

Application Note

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Abstract

This study evaluates the Lotix Solid Sampler (LSS) Boat Module's ability to analyze fertilizer for the presence of Total Organic Carbon (TOC). Accuracy and system precision, reported as percent Relative Standard Deviation (%RSD) of triplicate calibration standards, was used to evaluate the instrument's performance.

The LSS Boat Module is capable of analyzing liquid TOC samples, as well as difficult matrices like soil, sand, sediment, sludge, and particulate-laden liquid.



Introduction

The analysis of Total Organic Carbon (TOC) can be difficult due to the large variety of sample matrices. Large particulates can cause problems within TOC instruments such as blocked valves and tubing, leading to significant consumable costs and unnecessary downtime. The LSS Boat Module was designed to work in conjunction with the Lotix High-Temperature TOC Combustion Analyzer to overcome these challenges. The module can easily analyze a variety of difficult liquid and solid samples over a wide carbon concentration range with exceptional accuracy and precision.

Fertilizer is a traditionally challenging TOC sample matrix. Determination of TOC content in fertilizer is used to obtain a nitrogen to carbon ratio, which in turn is used to measure a fertilizer's effectivity.

Analytical Procedures

Instrumentation Methodology

The LSS Boat Module uses a quartz combustion tube packed with pre-conditioned cobalt oxide catalyst for oxidation. The TOC TekLink software default Total Carbon (TC) solids method was used for this analysis. Method parameters are shown in Figure 1. Inorganic Carbon (IC) in the sample was removed by the manual addition of H₃PO₄ solution in deionized water.

Figure 1 Lotix TekLink Default Total Carbon (TC) Solids Method Parameters

Parameter Name	Value	Units	Min	Max	Description
Boat Cooldown	10	sec	0	180	Time to sufficiently cool the LSS Solids Boat before new sample (normally a standard) is added to boat.
Carrier Gas Delay	5	sec	1	300	Time required to complete carbon combustion in static air stream.
Lotix Furnace	0	°C	0	450	Lotix furnace should be disabled when using solids module for total power consumed.
LSS Furnace	800	°C	650	1000	Solids furnace temperature used during analysis.

Standard Preparation

20,000 ppmC Stock Standard

1. "Indirect weighing" (weighing by difference) was used to measure 21.25 g of Potassium Hydrogen Phthalate (KHP) into a tared weigh boat. The KHP was then added to a 500 mL volumetric flask filled half way with deionized (DI) water. The flask was then brought to volume.
2. The solution was mixed thoroughly by the addition of a stir bar.

Working Calibration Standards

Working calibration standards for a 4-point concentration range of 0, 1,000, 5,000, and 20,000 ppmC were created from the 20,000 ppmC stock standard using serial dilutions.

A volume of 100 µL from each standard was injected with a syringe into the quartz boat lined with quartz wool. When prompted, the volume of standard was entered into the TOC TekLink software.

Sample Preparation

The fertilizer sample was mixed using an analytical mill to homogenize the sample. Between 0.0400 and 0.0800 g was weighed into a quartz boat for each replicate. The weight of each sample was recorded and 100 µl of 21% H₃PO₄ solution in deionized water was added to each sample to remove any Inorganic Carbon (IC) that may have been present. The sample was placed into a 100 °C oven to remove any water prior to analysis.

Sample Analysis

1. A sample schedule was created in TOC TekLink using the default “Solids TC” method.
2. A mid-range check standard of 10,000 ppmC was added to the schedule at the beginning and end of the schedule to ensure the accuracy of the results.
3. The quartz boat with fertilizer sample was placed on the module’s sample injector.
4. When prompted, the weight of the sample was entered into the TOC TekLink software. Once the weight was entered, the software automatically moved the sample into the combustion furnace for analysis and the concentration was calculated against the calibration curve.

Standard Results

The 4-point calibration curve yielded an r^2 value of 0.999 and low %RSD of the triplicates, shown in [Figure 2](#) and [Table I](#).

