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Particulate Carbon and Nitrogen Analysis

Preliminary Report

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PARTICULATE CARBON AND NITROGEN ANALYSIS

PRELIMINARY REPORT

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INTRODUCTION

This report is based on an investigation conducted by the Water Quality Branch, Pacific and Yukon Region, into the procedure used for the collection of water samples for particulate nitrogen and particulate carbon analysis. The question for which an answer was sought was whether or not the particulate carbon and nitrogen values determined for water samples filtered immediately in the field and water samples stored for a period of time before filtration, differed significantly. During the examination of this question, several other considerations, discussed in the text, became evident. Depending upon study objectives, these considerations could be relevant in the planning of a particular study. As a result of this preliminary examination, a detailed investigation is being made regarding procedures used for these two parameters.

MATERIALS AND METHODS

Samples were collected into 250 ml polyethylene bottles using the Water Quality Branch, Pacific and Yukon Region, replicate sampler (Oguss and Erlebach, 1975). Bottles were washed prior to use with chromic acid and rinsed (3 times) with deionized water. The lids of the bottles had polyethylene liners.

Samples were collected in quick succession at one point on each of two British Columbia rivers, the Duncan and the Fraser. The Duncan River station was approximately 500 meters downstream from Duncan Dam in the West Kootenays. The Fraser River station was at the foot of Fraser Street, Vancouver, in the North Arm of the Fraser River. These stations were chosen arbitrarily, the Duncan River being visited relatively often and the Fraser station because of its ready accessibility.

Samples were collected in sets of six using the WQB replicate sampler. Three from each group of six samples from the Duncan River were filtered in the field within three hours of collection; the other three were filtered in the laboratory one week later. Half of the Fraser River samples were treated in this manner. Alternate groups of six samples from the remaining Fraser River samples were either field-filtered or laboratory filtered.

The samples were filtered through glass filters (5 μm mean pore size) using a glass funnel - fritted disk arrangement. The filters were pre-fired in a muffle furnace at 525°C. After filtering, the filters were dried and stored until the time of analysis.

Determinations of particulate nitrogen and carbon were performed using a Hewlett-Packard C-H-N analyser. Analytical precision for particulate carbon at a level of 0.2 mg/l is ±0.010 mg/l, at a level of 1.2 mg/l is ±0.05 mg/l; for particulate nitrogen at a level of 0.01 mg/l is ±0.002 mg/l and at a level of 0.15 mg/l is ±0.009 mg/l.

Several statistical methods were used in comparing the results of the determinations.

RESULTS

The results of the determinations of particulate carbon and particulate nitrogen and preliminary statistical analyses are included as an appendix. A summary of statistical details is given in Table I. Particulate carbon was found to increase in samples which had been stored at 5°C (116 percent of field-filtered mean value for Duncan samples, 109-115 percent of field-filtered mean value for Fraser samples). Particulate nitrogen was found to decrease in the Duncan samples which had been stored (59 percent of field-filtered mean value), while it increased in Fraser River samples (120 to 178 percent of field-filtered mean).

Two-way analysis of variance showed significant differences between field and laboratory filtered samples for the Duncan River samples (carbon p. $< .05$, nitrogen p. $< .01$). No statistical differences were found with ANOVA testing of the hypotheses of equal means for the two treatments in the Fraser River samples (Table I).

TABLE I

SUMMARY OF STATISTICAL ANALYSES

Parameter	$\bar{x}$ & 95% Confidence Limit		F Significance		
	Field Filtered	Lab Filtered	Replication	Treatment	Interaction
Duncan River 28/6/75					
Particulate Nitrogen (alternates from set of six replicates)	.038 ± .021 n = 12	.0221 ± .014 n = 12 (59%)	N.S.*	.01	N.S.
Particulate Carbon (alternates from set of six replicates)	.205 ± .082 n = 12	.237 ± .129 n = 12 (116%)	N.S.	.05	.05
Fraser River 5/8/75					
Particulate Nitrogen (alternates from sets of six)	0.076 ± 0.084 n = 24	0.135 ± 0.325 n = 24 (178%)	.01	N.S.	N.S.
(set of 6 replicates)	0.085 ± 0.042 n = 24	0.102 ± 0.051 n = 24 (120%)	N.S.	N.S.	N.S.
Particulate Carbon (alternates from sets of six)	1.01 ± 1.44 n = 24	1.10 ± 2.37 n = 24 (109%)	.05	N.S.	N.S.
(set of 6 replicates)	1.03 ± 0.458 n = 24	1.19 ± 0.705 n = 24 (116%)	N.S.	N.S.	.05

* N.S. - Not Significant

DISCUSSION

The information in Table I shows that in the Duncan River there is a significant difference in determinations for particulate nitrogen and particulate carbon between field and laboratory filtered samples. On the other hand, this was not substantiated in extensive samples collected in the Fraser River, for either particulate nitrogen or particulate carbon. Therefore, we would recommend that during the preliminary study of a river the requirement for the less convenient field filtration be examined. In the absence of clear data to the contrary, samples should be filtered immediately after collection.

