACTERIZATION USING DISCRETE ANALYZERS AND ION CHROMATOGRAPHY

OVERVIEW

Process water, the non-drinkable water that many companies rely on for a variety of industrial applications, undergoes extensive treatment. This water must be analyzed and processed to meet the needs of specific industries. Several technologies can address a variety of water analysis needs and can increase the speed, quality, and consistency of process-water data analysis.

INTRODUCTION

Maintaining high-quality water is critical for many industries that use water as part of their manufacturing processes. Poor-quality water can corrode costly equipment, waste resources, and compromise the end product.

To meet the needs of a specific application, source water—which includes groundwater and raw water—must first be converted to deionized water using a series of treatment steps. Each water sample must be tested for multiple parameters, to ensure that the produced and reused process water is free of contamination. Companies must test the water samples at the point of use and multiple times throughout the industrial process, as the water quality changes as it passes through the various process stages (**figure 1**).

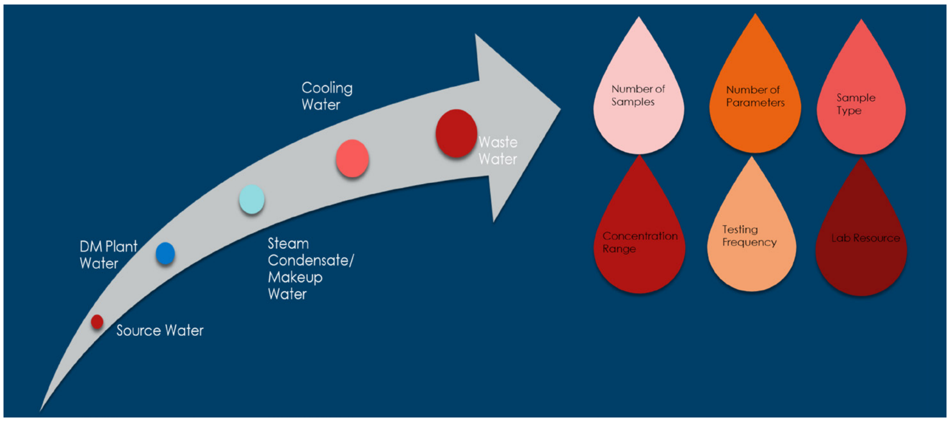

Figure 1: Sample types and testing parameters involved in process-water analysis. "DM" stands for "demineralized" in this figure.

Image credit: Thermo Fisher Scientific

Basic water analysis starts with tests for pH, conductivity, alkalinity, and total hardness. These parameters are critical for maintaining manufacturing plants that utilize or treat deionized water. Typically, these parameters are tested using pH and conductivity meters' auto titration system for all water samples. Corrosive anions, corrosion inhibitors, and corrosion products are tested by other wet chemical methods, such as spectrophotometry, colorimetry, and flow injection analysis. Many of these traditional testing methods can analyze only one or two parameters at a time, so multiple wet chemical instruments are often needed to characterize a water sample (**figure 2**).

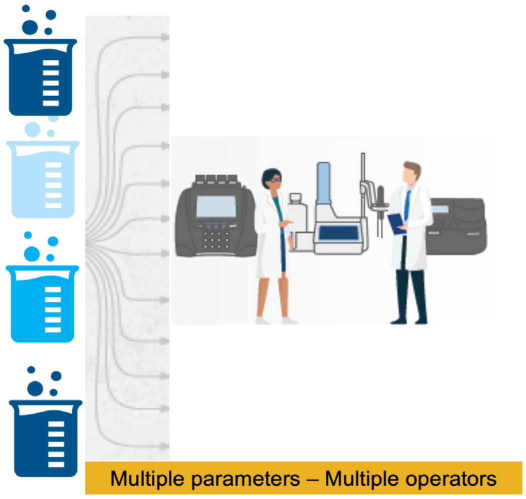

Figure 2: Problems and impact of multi-parameter analysis

Image credit: Thermo Fisher Scientific

This process can be labor intensive and time consuming, require large volumes of sample, and generate excessive amounts of waste. Further, instruments capable of simultaneously testing multiple parameters are available but must be run in a sequential rather than a parallel manner, so testing many samples still takes significant time and requires highly skilled operators.

