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The Effects of Furnace Configuration Using The Torch High Temperature Combustion Analyzer

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Abstract

In the evaluation of industrial process waters and wastewaters, of particular interest is organic carbon contamination from either microbial or synthetic derivatives, due to their possible environmental impact. High temperature combustion total organic carbon analyzers provide the most efficient way to measure organic carbon in these waters. The furnace parameters for high temperature combustion analyzers dictate the oxidation effectiveness of the sample carbon, which in turn affects the total organic carbon results. This presentation will cover the analytical effects of temperature, catalyst type and combustion tube configuration on total organic carbon analysis.

Introduction

Traditionally, High Temperature Combustion (HTC) analyzers have relied upon furnace temperature and catalyst function to demonstrate the best performance characteristics for the analyzer. Recent advancements have brought pressure, whether low or high, into the discussion of analyzer performance. Regardless of temperature, catalyst type, or pressure setting, the combustion tube has been the center point of all three instrument parameters. As such, its dimensions and dynamics are critical to the analyzer's optimal performance. The combustion tube used in this analysis was a quartz tube packed with alumina based catalyst layered in between two beds of quartz wool. The final strata were a platinum screen held in place by a solid sleeve. The Torch HTC Analyzer, developed by Teledyne Tekmar, uses a new catalyst that is better suited for difficult sample matrices. It is controlled by the user-friendly TekLinkTM software that has predefined instrument methods and numerous programmable parameters. This enables the user to begin sample analysis without delay by employing the preset methods, without losing the flexibility to design custom methods.

Torch Calibration Matrix			
0.50mL Sample Injection Volume			
	680oC	720oC	850oC
50 psig	FC 1	FC 2	FC 3
30 psig	FC 4	FC 5	FC 6

Table 1: Torch calibration analysis comparison matrix.
(FC -- Furnace Configuration)

Experimental - Instrument Conditions

In this study, the Torch HTC analyzer was configured with three different temperature settings, two different pressure settings and calibrated according to the matrix in Table 1. The general furnace method parameters used to generate all calibration curves are depicted in Table 2. The resulting curves were then compared to show the effects of changing instrument parameters on the analysis. A sample injection volume of 0.5mL was kept constant throughout the experiment.

Torch Analysis Overview

The following steps give a basic description of the TOC Torch methodology for direct TOC analysis:

1. Autosampler needle rinse. The syringe pulls deionized water and flushes the autosampler needle into a rinse station at the autosampler.

2. Acid addition into the Inorganic Carbon (IC) Sparger Chamber. The syringe pulls sample and dispenses it into the IC Sparger Chamber, followed by the addition of a 20% acid solution to bring the solution to a pH of 2.0 or less.

3. IC is sparged from the sample. Carbon dioxide (CO2) free air or oxygen is sparged through the sample in the IC Sparger Chamber to remove the IC in the sample. For TOC analysis the IC is vented to the atmosphere. For IC analysis it is sent to the detector.

4. Furnace Combustion. The sample is added drop-wise into the combustion tube, containing the catalyst, located inside of the furnace. The heat from the furnace oxidizes the carbon into the form of CO2. This oxidation process is promoted by the catalyst. The CO2 is sent through the sample pathway into the NDIR detector by the CO2 free air that is metered by a Mass Flow Controller (MFC).

5. Water and halide removal. Before the CO2 gas reaches the detector, it is sent through two water removal systems, a mist trap and permeation dryer. It is then sent to the halide scrubber to remove any chloride interference.

6. NDIR Detection. A valve located at the outlet of the detector prevents the escape of CO2 from the detector. Once all of the CO2 is inside the detector, a single measurement is made to determine the amount of CO2 gas in the detector cell. The reading correlates directly to the concentration of the carbon contribution from the sample.

7. Rinse. The syringe pulls in reagent water through the 7-Port Valve and rinses the IC Chamber. This rinse water is then removed prior to the next sample analysis.

