TOC Analysis of 3% Hydrogen Peroxide (H2O2) Using the Teledyne Tekmar Lotix TOC Analyzer

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Abstract

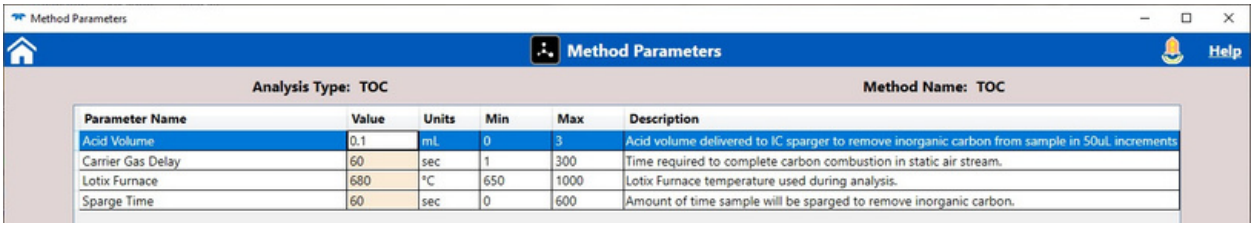
Hydrogen peroxide (H2O2) is used in numerous industries and has a variety of uses including sterilization, wastewater treatment, bleaching and as a propellant. The analysis of hydrogen peroxide for total organic carbon (TOC) content is a quality control (QC) test used to ensure purity. This application note will demonstrate that the Teledyne Tekmar Lotix TOC analyzer is robust enough to analyze the corrosive and acidic 3% hydrogen peroxide matrix and determine TOC content with analytical accuracy and precision.

Experimental Instrument Conditions

Method Parameters

Due to the ~6 pH of the 3% hydrogen peroxide matrix, the method's acid volume parameter was reduced to 0.1 mL. The sparge time was left unchanged at 60 seconds to remove any inorganic carbon in the sample. Additional method parameters to extend catalyst lifespan are shown in Figure 1.

Figure 1 TOC TekLink Method Parameters.



Standard and Sample Preparation

1000 ppmC Stock Standard Preparation

1. A weigh boat was tared on an analytical grade scale.
2. 1.0625 g of potassium hydrogen phthalate (KHP) was weighed to the nearest 0.0001 g in the tared weigh boat. The KHP was transferred to a 500 mL volumetric flask and brought to volume with lab grade reagent water (LRW).
3. The KHP solution was then thoroughly mixed using a stir bar until it was completely dissolved.
4. The weigh boat was then reweighed and subtracted from the original recorded weight to account for the residual KHP that was not transferred.
5. The actual ppmC of the stock standard was calculated using the formula below:

$$\frac{\text{Quantity of KHP transferred to flask}}{1.0625\ \text{g}} \times 1000\ \text{ppmC} = \text{Actual ppmC of stock standard}$$

Calibration Standard Preparation

Serial dilution of the 1000 ppmC stock standard was used to create calibration standards for a five-point calibration curve. Using a "Grade A" volumetric pipette, the amounts of stock standard shown in Table I were pipetted into 1000 mL volumetric flasks, then brought to volume with LRW to create the concentrations shown. The five calibration standard concentrations were then transferred to 40 mL vials and loaded into the analyzer's sample conveyor. Each calibration standard was analyzed in triplicate.

Table I Calibration Standard Concentrations for a Five-Point Calibration Curve		
Amount of 1000 ppmC Stock Std (mL)	Brought to Volume (mL)	Final Concentration (ppmC)
0	1000	0.0
1	1000	1.0
5	1000	5.0
10	1000	10.0
25	1000	25.0

Note: For accurate results, all glassware must be properly cleaned and free of any of residual carbon. The lab grade reagent water must contain minimal carbon levels.

Sample Preparation

A typical, over-the-counter 3% H2O2 topical solution was used for samples (shown to the right). 40 mL VOA vials were filled with sample, and loaded in the analyzer's sample conveyor. No sample preparation was performed. Each sample was analyzed in triplicate.

5 ppmC, 3% H2O2 Matrix-Matched Quality Control (QC) Check Standard Preparation

A matrix-matched QC check standard was prepared to ensure the H2O2 sample matrix was not affecting the accuracy of results. 10 mL of 30% ACS grade H2O2 was pipetted into a 100 mL volumetric flask to which 20 mL of the 25 ppmC calibration standard was then added. This resulted in a final standard concentration of 5 ppmC. The solution was then brought to volume with DI water, making the solution 3% H2O2. The solution was then shaken vigorously to homogenize.

5 ppmC Quality Control (QC) Check Standard

The 5 ppmC calibration standard solution was used as a QC check standard and analyzed after every five samples.

Results

Calibration Results

The five-point calibration curve yielded an r2 value of 0.99989 and the %RSD of calibration standard triplicates was less than 1%, with the exception of the 0 ppmC calibration standard (Figure 2). The r2 value easily exceeded the generally accepted linearity of >0.999, while the %RSD was significantly lower than the commonly accepted 5% (Figure 3).

Figure 2 Calibration Standard %RSD Results.