SPEEDING UP WATER ANALYSIS

The Thermo Scientific™ Discrete Analyzers (**figure 3**) is a fully automated, all-in-one platform that can substitute for multiple wet chemical techniques to perform rapid analyses of water samples. Unlike other multiple-parameter systems' sequential operations, the Thermo Scientific™ Gallery™ platform can conduct multiple tests in parallel, decreasing the time needed for testing. These high-throughput analyzers can perform up to 350 tests per hour; each test is on the microliter scale. The Gallery platform also uses only 200 microliters of sample per test versus the 50–100 milliliter of sample per test required for traditional methods—and generates milliliters rather than liters of waste. In the Gallery platform, all aspects of workflow, including reagent addition, incubation and waiting time, and measurement, are automatically performed after the user loads the sample and reagents into the system. As such, the user does not require much training to operate the Gallery discrete analyzers. The approach also is compatible with a range of analyte concentrations and water-sample types, including seawater, rainwater, groundwater, and wastewater.

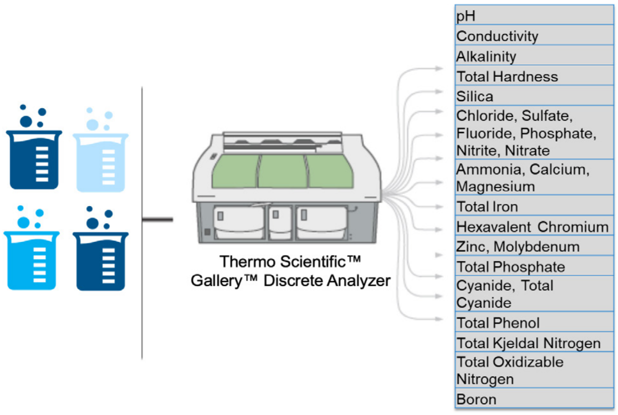

Figure 3: The Gallery Discrete Analyzer tests a wide range of parameters, offering multiple benefits and solutions.

Image credit: Thermo Fisher Scientific

The Gallery platform combines electrochemical and photometric methods to analyze water samples. The platform uses a Xenon lamp and a freely configurable filter wheel to measure up to 20 different photometric parameters simultaneously while the platform's electrochemistry module (ECM) can evaluate pH in the 2–12 range and conductivity within the 20 µS/cm–112 mS/cm range. Conductivity measurement with ECM is not suitable for water with low-to-medium ionic strength, such as deionized water or steam condensate.

The Gallery Discrete Analyzers can consolidate several different analyses needed to test process water and measure analytes, including corrosive anions such as fluoride and sulfate, corrosion inhibitors such as ammonia and phosphate, corrosion indicators such as total iron and hexavalent chromium, and scaling parameters such as silica and calcium. The Gallery platform can also conduct analyses needed to characterize wastewater, like total Kjeldahl nitrogen and free and total cyanide content, and perform basic water analyses such as measuring hardness and alkalinity.

PERFORM COMPREHENSIVE WATER ANALYSIS

Some water analyses can be challenging because of trace levels of analytes in the sample or matrices that can overwhelm instruments with unwanted signals. These applications—and those requiring the characterization of analytes not measurable by the Gallery platform—may be more suitable for Thermo Scientific™ Dionex™ ion chromatography (IC) systems, which can be configured for specific needs. Dionex IC systems can identify anions and cations, perform detection up to parts per trillion (ppt), and analyze a broad range of analytes—even if they reside within complex sample matrices. In addition, the technology can be adapted to perform continuous online monitoring, which gives users the ability to quickly respond to changes in process-water quality, prevent damage to equipment, reduce costly downtime, and maintain product yields.

As shown by an example analysis in **figure 4**, small-particle Thermo Scientific™ Dionex™ IonPac™ columns have the advantage of increasing sample throughput without compromising data quality. When the flow rate through a column is increased from 0.3 mL/min to 0.5 mL/min, the run time of the analysis is almost halved, from about 9 minutes to 5 minutes. The peaks obtained in the faster analysis remain sharp and separated, however, indicating that they can still be easily integrated to quantify the data.

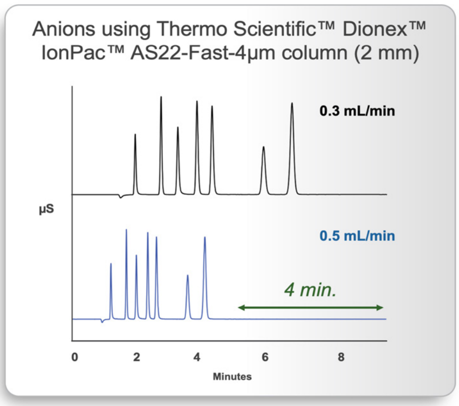

Figure 4: Impact of increasing flow rate through the Thermo Scientific™ Dionex™ IonPac™ AS22-Fast-4µm column (2 mm) on the chromatogram of inorganic anions.