TekLinkTM software has programmable parameter settings within the method. The gas flow rates are programmable and metered by the MFC. The use of a MFC to meter the gas flow rates not only allows instantaneous control of system flow but also the delay of gas flow to the furnace after sample introduction. The flexibility of flow control permits the end user to define the amount of time the sample is in contact with the catalyst. This combination of heat, pressure and flow control provides superior precision over flow through cell instruments.

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Parameter	Value
Sample Volume	0.50mL
Water Chase Volume	1.00mL 1:1
Dilution	1 On
Number of Injection Line Rinses	0.50mL
Injection Line Rinse	0.50mL
Injection Line Rinse Volume	200mL/min
Acid Volume	0.40 min
IC Sparge Flow	0.50 min Off
Carrier Gas Delay Time	500mL/min
IC Sparge Time	1.00 min
TN Detector Module Enable	500mL/min
Detector Sweep Flow	
Furnace Sweep Time	
System Flow	

Parameter	Value
Mixer Magnet Enable	Off
Sparge In Vial Enable	Off
Needle Rinse Volume	2.5mL
Vial Prime Volume	2.0mL
IC Sample Prime Volume	2.0mL
Baseline Stabilize Time	0.75 min
Detector Pressure Flow	175mL/min
Syringe Speed Waste	10
Syringe Speed Acid	7
Syringe Speed DI Water	7
NDIR Pressurization	50 psig
Syringe Speed Sample Dispense	7
Syringe Speed Sample Aspirate	7
Syringe Speed IC Dispense	7
NDIR Pressure Stabilize	7
Syringe Speed IC Aspirate	5
NDIR Pressure Stabilize	0.60 min
Sample Mixing	Off
Sample Mixing Cycles	1
Sample Mixing Volume	2.5
Syringe Speed Furnace Dispense	3
Syringe Speed Furnace Aspirate	5
Furnace Temperature	680oC

Table 2: Torch instrument method parameters. Parameters highlighted in red were changed during the experiment

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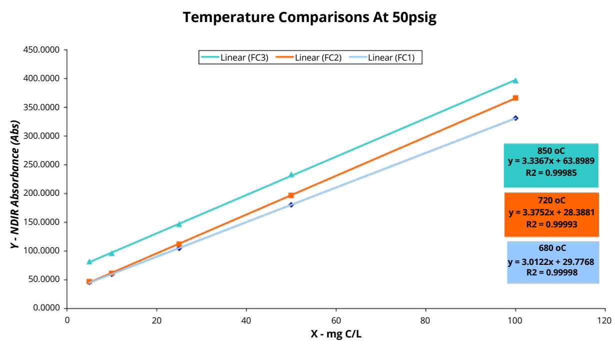
Temperature Comparisons At 50psig
Linear (FC3)
Linear (FC2)
Linear (FC1)
850 oC
y = 3.3367x + 63.8989
R2 = 0.99985
720 oC
y = 3.3752x + 28.3881
R2 = 0.99993
680 oC
y = 3.0122x + 29.7768
R2 = 0.99998
Y - NDIR Absorbance (Abs)
X - mg C/L
0.0000
50.0000
100.0000
150.0000
200.0000
250.0000
300.0000
350.0000
400.0000
450.0000
0
20
40
60
80
100
120

Figure 1: Calibrations compared at three furnace temperature settings with a pressure setting of 50 psig.

