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Committee D19 on Water
Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water

Research Report RR #D19-1163

Inter-Laboratory Study to Establish Precision Statements for ASTM D5997, Standard Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

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D5997-96

Committee D-19 on Water

RR: D19.6-xx

Interlaboratory Study for the Low Level Determination of Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity

Introduction

An interlaboratory study was conducted to determine the precision and bias for the determination of low level Total Organic Carbon (TOC) in water. Standards prepared in reagent water were sent to twelve (12) laboratories for evaluation. One laboratory's data was not used as the samples were not all reported due to an instrument malfunction. A second lab failed to get required recoveries (±10% on the QC sample). Of the remaining labs, three failed the ranking test, but the one closest to passing was retained for statistical purposes as defined in ASTM D2777-96, 10.3.2.1. The data from ten (10) laboratories was used for this evaluation.

Ten samples (five pairs) were analyzed at each laboratory for Total Organic Carbon (TOC). The study samples included eight samples made from sucrose (NIST 17d) and two samples were made from benzoic acid (J. T. Baker, cat. no. 0076-01). In addition, blanks and two practice samples were sent with concentrations noted on the labels to ensure proper instrument operation for the method.

Samples were shipped cold (4°C) by overnight delivery with instructions for analysis and reporting.

Blank corrections were made on all analyses. The average blank for a given lab was used for the correction after subjecting the blank values to a statistical outlier test.

No individual data values were found to be outliers.

Attachments

A	Preparation of Study Samples
B	Test Method
C	Participating Laboratory
D	Interlaboratory Test Program Instructions
E	Data Reports
F	Statistical Data Summary
G	Approval of Study Design

Research Report Summary

Data was evaluated as defined in ASTM D2777-96 as summarized in Table I. Lab 3 was excluded as it failed to complete the analytical run, Lab 4 data was also excluded as it failed to obtain acceptable recoveries on the known standards. Labs 1 and 11 were dropped as statistical outliers by ranking. Lab 2 also failed the ranking test but was retained as described in D2777-96 10.3.2.1. All other data was used in the statistical data.

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Table I. Statistics

Concentration: (µg/L)	10	11	25	28	50	55	100	110	495	550
Number of Retained Values	8	8	8	8	8	8	8	8	8	8
True Concentration	10.0	11.0	25.0	28.0	50.0	55.0	100.0	110.0	495.0	550.0
Mean Recovery	8.53	6.04	20.58	23.14	49.19	51.13	96.70	103.08	482.45	541.33
% Recovery	85.25	54.89	82.30	82.63	98.38	92.95	96.70	93.70	97.46	98.42
Overall Std. Dev. (S_t)	4.45	3.62	3.92	2.17	4.77	2.57	6.51	4.39	17.40	21.75
Overall Std. Dev., %	52.19	59.92	19.07	9.40	9.69	5.03	6.73	4.26	3.61	4.02
Number of Retained Pairs	8		8		8		8		8	
Single Std Dev. (S_o)	5.28		1.87		2.00		3.46		3.59	
Analyst Relative deviation, %	72.47		8.54		4.00		3.47		0.70	

The following statistics are developed from the data obtained in this study:

$$XBAR = 0.9860 + -3.1740 \quad (R^2 = 0.9999)$$
$$S_t = 0.0325 + 2.4919 \quad (R^2 = 0.9628)$$
$$S_0 = 0.0007 + 3.1417 \quad (R^2 = 0.0115)$$

where:

S_t = overall precision
S_o = single operator precision
C = concentration