Image credit: Thermo Fisher Scientific

To produce high-purity eluents for sample separation in real time, Thermo Fisher Scientific has created Thermo Scientific™ Dionex™ EGC Cartridges, which can be installed onto a Dionex IC system to generate eluents from deionized water using electrolysis. This reagent-free technology can enhance worker safety by avoiding the harsh chemicals usually required in the manual eluent preparation, improve consistency by removing variability and averting potential contamination, and allow users to take advantage of gradients without having to purchase a proportioning pump, which can be expensive.

Gradients can greatly facilitate the processing of chromatographic data to obtain highly reproducible results. **Figure 5** shows anions that had been eluted from a Dionex IonPac column using eluents having either a constant concentration (top) or a linearly changing concentration (bottom). While a constant concentration of eluent yielded a chromatogram in which all the components of interest resolved into separate peaks, the sharpness of the peaks and the separation between them were greatly improved when concentration changed over time.

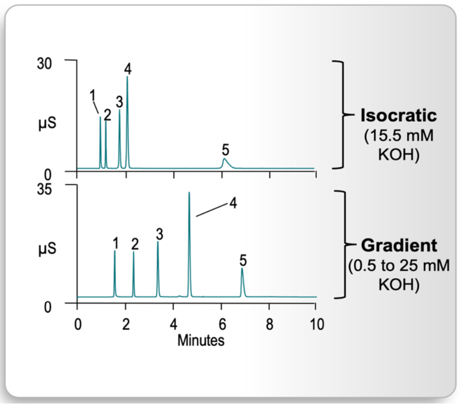

Figure 5: Chromatogram of water sample using isocratic versus gradient eluent concentrations.

Image credit: Thermo Fisher Scientific

TACKLE MATRIX INTERFERENCE AND CORROSION

The following examples show how Thermo Scientific Dionex IC solutions can aid in process water analysis.

Trace determinations of corrosive anions

One strength of ion chromatography is determining trace concentrations, which is particularly helpful when monitoring corrosive anions. **Figure 6** shows the measurement of corrosive anions in water using a Thermo Scientific™ Dionex™ Reagent-Free™ IC (RFIC™) system and a Dionex IonPac column. As the sharp peaks in the chromatogram illustrate, the instrument achieves high sensitivity because of its low background, low baseline noise, and high-purity eluents. Though the figure shows concentrations of parts per billion (ppb), concentrations in ppt can also be determined.

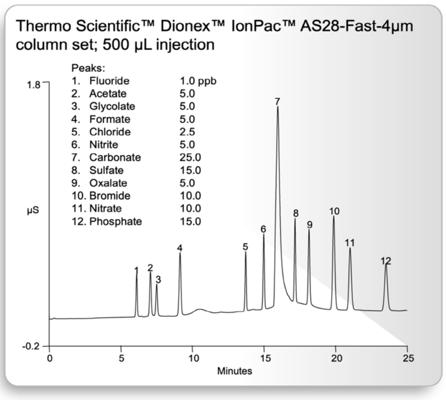

Figure 6: Achieving ppt-level determinations using Thermo Scientific™ Dionex™ IonPac™ AS28-Fast-4µm column set (500 µL injection)

Image credit: Thermo Fisher Scientific

Corrosion inhibitors

Determining the concentration of corrosion inhibitors is often part of a comprehensive corrosion-monitoring program. Some examples include using amines to control pH and scavenging oxygen using hydrazine. For these applications, ion chromatography with suppressed conductivity detection (**figure 7**) can determine the ppb to parts per million (ppm) concentration of common cations and amines.

The Dionex IonPac column set used to yield **figure 7** has been optimized in several ways. First, the column was designed to elute ethanolamine (Peak 3) before ammonium (Peak 4) so that a small ethanolamine peak was not obscured by a much larger ammonium peak. Second, a high-capacity column was used for the analysis to prevent overloading and ensure that none of the sample was lost to waste before analysis could be conducted. Thus, having an extensive column portfolio and being able to easily modify the method parameters can help optimize the analysis of process-water components.

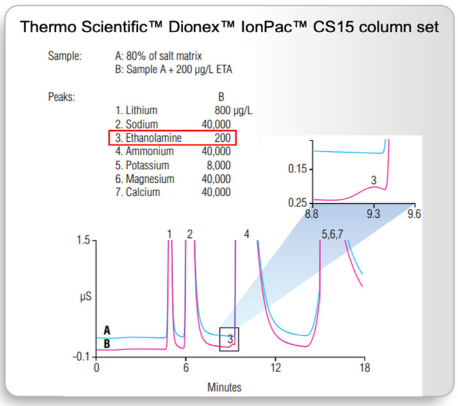

Figure 7: Detecting small concentrations of corrosion inhibitors in the presence of larger signals.

