High Throughput Water and Wastewater Analysis with the Discrete Analyzer

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Introduction

Water pollution is a global issue that adversely impacts the safety of individual communities. The water used for industrial processes and subsequently discharged must be regularly monitored according to strict environmental guidelines. To maintain end-product quality and limit exposure levels, process waters need to be free of contaminants.

The US EPA's Office of Wastewater Management, authorized by the Clean Water Act (CWA), oversees programs that manage the nation's wastewater. For protection of aquatic life and human health, water quality standards programs are established to reflect the latest scientific knowledge. Wastewater and process waters are specifically analyzed in order to provide relevant information for its optimized treatment before being released into the environment.

A number of different analytes can be examined with the automated Thermo Scientific™ Gallery™ and Gallery™ Plus discrete analyzers using either colorimetric or enzymatic methods. With the addition of an optional electrochemical (ECM) unit, pH and conductivity can also be determined. Discrete cell technology offers faster, reproducible results where multiple tests can be done on a single sample without the need for additional method changeover time. All necessary analysis steps are automated, providing true walk-away time for the operator. In less than ten minutes after insertion of samples into the analyzers, results are obtained.

Measuring ranges and method detection limits are presented. Precision studies demonstrate the repeatability and reproducibility of the methods used.

Materials and Methods

Thermo Scientific system reagents are ready-to use and are available in 4 × 20 mL packages for method flexibility. Reagent volumes are optimized for the system application; from 500 to 2000 test results can typically be reported from each kit.

Reagent vials are bar-coded, and embedded in the barcode, is the material lot number and expiration date. As part of its programming, the instrument sounds an alarm when the reagent is almost finished. It can also automatically calibrate itself after the insertion of a new reagent vial. In addition to the reagent's on-board stability, the instrument has the capability to fully trace reagents and store results, including the associated calibrations and reagent lot data.

Instruments and Applications

Thermo Scientific Gallery and Gallery Plus analyzers are pre-programmed with methods that are developed to measure both high and very low concentration samples. With small modifications, these pre-programmed methods can be tailored to closely follow the Environmental Protection Agency (EPA) recommendations for end-reaction cuvette concentrations. Application parameters are adapted to test less than 300 μL of total volume. The blank measurement is usually done after the sample and one reagent are dispensed (not the chromogenic reagent) to eliminate any color interference. The calibration curves used were linear or 2nd order. The 2nd order calibration allows the use of more than 6 calibration points as required by the EPA modification rule.

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TABLE 1. A list of methods available for full performance analysis according to the 40 CFR Part 136 Method of the CWA and the related flexibility rules in CWA Regulations 40 CFR 136.6.

Kit	EPA Approved Reference
Chloride	SM 4500-Cl-E
Total Oxidized Nitrogen	SM 4500-NO3-H
Nitrite	SM 4500-NO2-B
Phosphate	SM 4500-P-E
Sulfate	ASTM International D516-07

Calibration Solutions

Thermo Scientific 1000 ppm standard solutions: Chloride (984721), Nitrite as N (984723), Nitrate as N (984725), Phosphate as P (984729), and Sulfate (984727) were used.

Measuring Ranges, Method Detection Limits and References

Measuring ranges and method detection limits for each method are shown in Table 2. Applications are designed to incorporate automated dilutions to achieve these ranges.

TABLE 2. Measuring ranges for system applications and Method Detection Limits (MDL) .

Kit	Test Limits, Low Range	Test Limits, High Range	MDL
Chloride	20–100 mg/L	100–500 mg/L	0.035 mg/L* 0.349 mg/L** 0.14 mg/L***
Total Oxidized Nitrogen	0.5–2.5 mg/L	5–25 mg/L	0.6 µg/L as Nitrogen* 11.5 µg/L as Nitrogen** 6 µg/L as Nitrogen***
Nitrite	0.5–2.5 mg/L	N/A	0.4 µg/L As Nitrogen* 1.2 µg/L as Nitrogen** 0.2 µg/L as Nitrogen**
Phosphate	0.2–1.0 mg/L as Phosphorus	2–10 mg/L as Phosphorus	0.4 µg/L as Phosphorus* 3.6 µg/L as Phosphorus** 1 µg/L as Phosphorus***
Sulfate	20–100 mg/L	100–500 mg/L	0.26 mg/L* 0.07 mg/L***

MDL was determined in two different ways:
*MDL = 3.14 x SD (blank sample, n = 7), system application
**MDL = 3 x SD + average (blank sample, 5 batches, n = 50), system application
***MDL = 3.14 x SD (low concentration sample, n = 7), application tailored to match EPA required cuvette concentrations as close as possible

Results and Discussions

Precision Studies

Results of method performance studies determined with the Gallery analyzer are shown in Table 3.

TABLE 3. Method performance data as required by EPA .

Analyte	% Recovery	Within Run SD of % Recovery	MS/MSD Recovery	MS/MSD RPD	Precision as RSD%
Chloride	93–108%	≤ 2%	96–106%	≤ 2.0	1.52%/0.67% low/high application
Total Oxidized Nitrogen	98–108%	< 2%	103–105%	≤ 1.1	1.117%
Nitrite	101–105%	≤ 0.5%	95–107%	≤ 1.1	0.14%
Phosphate	91–103%	< 2%	95–105%	≤ 2/6	1.52%
Sulfate	93–105%	≤ 1%	92–98%	≤ 1.6	0.62%

SD= Standard Deviation, MS= Matrix Spike (a spike is typically a minimum of 10% of the routine sample, more than 4x MDL),
MSD=Matrix Spike Duplicate, RPD=Relative Percent Difference (see Method description paper for details of the formula),
RSD= Relative Standard Deviation n=20

Analysis Speed

An automated operating system allows laboratories to simultaneously measure multiple analytes while reducing total analysis time and increasing efficiency. The Gallery Plus analyzer is capable of completing a test panel for 100 samples in 2 hours and 30 minutes. The panel included tests for ammonia, chloride, total oxidized nitrogen, phosphate, and sulfate from each sample. First results are available in less than 18 minutes. To perform 100 sulfate measurements only, it takes less than 25 minutes with the first result available in seven minutes.

Conclusions

Thermo Scientific Gallery and Gallery Plus automated discrete analyzers have the capability to perform several tests from a single sample without the need for extra method changeover time. Since the analyzer can automate sample dilutions, methods are designed to accurately measure large concentration ranges. Methods are repeatable, an RSD% as low as 0.14% was achieved for nitrite analysis. All methods are supported by the application proposal and performance data according to 40 CFR Part 136 Method of the CWA and the related flexibility rules in the CWA Regulations of 40 CFR 136.6.

All necessary analysis steps are automated, providing true walk-away time for the operator. Results are fully traceable and designed for ease of use. Optimized applications ensure the use of very low volumes of reagents which result in a low cost per test. Discrete cell technology offers rapid process and quality testing for water samples. The analysis of 100 samples for sulfate is completed in less than 25 minutes with the first result available in seven minutes.

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