Meeting the US Environmental Protection Agency Drinking Water Requirements with a UV/Persulfate Analyzer

Application Note

Abstract

The rapid and precise measurement of organic carbon in trace levels of water is of interest to the pharmaceutical, drinking water and environmental industries where regulations restrict the amount of discharge or contamination. Total Organic Carbon (TOC) analysis is considered an effective indicator of organic contamination in water. Since the US EPA set limits on Disinfection By-Products (DBPs) in 1979 and subsequently required testing, TOC analysis has slowly taken the forefront of accurate and rapid testing for DBPs water contamination. Municipalities and other drinking water facilities follow various recognized national and international methodologies to ensure compliance with the US EPA regulations including US EPA 415.3, 9060A, and SM5310. This study will focus on using a UV/ Persulfate analyzer to meet US EPA drinking water regulations using SM5310.

QC Requirements

A typical laboratory quality control program for TOC analysis may include the following:

- Laboratory Reagent Blank (LRB)
- Calibration Standards (STDs)
- Independent Quality Control Samples (QCS)
- Laboratory Fortified Sample Matrix (LFM)
- Laboratory Duplicates (LD)
- End Calibration Check (ECC)
- Calibration Standards analyzed as Samples

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Application Note

QC Type	Batch Requirement	Acceptance Criteria
Laboratory Reagent Blank(LRB)	At the beginning of each batch and every 10th sample	< 0.350mg/L C
Calibration Standards (STDs)	4 point calibration standards under 10mg/L C	Fit first order regression ($R^2 \geq 0.99$)
Second Source Quality Control Sample (QCS)	Every 10^{th} sample	Within acceptance range set by the manufacturer
Laboratory Fortified Sample Matrix (LFM)	1 per batch	80 – 120 % recovery
Laboratory Duplicate (LD)	1 per batch	Relative % difference (RPD) <20%
End Calibration Check (ECC)	At the end of each run. Concentration at calibration curve mid-range.	90-110% recovery
Calibration Standards Analyzed as Samples	At the end of each run.	90-110% recovery

Table 1: Laboratory QC Requirements

Calibration

A calibration curve was generated on the Fusion TOC Analyzer, using standard points of 0.30, 2.0, 4.0 and 10mg/L carbon (C), by manual dilution from a 1000mg/L C potassium hydrogen phthalate source standard. Each calibration point was analyzed in triplicate measurements. Reagent water was used as the zero point on the curve and the linearity was excellent with an R^2 value of 0.99995. (Figure 1)

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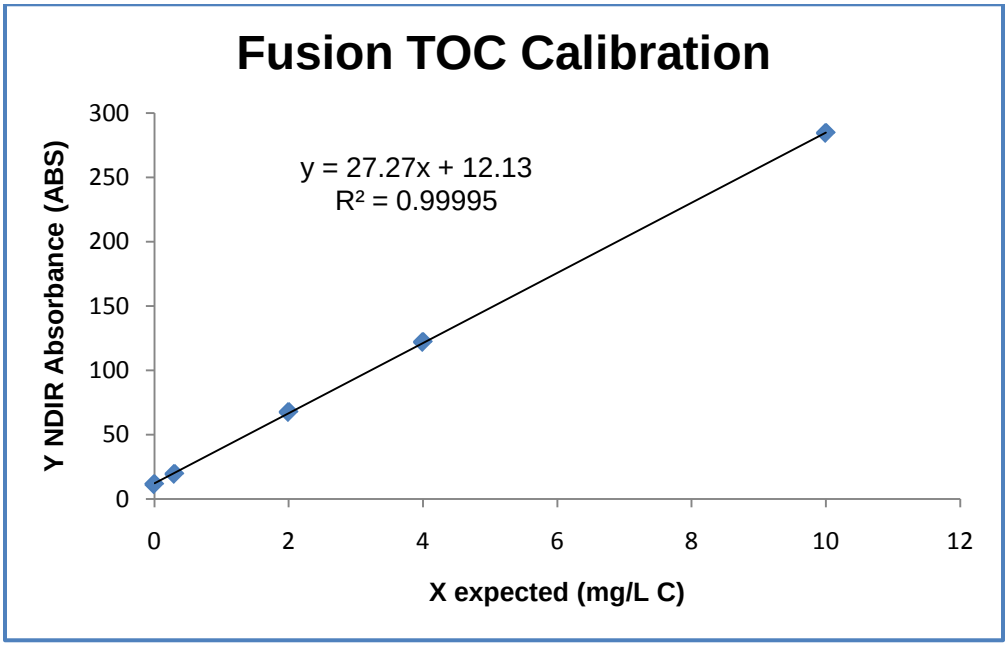