Schedule Summary

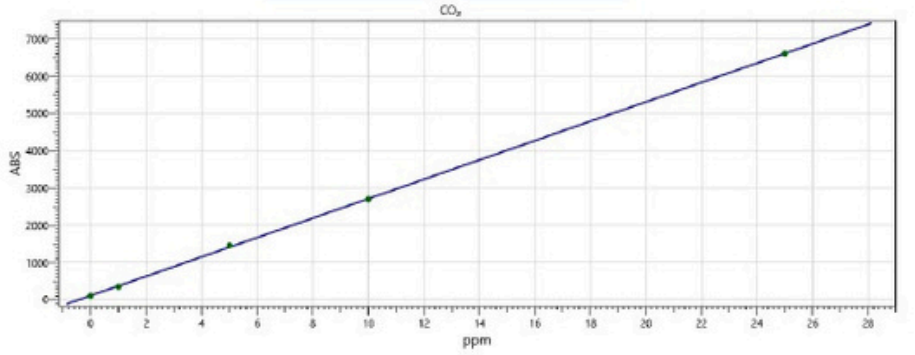
Pos	Sample Type	ID	ABS	Std Dev	%RSD
R	Rinse (TOC)		67.953	7.105	10.455
1	Cal Std (TOC)	0.0000 ppmC	101.695	11.286	11.098
2	Cal Std (TOC)	1.0000 ppmC	346.506	3.093	0.893
3	Cal Std (TOC)	5.0000 ppmC	1455.830	10.328	0.709
4	Cal Std (TOC)	10.0000 ppmC	2694.884	24.321	0.903
5	Cal Std (TOC)	25.0000 ppmC	6608.235	62.461	0.945

Figure 3 Coefficient of Determination (r2) and Five-Point Calibration Curve Graph.

Linear Curve: y = 260.081x + 108.763
Coefficient of Determination (r^2): 0.99989

Standard Results

Actual ppmC	ABS	Measured ppmC	When
0.00	101.695	-0.027	6/6/2019 3:54:17 PM
1.00	346.506	0.914	6/6/2019 4:11:25 PM
5.00	1455.830	5.179	6/6/2019 4:26:32 PM
10.00	2694.884	9.944	6/6/2019 4:41:49 PM
25.00	6608.235	24.990	6/6/2019 4:57:48 PM



Sample and QC Check Standard Results

A 5 ppmC QC check standard was run after every five samples to ensure calibration validity. All QC check standard results met the generally accepted <5 %RSD acceptance criteria (15% or 4.25-5.75 ppmC of the 5 ppmC check standard) (Figure 4).

Figure 4 QC Check Standards Passing Acceptance Criteria.

Schedule Summary

Pos	Sample Type	ID	ppm	Std Dev	%RSD
R	Rinse (TOC)	Rinse	92.583 (ABS)	35.030	37.836
1	Blank (TOC)	Blank	128.464 (ABS)	44.254	34.449
2	Chk Std (TOC)	5.0000 ppmC	4.900 Passed	0.065	1.334
3	Sample (TOC)	3% Hydrogen Peroxide	11.330	0.220	1.940
R	Rinse (TOC)	Rinse	61.203 (ABS)	23.731	38.775
4	Sample (TOC)	3% Hydrogen Peroxide	11.211	0.161	1.434
R	Rinse (TOC)	Rinse	63.408 (ABS)	21.684	34.198
5	Sample (TOC)	3% Hydrogen Peroxide	11.223	0.351	3.124
R	Rinse (TOC)	Rinse	58.751 (ABS)	24.042	40.922
6	Sample (TOC)	3% Hydrogen Peroxide	11.143	0.179	1.606
R	Rinse (TOC)	Rinse	74.384 (ABS)	45.177	60.735
7	Sample (TOC)	3% Hydrogen Peroxide	11.123	0.223	2.009
R	Rinse (TOC)	Rinse	62.470 (ABS)	32.427	51.908
8	Chk Std (TOC)	5.0000 ppmC	5.241 Passed	0.060	1.140

The sample result data was compiled for the 10, 3% H2O2 samples run in triplicate and is shown in Table II. Results were very good with a %RSD less than 5% and averaged 11 ppmC, which was well within the calibration curve.

Table II 3% H2O2 Sample Results		
Sample	Calculated ppmC	%RSD (n=3)
Sample1	11.330	1.940
Sample2	11.211	1.434
Sample3	11.223	3.124
Sample4	11.143	1.606
Sample5	11.123	2.009
Sample6	10.687	1.461
Sample7	10.816	1.681
Sample8	10.812	1.766
Sample9	10.728	1.372
Sample10	10.929	0.870
AVG	11.00	1.73
ST Dev	0.23	1.70
%RSD	2.11	1.74

5 ppmC, 3% H2O2 Matrix-Matched QC Check Standard Results

Results for the 3% H2O2 matrix-matched QC check standard spiked with 5 ppmC are shown in Table III and Figure 5. The results were well within 15% of the actual value, indicating that the matrix had negligible effect on the accuracy of the analytical results.

Table III Matrix-Matched 3% H2O2 QC Check Standard Results			
Check Standard	%RSD (Target <%5)	Calculated ppmC	15% Acceptance Criteria (ppmC) 4.25 - 5.75
3% H2O2 with 5 ppmC	1.941	5.60	

Figure 5 5 ppmC, 3% Hydrogen Peroxide Matrix-Matched Check Standard Results.

9	Sample (TOC)	5ppmC with 3% H2O2 Matrix check	5.600	0.109	1.941

Conclusion

The Teledyne Tekmar Lotix TOC analyzer is an easy to use, fast and accurate instrument, well-suited for a variety of difficult sample matrices, including 3% hydrogen peroxide (H2O2). The instrument exhibited excellent accuracy and reproducibility with standards and samples achieving acceptance criteria of 5% RSD and 15% accuracy. The TOC TekLink software made method customization and schedule creation simple and straight-forward.

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