

Challenge Standard in Drinking Water for Total Organic Carbon (EN 1484 and ISO 8245): Combustion Oxidation Mode

Introduction

EN 1484, a standard created by the European Committee for Standardization, and ISO 8245, a standard created by the International Organization for Standardization, are two of the worldwide standards for water monitoring that utilize combustion oxidation for the determination of total carbon, total organic carbon and total inorganic carbon in a variety of source waters. The standardization of water monitoring greatly improves water quality in drinking water, groundwater, surface water, sea water applications, and waste water monitoring.

Because of the variety of organic materials that can occur in source water, strong oxidation is required to achieve maximum CO recovery. According to EN 1484¹ and ISO 8245², historical data suggest that multiple oxidation techniques can potentially achieve the desired recovery. OI Analytical addresses this issue of multiple options through the use of both heated persulfate and combustion. This application note utilizes the Model 1080 (Figure 1) in determining recovery of copper phthalocyanine-tetrasulfonic acid, tetrasodium salt as a test material for EN 1484 and ISO 8245 compliance. Analyzing samples by combustion oxidation is a very thorough process that results in consistently high sample recoveries.

Background

Based on the theoretical molecular weight of the tetrasodium salt as stated in the two standards (EN 1484 and ISO 8245), the theoretical percent carbon is 39.049%; a 100-ppm C solution can be prepared using 0.256 g/L of tetrasodium salt. However, the Certificate of Analysis (COA) for the copper reagent's lot number states that the measured percent carbon is 53.48%. The acceptable carbon range for this product is 21.4–56.6% (as stated by the supplier). Using the measured percent carbon value from the COA, 0.187 g/L of copper reagent was used to prepare a 100-ppm C solution.



Figure 1. 1080 TOC Analyzer

Keywords

Combustion Oxidation
EN 1484
TOC
Total Organic Carbon

Methodology

Although the Model 1080 supports several different oxidation methods, the non-purgeable organic carbon (NPOC)-Only method was used to analyze copper phthalocyanine-tetrasulfonic acid, tetrasodium salt. In the NPOC Only mode, the sample is aspirated, transferred into the total inorganic carbon (TIC) chamber, acidified using 2 N hydrochloric acid, heated to 70 °C, and sparged.

During acidification, carbonate and bicarbonate ions convert to CO₂. This carbon dioxide and any other dissolved CO₂ are removed from the sample during sparging and vented to the atmosphere via the select manifold. After sparging these compounds from the sample, an aliquot is reclaimed by the syringe and injected into the total carbon (TC) furnace onto a quartz bed heated to 680 °C. The sample is consequently converted into a gaseous phase and forced through a layer of catalyst, ensuring the complete conversion of all carbon-containing compounds to CO₂.

The solid-state non-dispersive infrared (SS-NDIR) detector then measures the resulting CO₂. The SS-NDIR detector response is uninfluenced by changes in sample pH or temperature and avoids potential interferences from gases such as chlorine, chlorine dioxide, sulfur dioxide, and others. When the total organic carbon (TOC) in the sample has been calculated, sample results are reported as ppm or ppb TOC. By initially removing the TIC, replicates can be injected and processed very quickly, expediting the analysis process.

Results and Discussion

All TOC samples were run on a Model 1080 TOC Analyzer. The instrument was calibrated at 0, 1.0, 5.0, 10.0, and 25.0 ppm C using potassium hydrogen phthalate (KHP). The calibration data are listed in Table 1; method details are listed in Table 2.

Sample	Repetitions	Average Area (counts)	% RSD	Calibration R ²	Response Factor (µg C/k-cnt)
0 ppm C KHP	3	2,900	6.41	0.9997	0.1496
1 ppm C KHP	3	13,080	1.59		
5 ppm C KHP	3	65,442	1.25		
10 ppm C KHP	3	125,450	0.58		
25 ppm C KHP	3	303,055	0.64		

Table 1. Calibration Data

Mode	Sample Volume (mL)	Acid Volume (mL)	System Pressure (psi)
NPOC Only	1.0	0.1	20

Table 2. Method Details

As illustrated in Table 3, the Model 1080 achieved recoveries in excess of 98% operating in the combustion mode at both 10 and 100 ppm C levels based on actual yields.

These percentages clearly indicate the excellent oxidation capability provided by the combustion oxidation technique. The recovery values also show excellent performance when compared to the wet oxidation technique.

Theoretical yield is based on the stoichiometric formula used to synthesize the compound. In most cases, the theoretical yield is therefore higher than the actual yield. The actual yield, as discussed in "Background" on page 1, was obtained from the manufacturer and is an indication of the company's synthesis efficiency.

Note that although the curve was not optimized for measurements over 45 µg C, the inherent linearity of the instrument is shown, even at 180 µg C and beyond based on recovery.

Sample	Repetitions	Average Area (counts)	% RSD	Actual Recovery	Theoretical Recovery
10 ppm C KHP	3	126,304	0.87	101.80%	135.28%
10 ppm C Theoretical	3	170,865	0.77		
10 ppm C Actual	3	128,579	0.69		
100 ppm C KHP	3	1,177,078	0.57	98.33%	134.38%
100 ppm C Theoretical	3	1,581,749	1.50		
100 ppm C Actual	3	1,157,474	1.15		

Table 3. Recovery Results

Conclusion

The COA should always be consulted before using copper phthalocyanine-tetrasodium salt to prepare standards for operational checks. Reagent preparation should always be based on the percent carbon stated on the COA rather than on the theoretical percent carbon of copper phthalocyanine-tetrasodium salt.

The flexibility of the Model 1080 TOC Analyzer allows for the optimization of several difficult applications such as copper phthalocyanine-tetrasulfonic acid, tetrasodium salt.

References

1. EN 1484:1997. *Water analysis. Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*.
2. ISO 8245. *Water quality - Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)*; 1999-03-01.
3. Electronic Records; Electronic Signatures; Final Rule. *Code of Federal Regulations*, Part 11, Title 21; 1997.



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