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Research Report D19-1156

Interlaboratory Study to Establish Precision Statements for ASTM D5904, Standard Test Method for Total Carbon, Inorganic Carbon, and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

ASTM International
100 Barr Harbor Drive
West Conshohocken, PA 19428-2959

Interlaboratory Testing of Test Method for
Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity.

Collaborative Test

This method was evaluated at seven (7) laboratories. Three (3) labs used two (2) different instrument models, and one lab used two (2) different operators. The participating laboratories are given in Attachment A.

Four (4) samples were analyzed at each laboratory in triplicate on three different days for total inorganic carbon (TIC), total carbon (TC) and total organic carbon (TOC). The study samples included a reagent blank (Type II water), two (2) standards made from potassium acid phthalate and one (1) standard made from fulvic acid, which also contained carbonate and chloride. The fulvic acid study sample was made to represent a naturally occurring, complex organic material combined with potentially interfering inorganic carbon and chloride. A description of the samples is as follows:

Study Sample	Concentration	Source
A	reagent water	
B	1.25 mg/L TOC	5.0 ml stock / 2 L
C	20.0 mg/L TIC	280.0 mg Sodium Bicarbonate +
	10.0 mg/L TOC	40.210 mg Fulvic Acid
	250 mg /L chloride	Standard (50.00% C) +
		824.1 mg sodium chloride / 2 L
D	25.0 mg/L TOC	100.0 ml stock / 2 L
Stock Solution	500 mg/L TOC	531.8 mg KHP / L (NIST)

The KHP was obtained from the National Institute of Standards and Technology (NIST reference material 84j), the fulvic acid was obtained from the International Humic Substances Society (IHSS Suwannee Stream Standard Fulvic Acid) and the sodium carbonate and sodium chloride were ACS reagent grade materials.

The information sent to the participating laboratories and an example of the reporting sheet are given as Attachment B.

The analyzers used in this test measure TIC, TC and calculates TOC as the difference between those values. Study samples were supplied to the participating laboratories cold (4°C) but without acid preservation. The background variability seen in the uncorrected data is presumably due to the absorption of CO_2 therefore data was corrected for the daily background levels as determined with study sample A.

Removal of Outliers

Application of the outlier tests specified in ASTM D 2777-86 resulted in the elimination of 2 individual data points from one laboratory.

Precision

Separate determinations of precision were made for the TC & TOC measurement. The results of the least squared calculations are as follows:

TOC

$s_t = 0.027x + 0.090$

$s_o = 0.012x - 0.022$

TC

$s_t = 0.024x + 0.036$

$s_o = 0.007x + 0.006$

where:

x = average value found in mg C/L.

s_t = overall precision expressed in mg C/L.

s_o = single-operator precision expressed in mg C/L.

Both overall and single-operator precision is similar for both TOC and TC. Figures 1 and 2 show the plot of both overall and single-operator precision for TC and TOC.

Bias

Recoveries of known amounts of carbon in a series of prepared standards were as follows:

TOC

Amount Added, mg/L	Amount Found, mg/L	± Bias	± Bias %	Statistically Significant
1.25	1.22	0.03	3	Yes
10.0	9.67	0.33	3	Yes
25.0	24.78	0.22	1	No

TC

Amount Added, mg/L	Amount Found, mg/L	± Bias	± Bias %	Statistically Significant
1.25	1.22	0.03	2	No
30.1	27.83	2.27	7	Yes
25.0	24.93	0.08	0	No

Figures 3 and 4 plot the amount recovered vs. the amount added for TC and TOC.

The collaborative test data were obtained on reagent grade waters and simulated natural waters, including those high in chloride and containing carbonate. For other matrices, these data may not apply.

The raw data, corrected data and supporting statistical analyses is attached in spreadsheet format in Attachment C.