Figure 1: Multi-point Calibration Curve of the Fusion TOC Analyzer

Analyzer Method Parameters

TOC analyzers utilize a number of techniques to oxidize organic carbon in the sample to CO_2, the final form of the carbon in the sample that is detected and quantified. The Fusion TOC Analyzer utilizes UV/ Persulfate oxidation of carbon. UV/ Persulfate oxidation is highly efficient and reliable for ground water samples and waste water samples without large amount of particulates.

To detect and quantify CO_2 an NDIR detector is used. The TOC Fusion uses SPC technology in conjunction with NDIR detection. SPC technology is a process by which a single measurement of the CO_2 inside a pressurized NDIR detector is taken.
This is achieved by first oxidizing the carbon by UV-Persulfate. During the oxidation phase, the detector outlet is sealed allowing the CO_2 to be transferred inside the detector to a predetermined pressure set-point. Once the pressure setting has been achieved and all the CO_2 is pressurized within the detector, a single CO_2 measurement is taken. The amount of CO_2 detected correlates to the amount of carbon in the sample[1].

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Application Note

Fusion Method Parameters

Parameter	Value
Sample Volume	4.0mL
Dilution	1:1
Acid Volume	1.0mL
Reagent Volume	1.3mL
UV Reactor Prerinse	On
UV Reactor Prerinse Volume	6.0
Number Of UV Reactor Prerinses	3
Needle Rinse Volume	5.0mL
Vial Prime Volume	2.0mL
IC Sample Prime Volume	2.0mL
IC Sparge Rinse Volume	10.0mL
Baseline Stabilize Time	0.70 min
Detector Pressure Flow	200mL/min
Syringe Speed Waste	10
Syringe Speed Acid	7
Syringe Speed Reagent	7
Syringe Speed DI Water	7
NDIR Pressurization	45 psig
Syringe Speed Sample Dispense	7
Syringe Speed Sample Aspirate	4
Syringe Speed UV Dispense	7
Syringe Speed UV Aspirate	5
Syringe Speed IC Dispense	7
Syringe Speed IC Aspirate	5
NDIR Pressure Stabilize	0.50 min
Sample Mixing	Off
Sample Mixing Cycles	1
Sample Mixing Volume	10.0
Low Level Filter NDIR	Off

Table 3: The method parameters for analysis of ground water samples. *The use of a 4.0mL – 6.0mL sample volume is ideal for ground water sample analysis. It provides ample sample carbon mass to be deposited onto the detector and sample volume for multiple replicates.*

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Application Note

The use of a long Pre-Sparge Time and low Detector Pressure Flow allows for the analysis of samples without particulates to those with large amounts of very fine particulates.

