# Rapid and Mobile Testing for Monitoring and Ensuring Water Quality

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**Despite its simple chemical formula, water can pose a challenge for analytical test methods. For example, measurement of chemical oxygen demand (COD), regularly performed to test for pollutants, can be difficult when the water sample contains very high chloride levels such as those found in wastewater or seawater. With conventional tests, chloride causes interference and can result in erroneously higher COD values.**

Another challenge presented by water testing is the risk of sample deterioration or contamination; this is of special concern when samples must be transported to the lab for testing. Routing of samples back to the lab also limits the ability to make quick decisions on-site and adjust in-process controls in real-time. Additionally, with so many possible contaminants and parameters to monitor during water testing, the analysis process can become time-consuming, cumbersome and require a wide range of instrumentation.

New technologies for analysis can overcome the inherent limitations in conventional methods, facilitate analytical quality assurance, help identify matrix effects and accelerate time to results.

*Figure 1. The Spectroquant® Move 100 is a portable colorimeter offering over 100 preprogrammed and 35 user-defined methods.*

## The Need for Portability

Traditional systems for water analysis are not portable, meaning samples must be collected and transported to the lab for testing, increasing the risk of sample contamination and deterioration.

The Spectroquant® Move 100 (**Merck Millipore**, Darmstadt, Germany, *Figure 1*) is a portable colorimeter for the analysis of drinking and waste water. The instrument covers every important parameter of drinking and waste water analysis and includes more than 100 pre-programmed and 35 user-defined methods enabling a wide selection of measuring ranges without the need for additional instruments. Because the system is compact and mobile, it allows water to be tested immediately on site, speeding time to result. This enables laboratories to avoid sample deterioration and identify contamination events sooner.

## The Chloride Dilemma

Chemical oxygen demand (COD) measurements are regularly performed to test water for pollutants. Industrial wastewater and seawater can contain high levels of chloride, which may interfere with the determination of COD. Standard COD tests cannot be used as chloride interferes positively with results and can lead to erroneously higher COD levels.

To overcome this challenge, Merck Millipore has developed the first COD cell tests with unlimited chloride tolerance. The high chloride content is extracted by a special pretreatment step prior to adding the sample to the new COD tubes. The two new Spectroquant® COD cell tests facilitate measurements in the low range of 5.0 - 60.0 mg/l COD and in the higher range of 50 - 3000 mg/l COD, making them well-suited for analyzing a wide variety of industrial wastewater and seawater samples.

## Analytical Quality Assurance

Reliability of water quality monitoring data depends on strict adherence to standard operating procedures for sampling and analysis. Consistent application and monitoring of these procedures is referred to as analytical quality assurance (AQA).

Worldwide, there are a number of quality-assurance systems in place to ensure the safety and efficacy of substances and products. In the field of laboratory testing, this is generally regulated through good laboratory practice, or GLP, which stipulates the right documentation for quality assurance, as well as the implementation of three different levels of control. These controls are Installation Qualification, to assess whether instruments were correctly installed; Operational Qualification, to prove that the system operates as it should; and Performance Qualification, to check product-related performance by verifying method-specific standards.

*Figure 2. Spectroquant® PhotoCheck (left) used to check spectrophotometers Spectroquant® Pharo 100 and 300 (right).*

Operational qualifications of spectrophotometers, commonly used to evaluate water quality, should be verified by an instrument check which covers the assessment of the instrument itself.

With spectrophotometers, it is essential to check the wavelength accuracy, and the linearity of the absorbance measurement. Spectroquant® PhotoCheck solutions for use with Spectroquant® systems, consist of various highly stable solutions. Absorption is tested at various wavelengths and concentrations, in order to evaluate the instrument's performance. The system independently compares the measurement results with target values, and calculates tolerances. Hence, the user can tell at a glance whether the spectrophotometer has passed the test, and if it is operating properly.

UV Vis spectrophotometers should also be regularly checked for accurate and consistent results, as well as precise operation. The test includes parameters such as absorption, stray light, and wavelength accuracy. A UV Vis standard solution with a known concentration greatly facilitates monitoring and delivers secure results.

The next step is the performance qualification that evaluates the complete system; in this case, not only the instrument, but also the user's handling, the test kit, and pipettes are checked, using method-specific standards.

This comprehensive assessment can be easily accomplished with Spectroquant® CombiCheck standards, ready-to-use multi-parameter standards for the range of applications. The standard solution can be used to confirm the accuracy of the results from the photometer system as a whole (photometer, reagents, analytical procedure, and implementation). In addition to the CombiCheck, the user can choose from single-parameter standard solutions. The Certipur® standard solution (Merck Millipore) has a concentration of 1,000 mg/L; thus, any desired concentration can be prepared by making the appropriate dilution. In contrast, Certified Reference Materials (CRM) are available in varying concentrations, so that no further dilution is required.

Besides testing the overall system, it is also necessary to identify any measurement errors due to possible interferences within the sample. In waste water analysis, large differences can be expected in the composition, or matrix, of the samples to be analyzed. Besides the analyte, the sample often contains foreign substances or impurities, which can significantly influence the recovery of the analyte. If the exact composition of the matrix is not known, it is impossible to estimate the potential influence of foreign substances on the analysis. This is referred to as "the matrix effect."

Standard addition or dilution can be used to recognize measurement errors due to matrix effects. Any such interferences can be analyzed on the basis of the recovery rates and correspondingly rectified by taking appropriate countermeasures, such as pretreating the sample accordingly.

Spectroquant® CombiCheck also contains a highly concentrated addition solution. Due to the relatively high concentration, only small quantities need to be added, so that the matrix of the sample is hardly affected and only one measurement is needed. (*Figure 3*)

When using CRMs for standard addition, volumes of the sample solution are mixed with increasing volumes of a high-concentrated standard solution of the analyte. At least three spiked solutions should be prepared. The volume of the sample and that of the addition solution are entered into the spectrophotometer and are automatically indicated, as well as the target value. (*Figure 4*)

The instruments independently calculate the recovery rate, and clearly display whether a matrix effect is present. This is the case when the recovery rate lies considerably below 80%, or above 120%. A positive result, meaning the presence of a matrix effect, signifies that the sample in question cannot be properly investigated without appropriate pre-treatment, in order to achieve secure results, such as filtration, pH adjustment, or cracking.

The analysis of water can be a challenging task. With new technologies, however, testing can be faster, more mobile and deliver increased confidence in results.

MatrixCheck 07/30/12 15:37

Method: 51: 14558
Sample concentration: 2.25 mg/l $NH_4$-N
Standard ID: CombiCheck 10

| Sample [ml] | Standard [ml] | Target value [mg/l] | Actual value [mg/l] | |
|---|---|---|---|---|
| 1 | 0.1 | 5.25 | 5.21 | 99 % ✓ |

Back | Measure | | End

*Figure 3. CombiCheck addition solution used to check for interferences on Pharo 100 and 300 instruments*

MatrixCheck 03/20/12 16:52

Method: 51: 14558
Sample concentration: 2.25 mg/l $NH_4$-N
Standard ID: CRM 125027
Standard concentration: 50.4 mg/l $NH_4$-N

| Sample [ml] | Standard [ml] | Target value [mg/l] | Actual value [mg/l] | |
|---|---|---|---|---|
| 5 | 0.1 | 3.19 | 3.09 | 97 % ✓ |
| 5 | 0.2 | 4.10 | 3.94 | 96 % ✓ |
| 10 | 1 | 6.62 | 6.91 | 104 % ✓ |

Back | Measure | | End

*Figure 4. Use of CRM solution to check matrix effects of the sample.*