

# high sensitivity TOC analysis in concentrated sodium chloride solutions

## introduction

Total Organic Carbon (TOC) analysis provides valuable information for industrial processes where organic carbon must be analyzed. Accurate measurements of low mg/L TOC concentrations are critical for equipment protection, process control, and product quality. However, high concentrations of sodium chloride (NaCl), chlor-alkali and pharmaceutical leachables and extractables pose a significant challenge to TOC analysis. These challenges include:

- Inorganic interferences from cationic and anionic species
- Oxidation limitation due to Chloride scavenging oxidizers
- High suspended or dissolved solids
- Fouling combustion catalyst
- Instrument degradation by hydrochloric acid (HCl)
- Limited sensitivity below 5 mg/L carbon

## solution

The Sievers\* InnovOx ES Laboratory and On-Line (OL) TOC Analyzers are uniquely suited to successfully analyze organic carbon in high salt samples. Its Super Critical Water Oxidation (SCWO) technology provides precise, accurate, and sensitive TOC quantitation, maximizing oxidation efficiency by eliminating inorganic interference. This technique achieves 50 µg/L carbon Limit of Detection (LOD) in 6M NaCl by increasing analyte in the reactor and more precisely controlling both gas and sample flows.

The instrument's robustness and sample handling capabilities provide a high tolerance to corrosive HCl and is ideal for samples with high dissolved and suspended solids. Other benefits that make the

Sievers InnovOx Lab/OL ideal for high salt samples include:

- No catalyst or combustion tube fouling or degradation
- Lower consumables cost
- No air pumps required
- Online, real time process data generation (InnovOx Online model)

## technology

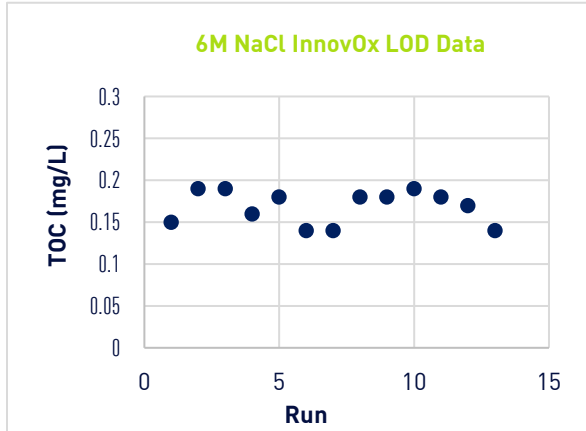
SCWO oxidizes organic carbon molecules into CO<sub>2</sub>, which is then quantified using nondispersive infrared (NDIR) detection technology. The sample is heated and pressurized above water's thermodynamic critical point. Under these conditions (375°C and 220 bar), water becomes a supercritical fluid in which organics are highly soluble and inorganic salts are insoluble. Even in the presence of high inorganic salt concentrations, these conditions generate an increased oxidation efficiency with the ability to measure TOC in aggressive and complex matrices.

The NDIR detector is stable and reliable with a calibration frequency of 6 months. The Sievers InnovOx Lab/OL can accurately measure TOC in samples containing suspended and dissolved solids (<800µm).

## performance data

The Sievers InnovOx Lab/OL LOD specification is 50 µg/L. The LOD is different from the method detection limit (MDL). The MDL was determined by running fifteen repetitions of 6M NaCl blank samples made with ultra-high purity water. The standard deviation of the TOC results was used as  $\sigma$  and the MDL was equal to  $3\sigma$ . The standard

deviation of the TOC data plotted in **Figure 1** is 20 µg/L, resulting in an MDL of 60 µg/L. This is similar to the instrument's LOD specification of 50 µg/L, demonstrating that the Sievers InnovOx Lab/OL's ability to detect organic carbon down to 250 µg/L is not affected by the saturated NaCl solution.

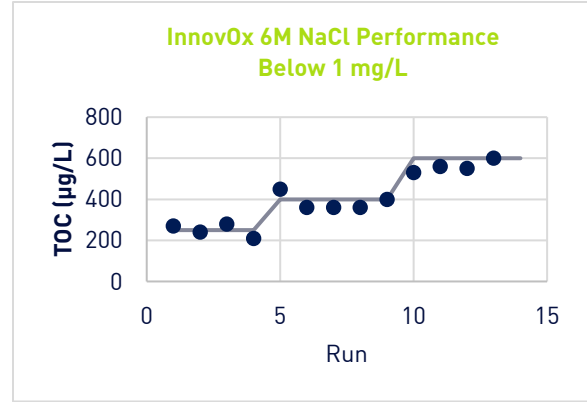


**Figure 1. 6M NaCl method detection limit data**

The TOC recovery precision and accuracy was determined by spiking 6M NaCl with 250, 400, and 600 µg/L sucrose and analyzing using the Sievers InnovOx Lab/OL. The analysis parameters were 3 vol% HCl, 25 vol% sodium persulfate oxidizer, and 0.8-minute sparging time. The results in **Table 1** show recovery within 5% relative to the standard spike and even below 500 µg/L was better than 15% to the relative precision. The recovery/standard comparison is displayed graphically in **Figure 2**. The recovery performance over the range from 250 µg/L to 600 µg/L further demonstrates that the Sievers InnovOx Lab/OL is not affected by 6M NaCl matrix effects, even below 1 mg/L TOC.

**Table 1. Sievers InnovOx Lab/OL TOC Analytical Performance below 1 mg/L in 6M NaCl**

6M NaCl				
Spiked TOC (mg/L)	TOC (mg/L)	$\sigma$	%RSD	%Dev.
0.25	0.25	0.03	13%	0.00%
0.4	0.39	0.04	10%	4%
0.6	0.57	0.03	5%	5%



**Figure 2. TOC recovery below 1 mg/L in 6M NaCl**

## conclusions

Sievers InnovOx Lab/OL analyzers were designed for harsh environments and complex aqueous matrices. The SCWO technique and NDIR detection enables ppb range TOC quantitation in saturated salt solutions.