

# A New Approach for Single Instrument Total Organic Carbon (TOC) Analysis of Aqueous and Solid Samples

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## INTRODUCTION

Laboratories often need to test the total organic carbon (TOC) content of solid samples in addition to aqueous samples and extracts for compliance with waste management regulations, or for product quality and/or process control purposes. Representative sampling and chemical analysis is complicated by the heterogeneous nature of solid and slurry samples such as garbage, sediments, biosolids, industrial minerals, and process streams. Aqueous samples may also be laden with particulate matter requiring filtration and separate analyses of liquid and solid fractions for accurate assessment of chemical composition.

This poster describes performance characteristics of a module that enables TOC analysis of both solid and aqueous samples using a single instrument. Also described is a pre-treatment technique employing a special TOC solids crucible used for removing inorganic forms of carbon from solid samples prior to high temperature combustion.

## EXPERIMENTAL

Instrumentation used for this study included an OI Analytical Aurora 1030W TOC analyzer connected to a 1030S TOC Solids Module, (Figure 1). Organic carbon compounds in aqueous samples and extracts are oxidized by heated sodium persulfate, generating carbon dioxide (CO<sub>2</sub>) that is measured by a non-dispersive infrared detector (NDIR) in the Aurora 1030W. Solids samples are oxidized by high temperature combustion in a quartz crucible within the furnace chamber of the 1030S module. The combustion gases are collected and transferred to the NDIR of the Aurora 1030W for measurement of CO<sub>2</sub> and TOC content of the samples.

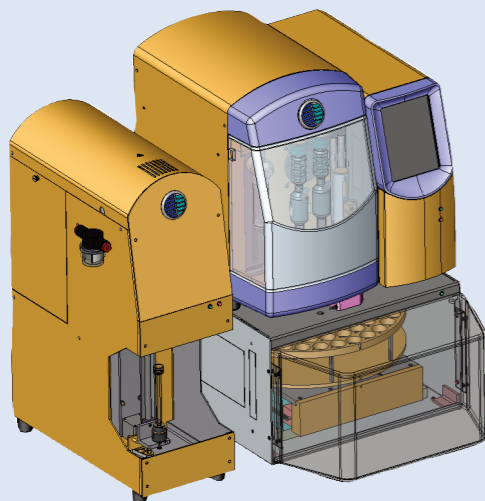


Figure 1. OI Analytical Aurora 1030W TOC and 1030S TOC Solids Module

## RESULTS & DISCUSSION

The solids module was calibrated for total carbon using microliter injections of 10% Sucrose Solution. Calibration ranges are selected by the method and vary according to the 1030W syringe injection volume. Figures 2 and 3 illustrate typical calibrations for solids analysis. The method detection limit (MDL) was determined using 100-mg portions of low carbon sediment. The calculated MDL from seven replicate analyses was 0.04% C, (40 ppm).

Results of analyses performed on selected solids samples are presented in Table 1.

### Separating Inorganic and Organic Forms of Carbon in Solid Samples

Accurate organic carbon analysis requires the capability to differentiate between inorganic and organic forms of carbon present in samples. Inorganic carbon is almost always present as carbonate minerals, either as calcium carbonate (calcite and/or aragonite), or calcium magnesium carbonate (dolomite). Organic carbon may be present as naturally occurring remnants of decaying life forms, synthetic compounds such as solvents, pesticides, and polymers, or as a combination of natural and synthetic compounds. All carbonate minerals are readily soluble in heated acid solution providing a means of distinguishing inorganic and organic carbon. Despite slight losses in volatile organic compounds (VOCs), the separation of carbonate from organic carbon by acid digestion is widely used<sup>1</sup>.

A special TOC solids crucible design, similar to a Gooch crucible commonly used to analyze total suspended solids (TSS) in environmental samples, provides a means to separate inorganic carbon compounds from organic carbon compounds, (Figures 4 and 5). This crucible design eliminates the requirement to evaporate samples to dryness after carbonate removal and, instead, separates organic carbon from the filtrate. Table 2 compares results obtained by the "filtration" method with the traditional acid digestion and evaporation method used for determining organic carbon in the presence of carbonate minerals.

The TOC Solids crucible design also allows physical separation of particulate matter suspended in water samples to improve the precision of TOC measurements. Particles can be filtered and analyzed as solids (suspended organic carbon) with the filtrate analyzed as a liquid (dissolved organic carbon). The addition of suspended and dissolved organic carbon provides a more accurate and meaningful total organic carbon result<sup>2</sup>.