Figure 2 TOC TekLink Report Showing Calibration Curve Results

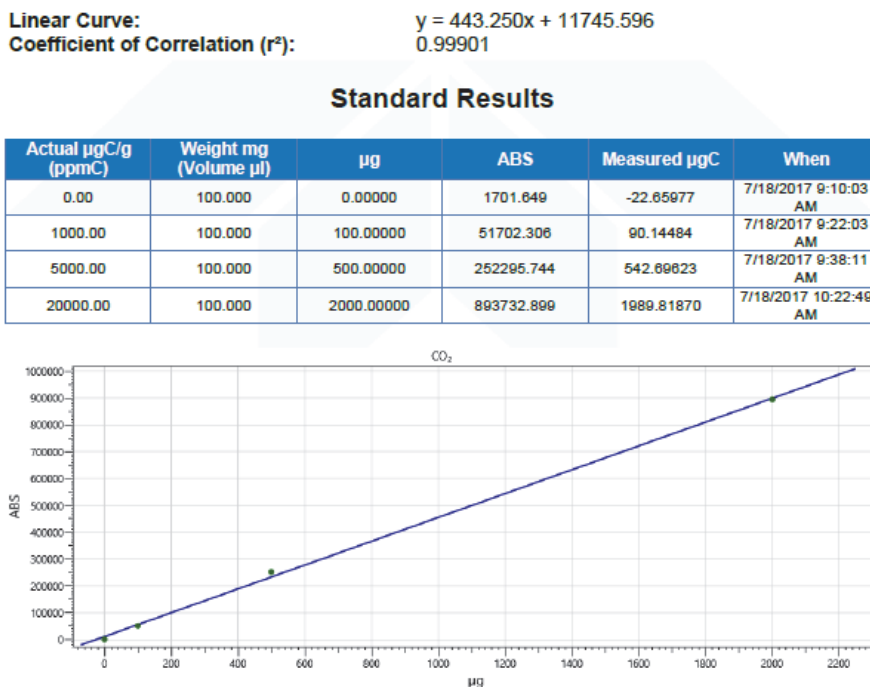


Table I %RSD of the Calibration Standard Triplicates				
Sample Type	ID	ABS	Std Dev	%RSD
Clean (TC)	Clean			
Cal Std (TC)	0.000 ppmC	1701.649	286.360	16.828
Cal Std (TC)	1000.000 ppmC	51702.306	1027.793	1.988
Cal Std (TC)	5000.000 ppmC	252295.744	312.718	0.124
Cal Std (TC)	20000.000 ppmC	893732.899	27038.779	3.025

Sample Results

Fertilizer samples were run in duplicate. The results demonstrated <5% RSD. The check standards passed with a %RSD of <1% and was within $\pm 5.0\%$ of the true value, indicating the calibration curve was valid throughout the analysis (Table II). The LSS Boat Module was able to accommodate an extensive analytical range using a single calibration curve.

Table II Sample and Check Sample Results				
Sample Type	ID	$\mu\text{g/g}$ (ppm)	Std Dev	%RSD
Clean (TC)	Clean			
Blank (TC)	Blank	1793.746 (ABS)	0.000	0.000
Chk Std (TC)	10000.000 ppmC	10297.199 Passed	58.396	0.567
Clean (TC)	Clean			
Sample (TC)	Fertilizer 20-14-13	9616.955	423.083	4.399
Clean (TC)	Clean			
Clean (TC)	Clean			
Chk Std (TC)	10000.000	10193.011 Passed	10.632	0.104

Conclusion

The Lotix Solid Sampler (LSS) Boat Module produced accurate and precise results when analyzing the fertilizer sample matrix. Analysis was simple, straightforward and without complication. Combining the LSS Boat Module with the Lotix Combustion TOC Analyzer provides a comprehensive analytical solution for TC and TOC analysis in a wide range of liquid and solid sample matrices. Additionally, the LSS eliminates the possibility of clogged valves and tubing, reducing maintenance and consumable costs. For laboratories who routinely analyze samples containing particulates greater than 0.8 mm in diameter, the LSS Boat Module is a cost-effective and reliable solution.