Sample ID	Fusion TOC Results (mg/L C)
Check Standard - (1.80 mg/L C certified value)	1.9350
Blank	0.0942
Basin #7 Treated 07/07/10	2.5861
Basin #7 Treated 07/07/10 Spiked @ 4mg/L C (LFM1)	6.6756
Basin #7 Treated 08/04/10	1.9647
Basin #4 Treated 07/07/10	2.1431
Basin #4 Treated 07/07/10 Spiked @ 4 mg/L C (LFM2)	6.5952
Basin #1 Treated 07/07/10	2.2215
Basin #3 Treated 08/10/10	1.8317
Basin #4 Untreated 07/07/10	3.0078
Basin #5 Treated 08/10/10	4.8528
Basin #6 Treated 08/10/10	3.5537
Blank	0.1066
Check Standard - (4.32mg/L C certified value)	4.3788
Basin #4 Treated 07/07/10	2.0717
Basin #1 Untreated 07/07/10	3.3079
Basin #1 Untreated 08/04/10	3.2957
Basin #1 Treated 08/04/10	2.3404
Basin #2 Treated 8/10/10	3.3351
Basin #4 Treated 07/07/10 Spiked @ 4mg/L C (LFM2 Duplicate)	6.8022

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Basin #4 Untreated 08/10/10	2.0552
Check Standard (4.32mg/L C certified value)	4.5501
ECC4	3.9443
4 ppm Calibration Standard	4.2410
10 ppm Calibration Standard	9.9385

Table 4: The sample analyses of ground water samples

Method Tests	Requirement	Fusion TOC Analyzer Performance	Fusion Exceeds Specification
Laboratory Reagent Blank (LRB)	< 0.350mg/L C	0.0942mg/L C 0.1066mg/L C	✓
Calibration Standards (STD)	Fit first order regression (R^2≥0.99)	0.99995	✓
Second Source QCS	1.5205 - 2.0808mg/L C 3.9312 - 4.8816mg/L C	1.9350mg/L C 4.3788mg/L C	✓
Laboratory Fortified Sample Matrix (LFM)	80 – 120 % recovery	99 % recovery	✓
Laboratory Duplicates (LD)	RPD < 20%	3 %	✓
End Calibration Check (ECC)	90-110% recovery	99 %	✓
Calibration Standards run as Samples	90-110% recovery	106 % 99 %	✓

Table 5: Fusion TOC Analyzer Results for Laboratory QC Requirements

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Application Note

Conclusions

The Fusion TOC Analyzer utilizes UV/ Persulfate oxidation and NDIR detection with enhanced SPC technology that directly and specifically measures CO_2 gas generated from the organic carbon in the sample. This method offers the only interference-free detection of TOC. Since the sample is in a gaseous form, no direct contact is made between the sample gas and the detector – adding years of life to the detector. Automatic calibration and a self-calibrating detector make the Fusion a great choice in analyzing TOC, per SM5310.

The Fusion can effectively help your facility implement USEPA's 415.3 new regulations by providing:

- **Outstanding Precision and Accuracy**
  - ✓ +/- 1% RSD
  - ✓ Limit of detection: 0.2 ppb
- **Easy, Fast and Linear Auto-Calibration and Auto-Calibration Verifications**
- **Default Method Parameters**
  - ✓ Can be used to meet most laboratory analysis needs without modification
- **NDIR Detection coupled with SPC Technology**
  - ✓ Provides reliable, rugged and interference free CO_2 detection for TOC analysis

References

1. TOC Fusion Manual
2. Standard Methods for the Examination of Water and Wastewater, 19th Edition 1995
3. Potter, B. and Wimsatt, J. "USEPA Method 415.3 – Determination of TOC and SUVA at 254nm in Source Water and Drinking Water." National Exposure Research Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, June 2003.

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Application Note

Abbreviations

TOC	Total Organic Carbon
SM5310	Standard Methods 5310
US EPA	United States Environmental Protection Agency
NDIR	Non-Dispersive Infrared
ABS	Absorbance
SPC	Static Pressure Concentration
DBPs	Disinfection By-Products
UV	Ultra Violet
IC	Inorganic Carbon
min	Minutes
mL	Milliliters
HTC	High Temperature Combustion
CO_2	Carbon Dioxide
mg/L	parts per million
C	Carbon
QCS	Quality Control Sample
ECC	End Calibration Check
LD	Laboratory Duplicates
LFM	Laboratory Fortified Sample Matrix
STDs	Calibration Standards
LRB	Laboratory Reagent Blank

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