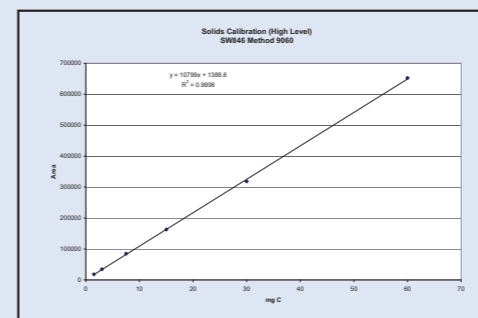


Figure 2. TOC Solids Calibration (3.5–60.0 mg/C)

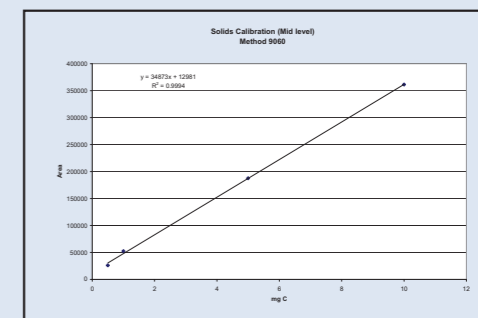


Figure 3. TOC Calibration (0.5–10 mg/C)

Table 1. Analysis of Carbon in Selected Solid Samples

Sample	Material (known value)	Amount Found
Soil Fertility	Agricultural Soil (2.2% LOI)	2.2 OM (1.3% OC)
Sediments	NIST 2702 (3.36%C)	100 mg 3.63% C
		250 mg 3.28% C
		500 mg 3.16% C
Residential Waste	Municipal Sludge	19.9% C (34% OM)
Reagent Purity	Oxalic Acid (19% C)	19.3% C
Sawdust Absorbent for Oil Spill Cleanup	Before (N/A)	37.2% C
	After (N/A)	38.2% C
Filter Media	N/A	11 micrograms C

OM = Organic Matter  
LOI = Loss of Ignition  
OC = Organic Carbon

Table 2. Comparison of Acid Digestion followed by Filtration and Acid Digestion followed by Evaporation

Component	Filtration (%Organic Carbon)	Evaporation (%Organic Carbon)
Limestone	0.44	7.64/6.84
Calcite + Humic Acid (added 6.27%)	7.64/6.84	8.48/9.22
NIST 2702 (3.27%)	3.04	—

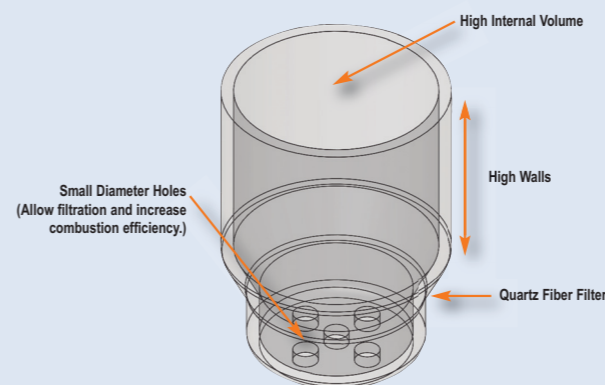


Figure 4. Illustration of TOC Solids Crucible

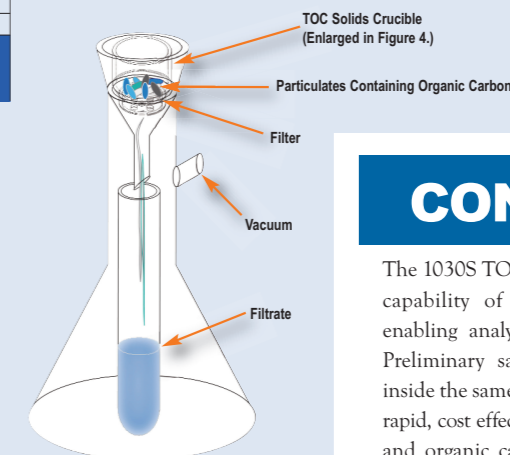


Figure 5. TOC Solids Crucible on Filtration Apparatus

## CONCLUSIONS

The 1030S TOC Solids Module expands the analytical capability of an Aurora 1030W TOC analyzer, enabling analysis of total carbon in solid samples. Preliminary sample preparation can be conducted inside the same crucible used for combustion, allowing rapid, cost effective, quantitative speciation of inorganic and organic carbon. A proprietary accumulator and utilization of the Aurora 1030W wide dynamic range detector allows analysis of heterogeneous solid samples with widely varying carbon content.

### REFERENCES

- USEPA SW846 Method 9060, Total Organic Carbon in Soil, Revision 11/16/1999
- Standard Methods for the Examination of Water and Wastewater 20<sup>th</sup> Ed. 5310B