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Total Nitrogen and Total Organic Carbon Analysis by High Temperature Combustion

Abstract

Modern High Temperature Combustion (HTC) technology with Chemiluminescence detection (CLD) provides a fast and efficient way to monitor nitrogen loading by Total Nitrogen (TN) analysis. Nitrogen monitoring can be an integral function for the process control of wastewater treatment and other industrial applications, including seawater analysis. Due to the fact that TN analysis can be performed simultaneously with traditional Total Organic Carbon (TOC) analysis, the analytical benefits can be achieved with minimal labor and capital expenditure, boosting productivity and lowering costs over existing nitrogen analysis techniques. This presentation demonstrates the efficiency of a different approach to HTC technology combined with total nitrogen analysis for a variety of key applications.

I ntroduc tion

Although the Total Kjeldahl Nitrogen (TKN) method is the current standard method for organic nitrogen analysis, it is acknowledged as being extremely tedious, time consuming and detrimental to the environment. A quicker and easier way to monitor carbon and nitrogen simultaneously (TOC/TNb) is through HTC technology coupled with CLD. Bound nitrogen analysis (TNb) is defined as organically and inorganically bound nitrogen. However, it does exclude elemental nitrogen. During HTC/ CLD analysis, the sample is combusted at temperatures up to 1,000oC. This combustion converts the nitrogen in the sample to nitric oxide. Ozone is then combined with the nitric oxide to produce an excited state of nitrogen dioxide (NO_2*). As NO_2* decays to its ground state, it emits light. The amount of light present is quantifiable with a chemiluminescence detector and correlates to a specific amount of nitrogen in the sample.

**denotes that the compound is in its excited state.*

Static Pressure Concentration Technology

The Torch HTC analyzer not only uses the latest HTC/ CLD and non dispersive infrared detector (NDIR) technology, but is coupled with Static Pressure Concentration (SPC) technology (patent pending). SPC technology is a process by which a single measurement of the CO_2 inside a NDIR detector is taken. The exit valve of the detector is closed during the oxidation of the sample, which occurs within the combustion tube inside the furnace. The entire sample pathway is then pressurized to a predetermined pressure setting with an inert carrier gas. This pressure forces the CO_2 towards the detector. Once the preset pressure setting is achieved and all the CO_2 is pressurized within the detector, a single measurement is taken. The amount of CO_2 detected correlates to the amount of carbon within the sample and this value is reported in parts per million (ppm).

Experimental-Instrument Conditions

A generic method template for TNb analysis, depicted in Table 1, was used in this study. TekLinkTM software allows manipulation of all method parameters for greater sample analysis flexibility.

General Parameters	Value
Sample Volume	0.50mL
Waste Chase Volume	1.00mL 1:1
Dilution	1 On
Number Of Injection Line Rinses	0.50mL
Injection Line Rinse	0.40 min
Injection Line Rinse Volume	0.00 min
Carrier Gas Delay Time	500mL/min
Pressure Flow Time	1.00 min
Detector Sweep Flow	200mL/min
Furnace Sweep Time	500mL/min
System Flow	1.00 min
Detector Sweep Flow	500mL/min
Furnace Sweep Time	
System Flow	

Advanced Parameters	Value
Mixer Magnet Enable	Off 2.5mL
Needle Rinse Volume	2.0mL 0.25
Vial Prime Volume	min
Baseline Stabilize Time	175mL/min
Detector Pressure Flow	10 7 50
Syringe Speed Waste	psig 7 7 7 5
Syringe Speed DI Water	0.60 min
NDIR Pressurization	Off 3
Syringe Speed Sample Dispense	5
Syringe Speed Sample Aspirate	750oC
Syringe Speed IC Dispense	0.25 min
Syringe Speed IC Aspirate	1.25 min
NDIR Pressure Stabilize	
Low Level Filter NDIR	
Syringe Speed Furnace Dispense	
Syringe Speed Furnace Aspirate	
Furnace Temperature	
TN Expansion Stabilization Time	
TN Detector Sweep Time	

Table 1: Preset method parameters used for the generation of the calibration curve and check standard analyses.

