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Application News

No. TOC-005

Total Organic Carbon Analysis

TOC Analysis for Standard Methods 5310B: High-Temperature Combustion Method

■ Introduction

Total Organic Carbon (TOC) is a rapid method that analyzes for organic carbon and expresses the result as the amount of carbon found. It is a non-specific method unable to distinguish between various organic species and only indicates that organic carbon compounds are present. Organic carbon analyzers operate by the determination of the amount of total carbon present in a sample aliquot. Total carbon consists of inorganic and organic carbon. The inorganic carbon, present as carbonate or bicarbonate ions, must be removed or quantified prior to the analysis of organic carbon. Once the inorganic carbon is removed, subsequent analysis of the sample aliquot assumes that all carbon remaining is organic.

■ Discussion

Methodology used to remove inorganic carbon relies on acidification that converts all bicarbonate and carbonate ions to carbon dioxide that is then purged out of the sample using CO_2 free gas. If quantification of inorganic carbon is desired, it is purged into a detector, otherwise, it is vented to atmosphere. Once the inorganic carbon is removed, the remaining organic carbon is oxidized to carbon dioxide that is purged by CO_2 free gas into the detector. TOC analysis is applicable to both drinking water and wastewater.

■ TOC in Drinking Water

Control of disinfection byproduct formation (DBP) is accomplished by minimizing DBP precursors. Chlorine reacts with naturally occurring organic matter to form Trihalomethanes and Haloacetic acids. Organic matter in drinking water sources and finished drinking water is measured as Total Organic Carbon (TOC). Water systems that use traditional filtration methods for removal of organic matter are required to remove certain percentages of TOC depending upon the concentration of TOC and alkalinity in the source water.

This rule only applies to systems treating surface water. In essence, a system measures the source water and finished water once monthly for TOC and calculates the % TOC removal. Table 1 shows the percent TOC removal requirements as a function of TOC and alkalinity concentration.

■ TOC in wastewater

TOC can be correlated to the Biochemical Oxygen Demand (CBOD) and to the Chemical Oxygen Demand (COD). CFR 40 Part 133.104 (b) allows substitution of TOC for BOD or COD provided long-term (usually once/month) comparison data is collected. A factor generated from the data enables calculation of BOD from the TOC result. Using TOC instead of BOD is especially useful in tertiary effluents and process control.

TOC concentration for drinking water can range from less than 100 ppb to more than 25 ppm, and wastewater may contain much higher levels of organic compounds, *i.e.* >100 ppm C. Thus, it becomes important for a TOC analyzer to be capable of precise measurement for both the low-end (ppb) and high-end (ppm), to be capable of performing auto-dilutions for over-range samples, and to be capable of utilizing multiple calibration curves and selecting the correct curve to use for each sample.

Source Water TOC (mg/L)	Source Water Alkalinity (mg/L as $CaCO_3$)		
	0 – 60	60 – 120	Greater than 120
2.0 – 4.0	35.0 %	25.0 %	15.0 %
4.0 – 8.0	45.0 %	35.0 %	25.0 %
Greater than 8.0	50.0 %	40.0 %	30.0 %

Table 1: TOC removal requirements

■ Procedure

Standard Methods 5310B describes the procedure and requirements for analysis by high-temperature catalytic oxidation (HTCO) with non-dispersive infrared (NDIR) detection. The sample is homogenized and diluted as necessary, and a small amount is injected into a heated reaction chamber packed with a platinum catalyst. Water vaporizes, and organic carbon compounds are oxidized to CO_2 and H_2O. The CO_2 from the oxidation of organic and inorganic carbon are measured by the NDIR.

■ Experimental

A 1000 ppm C stock standard was prepped by dissolving 2.125 g of potassium hydrogen phthalate (KHP) in 1 L of DI H_2O. Two high standards were prepared by diluting this stock standard to concentrations of 1 ppm and 100 ppm, and two calibration curves were generated using auto-dilution of these standards. These curves are presented in Figures 1 and 2, with a summary provided in Tables 2 and 3. Instrument parameters are shown in Table 4.