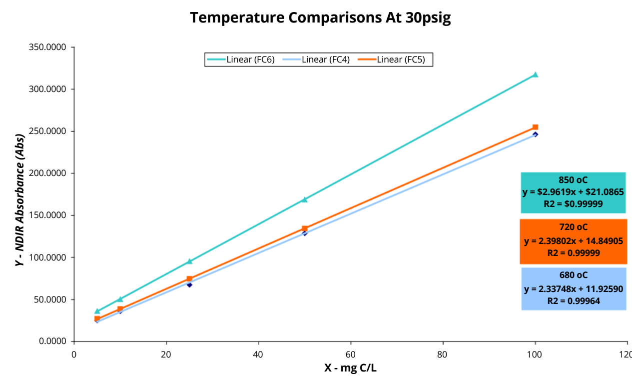
Temperature Comparisons At 30psig
Linear (FC6)
Linear (FC4)
Linear (FC5)
850 oC
y = $2.9619x + $21.0865
R2 = $0.99999
720 oC
y = 2.39802x + 14.84905
R2 = 0.99999
680 oC
y = 2.33748x + 11.92590
R2 = 0.99964
Y - NDIR Absorbance (Abs)
X - mg C/L
0.0000
50.0000
100.0000
150.0000
200.0000
250.0000
300.0000
350.0000
0
20
40
60
80
100
120

Figure 2: Calibrations compared at three furnace temperature settings with a pressure setting of 30 psig.

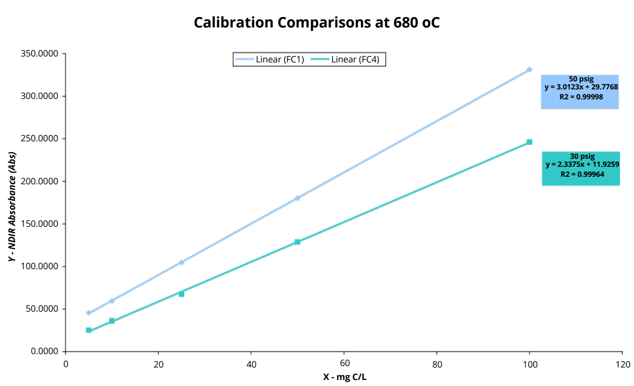

Figure 3: Comparison of two calibrations generated at two pressure settings at 680oC.

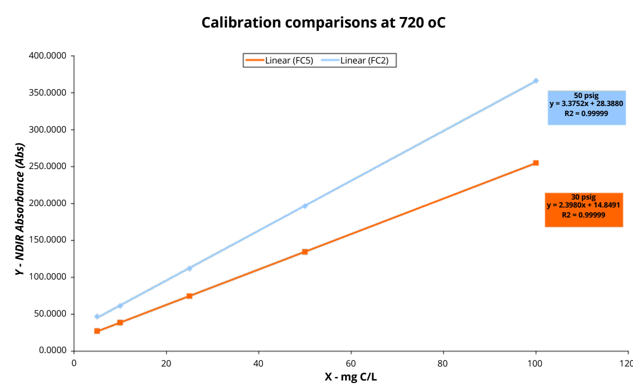

Figure 4: Comparison of two calibrations generated at two pressure settings at 720oC.

FC - 1 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	**5.3387 ppm (PASS)**	0.0585 ppm	1.1%
10.0000	1:10	**9.0280 ppm (PASS)**	0.1166 ppm	1.3%
25.0000	1:4	**24.5576 ppm (PASS)**	0.2347 ppm	1.0%
50.0000	1:2	**49.7177 ppm (PASS)**	0.2563 ppm	0.5%
100.0000	1:1	**100.8522 ppm (PASS)**	2.7885 ppm	2.8%

FC - 4 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	5.4720 **ppm (PASS)**	0.1514 ppm	2.8%
10.0000	1:10	9.7242 **ppm (PASS)**	0.0884 ppm	0.9%
25.0000	1:4	26.0439 **ppm (PASS)**	0.1712 ppm	0.7%
50.0000	1:2	52.5993 **ppm (PASS)**	0.8153 ppm	1.6%
100.0000	1:1	[illegible] **ppm (PASS)**	[illegible] ppm	[illegible]

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Table 3: Comparison of calibration check standard analyses. Each set of check standards were analyzed at 680oC.