Calibration

Using the preset method parameters listed in Table 1, a calibration curve was generated using a 5.0ppm working nitrogen standard made from potassium nitrate. The autodilution feature of TekLinkTM was used to create the remaining points of the calibration curve as depicted in
Figure 1.

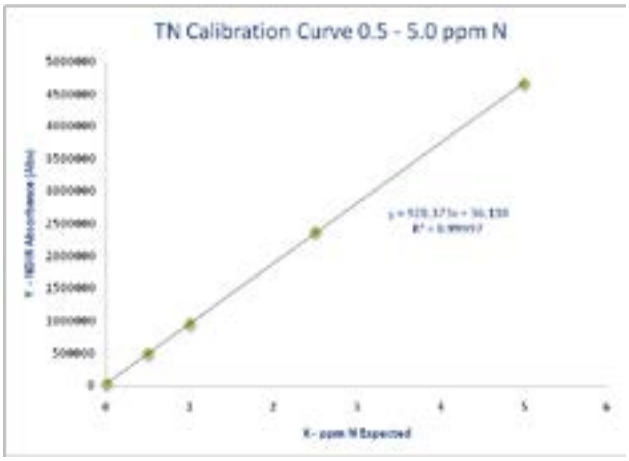

Figure 1: Nitrogen calibration curve generated using the instrument default method parameters.

Concentration (ppm N)	Dilution	Sample ID	Min / Max (% dev)	Result (ppm N)	Std. Dev.	RSD	Recovery
1.0000	1:5	[TN] Checks [1.000 ppm]	0.9000 / 1.1000 (90% / 110%)	1.0191 ppm (PASS)	0.0204 ppm	2.00%	101.9%
2.5000	1:2	[TN] Checks [2.500 ppm]	2.2500 / 2.7500 (90% / 110%)	2.6215 ppm (PASS)	0.0194 ppm	0.74%	104.9%
5.0000	1:1	[TN] Checks [5.000 ppm]	4.5000 / 5.5000 (90% / 110%)	4.9275 ppm (PASS)	0.0174 ppm	0.35%	98.6%

Table 2: Torch calibration check standards.

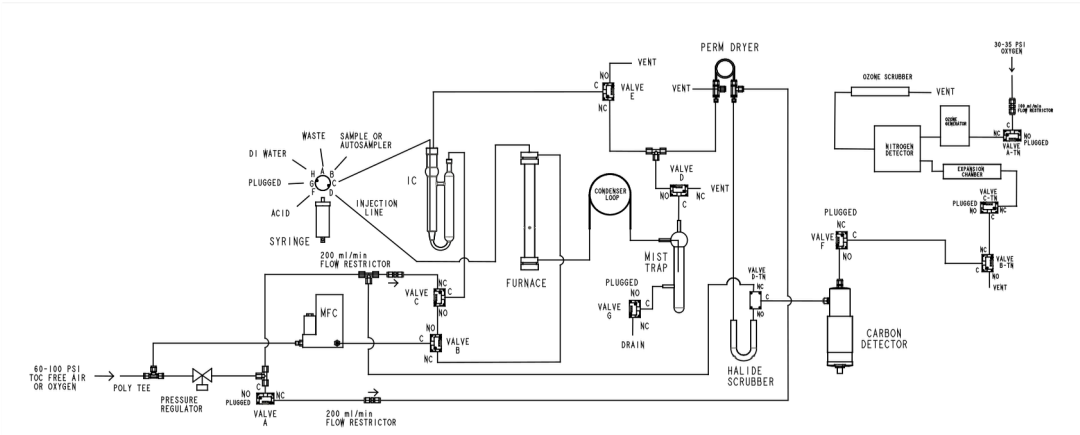

Image 1: Torch TOC Combustion Analyzer with TN module plumbing diagram.

Results Data / Conclusion

The calibration curve generated was extremely linear as the coefficient of determination (R2) value was greater than 0.9999. Three replicates were analyzed for each check standard. The results in Table 2 reflect the excellent precision of the Torch's TN capability. All results have a standard deviation of 20ppb or less. The check standard results were also highly accurate as the recovery values were all within 5% of the true value, see Table 2. The Torch offers superior accuracy and precision along with the flexibility of simultaneous TOC/TN analysis using a single instrument and autosampler platform.