Further examination of problems associated with the sampling and analysis procedure is continuing at Water Quality Branch, Pacific and Yukon Region laboratory.

SUMMARY

It appears that immediate field filtering of samples for particulate carbon and nitrogen is necessary to obtain good estimates of true values. Further examination of this problem is necessary.

REFERENCES

Oguss, E. and W.E. Erlebach (1975). Limitations of Single Water Samples as Representatives of Mean Water Quality. I. Thompson River at Shaw Springs, B.C., in preparation.

APPENDIX

1. DUNCAN RIVER - 28/06/75

(i) Particulate Carbon (mg/l) - Alternate Samples Filtered Simultaneously.

Analytical Data

Field filtered	.240 (1)	.250 (3)	.260 (5)
Laboratory filtered	.250 (2)	.260 (4)	.240 (6)
Field filtered	.220 (7)	.240 (9)	.190 (11)
Laboratory filtered	.160 (8)	.220 (10)	.150 (12)
Field filtered	.130 (13)	.150 (15)	.200 (17)
Laboratory filtered	.220 (14)	.400 (16)	.280 (18)
Field filtered	.170 (19)	.230 (21)	.180 (23)
Laboratory filtered	.170 (20)	.250 (22)	.250 (24)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance - Particulate Carbon

Source of Variation	df	ss	Mean	F	
Replication	3	.0063	.0033	1.77	Not significant
Treatment	1	.010	.0063	3.35	Significant at .05
T X R	3	.026	.0089	4.72	Significant at .05
Error	16	.030	.0018		
Total	21	.0734			

(ii) Particulate Nitrogen (mg/l) - Alternate Samples Filtered Simultaneously.

Analytical Data

Field filtered	.044 (1)	.045 (3)	.048 (5)
Laboratory filtered	.022 (2)	.032 (4)	.021 (6)
Field filtered	.027 (7)	.054 (9)	.049 (11)
Laboratory filtered	.016 (8)	.024 (10)	.018 (12)
Field filtered	.028 (13)	.016 (15)	.036 (17)
Laboratory filtered	.016 (14)	.039 (16)	.023 (18)
Field filtered	.034 (19)	.042 (21)	.036 (23)
Laboratory filtered	.012 (20)	.023 (22)	.021 (24)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance - Particulate Nitrogen

Source of Variation	df	ss	Mean	F	
Replication	3	.00153	.00009	1.36	Not significant
Treatment	1	.00028	.00153	22.06	Significant at .001
T X R	3	.00049	.00016	2.35	Not significant
Error	16	.00111	.000069		
Total	21	.0034			

2. FRASER RIVER - 05/08/75

(i) Particulate Nitrogen (mg/l) - Alternate Samples Filtered Simultaneously.

Analytical Data

Field filtered	.260 (1)	.064 (3)	.100 (5)
Laboratory filtered	.480 (2)	.110 (4)	.410 (6)
Field filtered	.051 (7)	.053 (9)	.061 (11)
Laboratory filtered	.074 (8)	.068 (10)	.080 (12)
Field filtered	.050 (13)	.057 (15)	.047 (17)
Laboratory filtered	.073 (14)	.087 (16)	.064 (18)
Field filtered	.107 (19)	.086 (21)	.086 (23)
Laboratory filtered	.071 (20)	.110 (22)	.066 (24)
Field filtered	.100 (25)	.054 (27)	.061 (29)
Laboratory filtered	.068 (26)	.070 (28)	.130 (30)
Field filtered	.057 (31)	.110 (33)	.056 (35)
Laboratory filtered	.061 (32)	.068 (34)	.066 (36)
Field filtered	.070 (37)	.046 (39)	.054 (41)
Laboratory filtered	.062 (38)	.074 (40)	.071 (42)
Field filtered	.057 (43)	.092 (45)	.066 (47)
Laboratory filtered	.077 (44)	.078 (46)	.058 (48)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance - Particulate Nitrogen - Alternate Samples

Source of Variation	df	ss	MS	F	
Replicates	7	.147	.021	6.266	Significant at .01
Treatment	1	.011	.011	3.302	Not significant
Interaction	7	.046	.006	1.971	Not significant
Error	32	.107	.003		
Total	47	.313			

(ii) Particulate Nitrogen (mg/l) - Alternate Sets of 6 Replicates Filtered Simultaneously.