FC - 5 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	**5.1197 ppm (PASS)**	0.1837 ppm	3.6%
10.0000	1:10	**10.0741 ppm (PASS)**	0.1845 ppm	1.8%
25.0000	1:4	**26.5163 ppm (PASS)**	0.4210 ppm	1.6%
50.0000	1:2	**52.9563 ppm (PASS)**	0.2969 ppm	0.6%
100.0000	1:1	**102.3318 ppm (PASS)**	2.5108 ppm	2.5%

FC - 2 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	5.4031 **ppm (PASS)**	0.0647 ppm	1.2%
10.0000	1:10	10.3458 **ppm (PASS)**	0.5203 ppm	5.0%
25.0000	1:4	25.0892 **ppm (PASS)**	0.5596 ppm	2.2%
50.0000	1:2	50.1484 **ppm (PASS)**	0.5305 ppm	1.1%
100.0000	1:1	99.8891 **ppm (PASS)**	0.5073 ppm	0.5%

Table 4: Comparison of calibration check standard analyses. Each set of check standards were analyzed at 720oC.

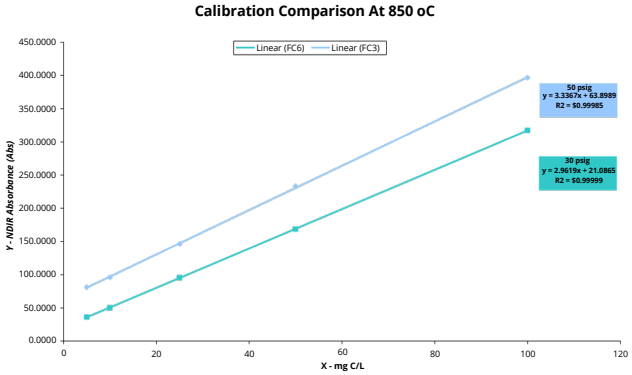

Figure 5: Comparison of two calibrations curves generated at two pressure settings at 850oC.

FC - 6 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	**4.8182 ppm (PASS)**	0.1801 ppm	3.74%
10.0000	1:10	**10.3289 ppm (PASS)**	0.3536 ppm	3.42%
25.0000	1:4	**26.0589 ppm (PASS)**	0.6137 ppm	2.36%
50.0000	1:2	**53.2665 ppm (PASS)**	1.7843 ppm	3.35%

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100.0000	1:1	**105.6098 ppm (PASS)**	4.1293 ppm	3.91%

FC - 3 Checks				
Concentration (ppm)	Dilution	Result	Std. Dev.	RSD
5.0000	1:20	**5.3387 ppm (PASS)**	0.0585 ppm	1.10%
10.0000	1:10	**9.0280 ppm (PASS)**	0.1166 ppm	1.29%
25.0000	1:4	**24.5576 ppm (PASS)**	0.2347 ppm	0.96%
50.0000	1:2	**49.7177 ppm (PASS)**	0.2563 ppm	0.52%
100.0000	1:1	**100.8522 ppm (PASS)**	2.7885 ppm	2.76%

Table 5: Comparison of calibration check standard analyses. Each set of check standards were analyzed at 850oC.

Benefits/ Conclusion

The calibration curve efficiency, for each pair of curves, was evaluated through analyzing the same points of the calibration curves as check standards using the automated dilution feature of the TekLinkTM software. The Torch HTC system performed very well on all calibration curves for linearity and precision as indicated in Tables 3-5. A comparison of the calibration curves in Figures 1 and 2 demonstrates that regardless of pressure settings, an increase in temperature results in an increase of background, but this does not significantly affect linearity or precision. The new Torch HTC Analyzer has a combined instrument and autosampler platform, user friendly TekLinkTM software, and Static Pressure Concentration (SPC) technology (patent pending). All of these features combined demonstrate Teledyne Tekmar's continuous leadership in instrument manufacturing.

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