TOC Analysis of 30% Hydrogen Peroxide (H2O2) Using the Teledyne Tekmar Lotix TOC Analyzer

Joy Osborne, TOC Technical Product Manager; Teledyne Tekmar

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 30% hydrogen peroxide matrix and determine TOC content with analytical accuracy and precision.

Experimental Instrument Conditions

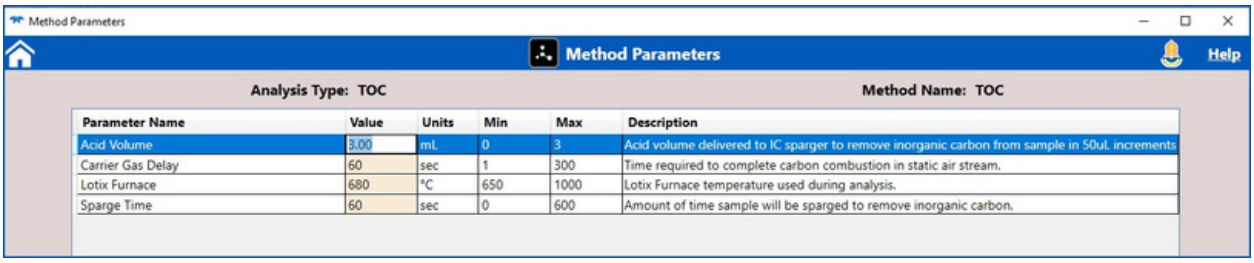
Instrument Setup

The acid reagent bottle was filled with DI water (rather than acid) to act as a diluent.

Method Parameters

For this application, the acid bottle was filled with DI water and the method's acid volume parameter increased to 3.0 mL to act as a diluent for the ~1 pH, 30% hydrogen peroxide (a harsh sample matrix). 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.

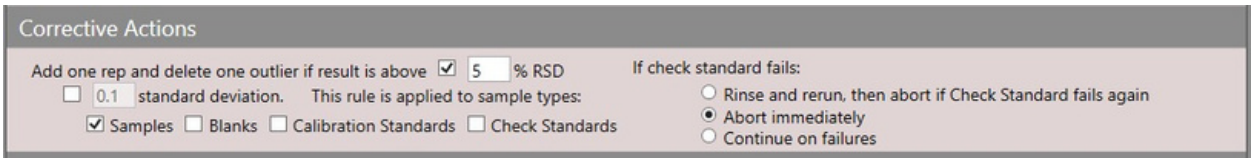
Figure 1 Lotix TOC TekLink Method Parameters.



Configuration Setup

Due to the uncertainty related to the sample matrix, the "Add one rep and delete one outlier if result is above 5% RSD" corrective action was selected in the Lotix TOC TekLink software.

Figure 2 Lotix TOC TekLink Software Corrective Actions



Sales/Support : 800-874-2004 •Main : 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

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 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)
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

An ACS grade 30% H2O2 solution was used to prepare 70 samples. 40 mL VOA vials were filled with sample and loaded in the analyzer's 30 vial sample conveyor. No sample preparation was performed. Each sample was analyzed in triplicate for a total of 210 replicates. Rinses were run between each sample to extend catalyst life.

10 ppmC Quality Control (QC) Check Standard

The 10 ppmC calibration standard solution was used as a QC check standard and analyzed after every ten samples.

Sales/Support: 800-874-2004 ·Main: 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

Results

Calibration Results

The calibration curve yielded an r2 value of 0.99912 and the %RSD of calibration standard triplicates was less than 5%. 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 and Figure 4).

Figure 3 Calibration Standard %RSD Results.