Analytical Data

Field filtered	.055 (1)	.071 (2)	.058 (3)	.110 (4)	.060 (5)	.061 (6)
Laboratory filtered	.099 (7)	.084 (8)	.170 (9)	.170 (10)	.140 (11)	.130 (12)
Field filtered	.110 (13)	.140 (14)	.110 (15)	.110 (16)	.081 (17)	.110 (18)
Laboratory filtered	.085 (19)	.094 (20)	.088 (21)	.090 (22)	.090 (23)	.075 (24)
Field filtered	.088 (25)	.082 (26)	.076 (27)	.086 (28)	.077 (29)	.086 (30)
Laboratory filtered	.088 (31)	.091 (32)	.110 (33)	.099 (34)	.110 (35)	.088 (36)
Field filtered	.086 (37)	.083 (38)	.080 (39)	.069 (40)	.070 (41)	.086 (42)
Laboratory filtered	.102 (43)	.084 (44)	.095 (45)	.089 (46)	.100 (47)	.080 (48)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance - Particulate Nitrogen - Alternate Sets

	df	SS	MS	F	
Replicates	3	.0018	.000	2.107	Not significant
Treatment	1	.0034	.003	11.638	Not significant
Interaction	3	.011	.003	12.713	Significant at .05
Error	40	.011	.000		
Total	47	.028			

(iii) Particulate Carbon (mg/l) - Alternate Samples Filtered Simultaneously.

Analytical Data

Field filtered	3.990 (1)	.870 (3)	1.140 (5)
Laboratory filtered	4.390 (2)	1.080 (4)	5.520 (6)
Field filtered	.620 (7)	.620 (8)	.730 (9)
Laboratory filtered	.770 (10)	.720 (11)	.710 (12)
Field filtered	.620 (13)	.710 (15)	.590 (17)
Laboratory filtered	.710 (14)	.800 (16)	.650 (18)
Field filtered	2.990 (19)	.960 (21)	.980 (23)
Laboratory filtered	.710 (20)	1.000 (22)	.730 (24)
Field filtered	1.140 (25)	.670 (27)	.700 (29)
Laboratory filtered	.710 (26)	.700 (28)	1.440 (30)
Field filtered	.630 (31)	1.530 (33)	.670 (35)
Laboratory filtered	.650 (32)	.630 (34)	.670 (36)
Field filtered	.700 (37)	.560 (39)	.640 (41)
Laboratory filtered	.610 (38)	.640 (40)	.650 (42)
Field filtered	.670 (43)	1.180 (45)	.790 (47)
Laboratory filtered	.700 (44)	.720 (46)	.630 (48)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance

	df	SS	MS	F	
Replicates	7	21.077	3.011	5.131	Significant at .05
Treatment	1	.114	.114	.194	Not significant
Interaction	7	6.167	.881	1.501	Not significant
Error	32	18.778	.586		
Total	47	46.137			

(iv) Particulate Carbon (mg/l) - Alternate Sets of Six Replicates Filtered Simultaneously.

Analytical Data

Field	.690 (1)	.880 (2)	.740 (3)	1.090 (4)	.830 (5)	.760 (6)
Laboratory	1.240 (7)	1.210 (8)	2.680 (9)	1.430 (10)	1.620 (11)	1.280 (12)
Field	1.380 (13)	1.500 (14)	1.200 (15)	1.440 (16)	.990 (17)	1.480 (18)
Laboratory	1.010 (19)	.990 (20)	1.070 (21)	.950 (22)	1.010 (23)	.930 (24)
Field	.970 (25)	1.010 (26)	1.090 (27)	1.090 (28)	1.100 (29)	1.020 (30)
Laboratory	1.100 (31)	1.080 (32)	1.270 (33)	1.200 (34)	1.230 (35)	1.010 (36)
Field	.970 (37)	1.040 (38)	.670 (39)	.860 (40)	.980 (41)	1.090 (42)
Laboratory	1.070 (43)	.960 (44)	1.010 (45)	.950 (46)	1.210 (47)	.950 (48)

(Sample Numbers in Parenthesis)

Two-Way Analysis of Variance

	df	SS	MS	F	
Replicates	3	.343	.114	2.15	Not significant
Treatment	1	.270	.270	5.09	Not significant
Interaction	3	1.790	.596	11.26	Significant at .05
Error	40	2.120	.053		
Total	47	4.523			