Calibration Standard (ppm)	Std. Dev.	RSD
0	0.09218	7.82%
1	0.03950	1.17%
2.5	0.07136	1.00%
5	0.13870	1.01%
10	0.47930	1.79%

r	0.9998
Det. Limit	0.03 ppm

Table 2: Calibration curve summary for "Drinking Water" curve

Calibration Standard (ppm)	Std. Dev.	RSD
0	0.09164	10.94%
10	0.49500	0.19%
25	0.48790	0.74%
50	2.12100	1.61%
100	3.25300	1.21%

r	0.9999
Det. Limit	0.04 ppm

Table 3: Calibration curve summary for "Wastewater" curve

Parameter	Value
Analysis Method	NPOC (non-purgeable organic carbon)
Furnace Temperature	680 °C
Injection Volume	50 µL
Acid Addition	2%
No. of Injections	3/5

Table 4: Instrument parameters

Additionally, a 5 ppm check standard (for drinking water) and 50 ppm check standard (for wastewater) were both analyzed after calibration. The results are summarized in Table 5:

Check Standard	Result	Recovery	Std. Dev.	RSD
5 ppm	4.960	100.8%	0.11780	0.854%
50 ppm	49.39	101.2%	1.98700	1.505%

Table 5: Analysis of check standards

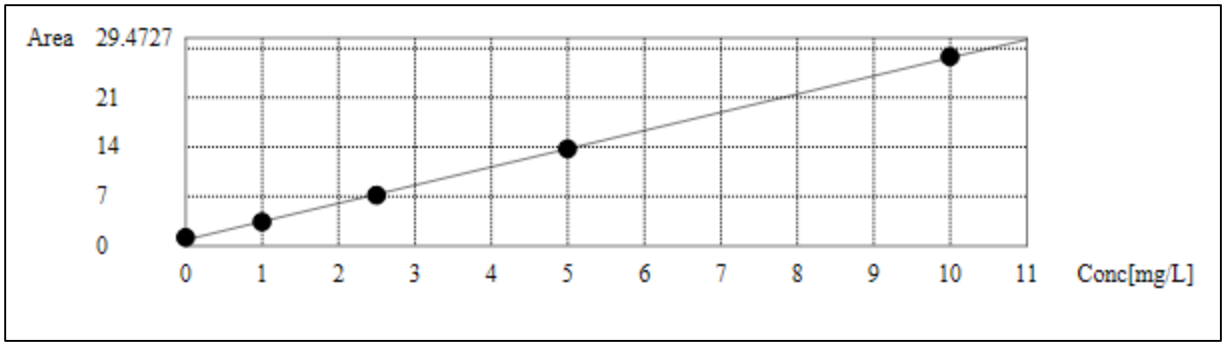

Figure 1: "Drinking Water" Calibration Curve

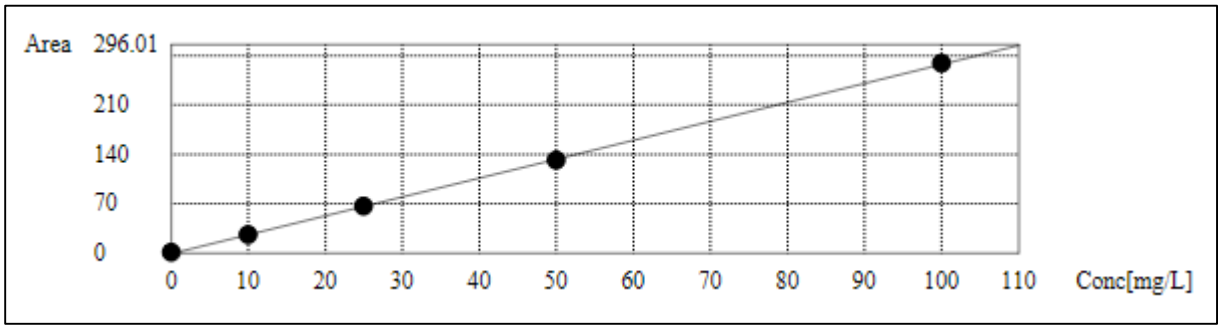

Figure 2: "Wastewater" Calibration Curve

■ Conclusion

The Shimadzu TOC-L total organic carbon analyzer is ideal for TOC measurement by Standard Methods 5310B. The TOC-L, with a 680 °C furnace and platinum catalyst, utilizes high-temperature catalytic oxidation to completely oxidize organic carbon.

Moreover, the ability to auto-generate a calibration curve from a single standard, coupled with the capability to auto-dilute and re-inject over-range samples, ensures that one method can analyze all samples.

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