Image credit: Thermo Fisher Scientific

Corrosion indicators

Transition metals can contaminate process water and can indicate when corrosion has occurred. High levels of transition metals, for example, can signal potential equipment failure. **Figure 8** shows a chromatogram of a Dionex IonPac column set that has been optimized to select for cations.

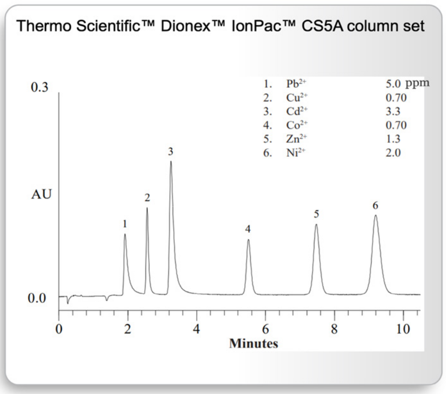

Figure 8: Chromatogram of various transition metals in water using Thermo Scientific™ Dionex™ IonPac™ CS5A column set.

Image credit: Thermo Fisher Scientific

Complex matrices

Matrix components can interfere with analytical determinations by, for instance, overwhelming the system so that the signal from analytes is lost or by absorbing at the wavelengths that a colorimetric assay is measuring. Users can overcome matrix interference by modifying their IC system configuration. For example, users can use selective, high-capacity columns, which can tolerate high matrix load; alternative detectors that are not adversely impacted by sample content, like UV-vis or electrochemical measurements; and pre-processing procedures, such as diverting matrix components into waste and concentrating analytes of interest prior to separation.

Figure 9 shows a chromatogram of a sample of water that contained high levels of boron (2,000 ppm) and a lower level of lithium (3.8 ppm). In this method, a large sample volume (2 mL) was loaded onto a Dionex IonPac concentrator column and rinsed with 10 mL of solution so that most matrix components were rinsed into waste. Meanwhile, the analytes of interest—the trace ions within the water—remained. The materials were then loaded and separated on another Dionex IonPac column. As seen in the figure, the ions resolved as separate peaks even though their concentrations were initially much lower (ppb level) than that of other matrix components (parts per thousand level).

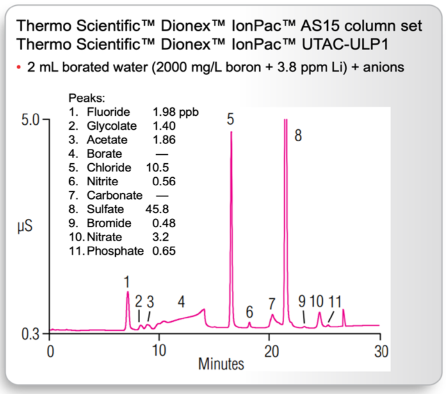

Figure 9: Chromatogram of trace ions in water containing higher concentrations of boron and lithium.

Image credit: Thermo Fisher Scientific

CONCLUSION

Process-water analysis can be challenging, but the right instruments and methods can address those challenges. The Gallery platform (**figure 10**) provides routine, high-throughput analysis for many parameters of interest, including pH, conductivity, and concentration. The platform can achieve up to ppb levels of detection and does not require skilled operators. Thus, the Gallery Discrete Analyzer is a "walkaway" solution for many water analysis needs.

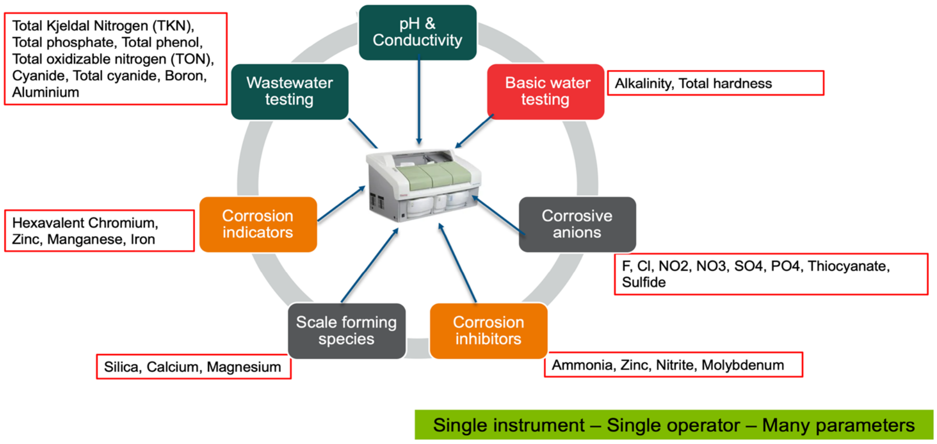

Figure 10: Example analyses performed by the Thermo Scientific™ Gallery™ Plus Discrete Analyzer.