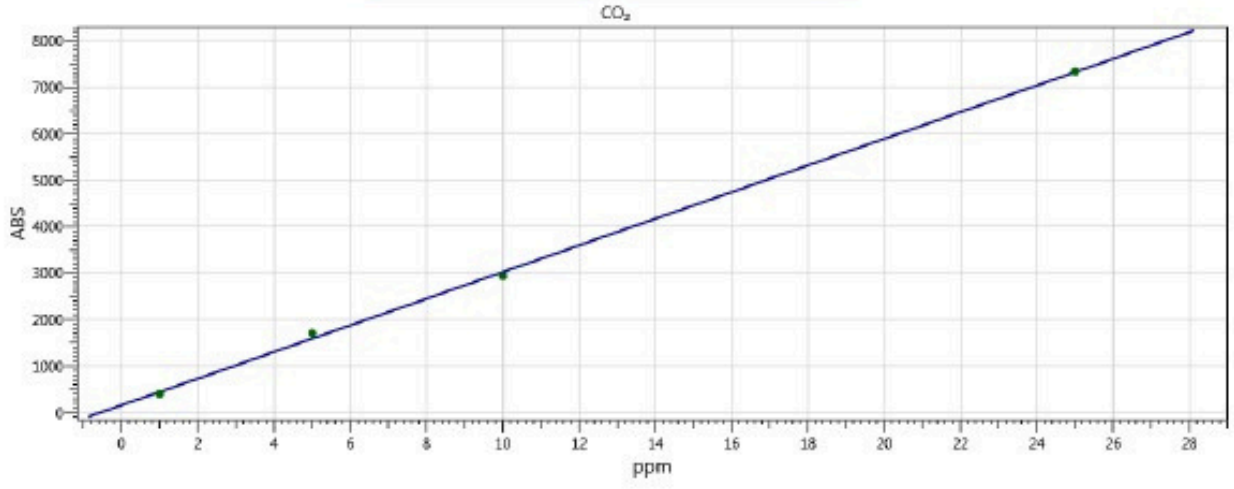
Pos	Sample Type	ID	ABS	Std Dev	%RSD
1	Cal Std (TOC)	0.0000 ppmC	294.236	4.404	1.497
2	Cal Std (TOC)	1.0000 ppmC	396.883	3.878	0.977
3	Cal Std (TOC)	5.0000 ppmC	1704.442	33.186	1.947
4	Cal Std (TOC)	10.0000 ppmC	2931.231	94.235	3.215
5	Cal Std (TOC)	25.0000 ppmC	7337.490	190.176	2.592

Figure 4 Coefficient of Determination (r2) and Five-Point Calibration Curve Graph.

Linear Curve: y = 286.850x + 152.298
Coefficient of Determination (r): 0.99912

Standard Results

Actual ppmC	ABS	Measured ppmC	When
1.00	396.883	0.853	6/19/2019 5:08:10 PM
5.00	1704.442	5.411	6/19/2019 5:23:12 PM
10.00	2931.231	9.688	6/19/2019 5:38:41 PM
25.00	7337.490	25.049	6/19/2019 5:54:52 PM



Sales/Support: 800-874-2004 ·Main: 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

Sample and QC Check Standard Results

A total of 70 samples were run to validate calibration longevity and catalyst integrity (Table II). A 10 ppmC QC check standard was run after every ten samples. All QC check standard results met the generally accepted <5 %RSD acceptance criteria (20% or 8-12 ppmC of the 10 ppmC check standard) as shown in Table III.

Table II Example Sample Schedule to Prevent Catalyst Decay (10 Samples)

Position	Sample Type (Method)	Sample ID
R	Rinse (TOC)	Rinse
1	Chk Std (TOC)	10.000 ppmC
2	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
3	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
4	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
5	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
6	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
7	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
8	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
9	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
10	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
11	Sample (TOC)	30%H2O2
R	Rinse (TOC)	Rinse
12	Chk Std (TOC)	10.000 ppmC

Sales/Support: 800-874-2004 ·Main: 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com

The results data was compiled from 70 samples (210 replicates) of 30% H2O2 samples run in triplicate and shown in Table III. Sample results were very good with a %RSD less than 5%. The software's "Add one rep and delete one outlier if result is above 5% RSD" corrective action was needed, but was used less than five times. Measured concentrations averaged 5.683 ppmC, which was well within the calibration curve.

Table III 30% Hydrogen Peroxide Sample Results (10 Samples/2 Check Standards Per Day)

Day	Avg. ppm	Chk Std (Bracketing 10 Samples)
1	5.850	9.710 (Passed)
		9.519 (Passed)
2	5.535	9.634 (Passed)
		9.449 (Passed)
3	5.724	9.771 (Passed)
		9.323 (Passed)
4	5.596	9.959 (Passed)
		9.323 (Passed)
5	5.628	9.583 (Passed)
		9.262 (Passed)
6	5.794	9.542 (Passed)
		9.236 (Passed)
7	5.652	9.339 (Passed)
		9.276 (Passed)
AVG	5.683	9.494
STDEV	0.209	0.218
%RSD	3.789	2.304

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 30% 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. The software's corrective action feature was used and successfully managed this difficult sample matrix's propensity for high %RSD.

Sales/Support: 800-874-2004 ·Main: 513-229-7000
4736 Socialville Foster Rd., Mason, OH 45040
www.teledynetekmar.com