Image credit: Thermo Fisher Scientific

While the Gallery platform is suitable for a wide range of user needs, those who require a comprehensive solution for water analyses should consider also incorporating into their lab a Dionex IC system (**figure 11**), which can characterize a much broader range of anions and cations and achieve up to ppt levels of detection. IC can also help minimize signal interference from a sample matrix.

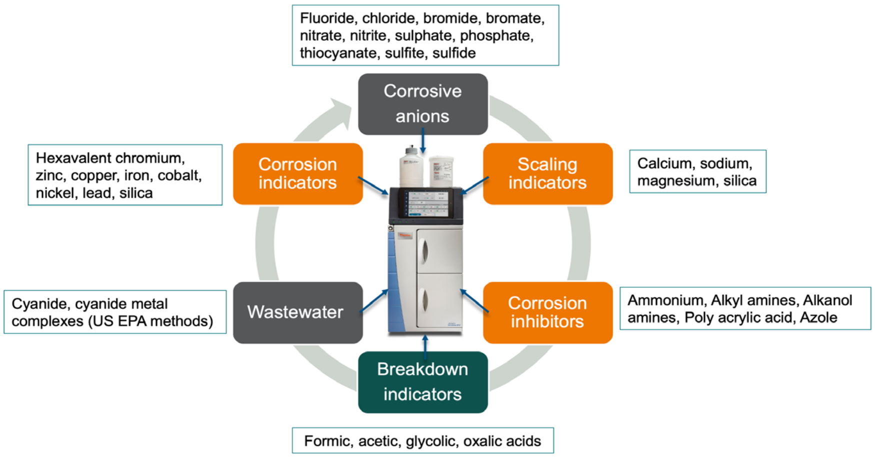

Figure 11: Example analyses performed by Thermo Scientific™ Dionex™ ion chromatography systems.

Image credit: Thermo Fisher Scientific

For users that want the benefits of both a discrete analyzer and an ion chromatography system, Thermo Fisher Scientific has combined these two technologies into one package: the Thermo Scientific™ Disc-IC™ System (**figure 12**). The fully automated Disc-IC system combines direct measurement and discrete analysis to support a comprehensive water analysis workflow, offering flexibility and high throughput to reduce overall cost per analysis. Users can load a large series of groundwater, process water, and wastewater samples in the Disc-IC system and walk away as the instrument performs comprehensive and routine water analysis that is essential for optimal process-water quality control.

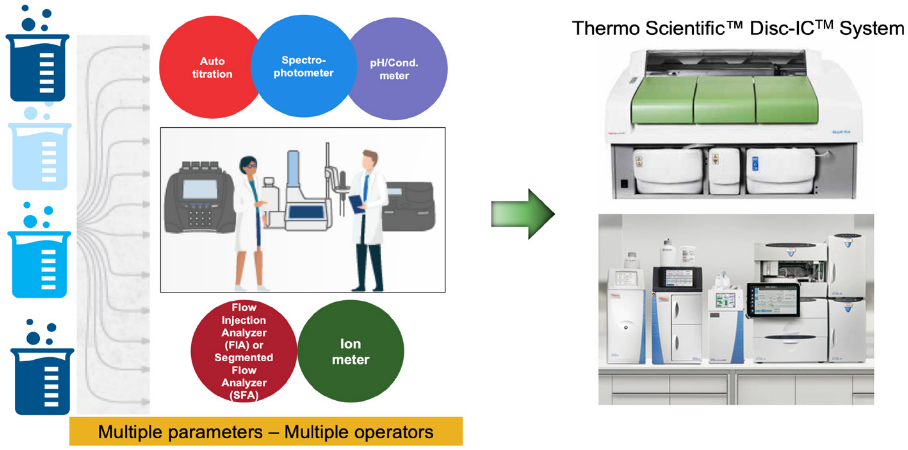

Figure 12: Perform discrete analyzer and ion chromatography analyses using the Thermo Scientific™ Disc-IC™ System.

Image credit: Thermo Fisher Scientific