# SELECTING SIEVE FRACTIONS AND SAMPLE PREPARATIONS TO ENHANCE OVERBANK- AND STREAM-SEDIMENT SURVEYS

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#### ABSTRACT

Samples of active stream sediment and overbank sediment were collected from part of NTS map area 1M, on the south coast of Newfoundland. During sieving, a variety of mesh sizes were used, and two methods of sample preparation were used for the overbank-sediment analyses. The samples were analyzed for 39 elements. In general, element data from the fine (<63 µm) fractions of both sediment types were found to be more useful than the element data derived from fractions that include coarser material. Element concentration levels and analytical precision are usually higher in the fine fraction, thus yielding data that have improved signal-to-noise ratios. Element data that are most enhanced in the fine fraction of overbank sediment include Ag, Au, Br, Cd, Ce, Cu, Hf, Pb, Sm, Ta and U. Elements that are most enhanced in the fine fraction of active stream sediment include As, Au, Br, Ce, Cu, Hf, La, Ni, Sm, Ta, Tb, U and Yb.

Exceptions to the general trend include Cr, for which the coarse fraction of overbank sediment seems to yield better data, and Mo and W, for which the coarser fraction of active sediment yields better data.

A sample preparation procedure of oxidizing overbank sediment with 30 percent  $H_2O_2$  solution to break down organic aggregations prior to sieving, enhances the signal-to-noise ratio for most data in comparison with data from conventionally prepared samples.

#### INTRODUCTION AND OBJECTIVE

Commonly, samples from stream-sediment surveys are dried, sieved to <180  $\mu$ m (<80 mesh) and sent off to a laboratory for analysis. This study suggests that depending upon which elements are of primary interest, the choice of sieve size and sample preparation technique can have a substantial effect on the resulting data. The proper selection can enable the geochemist to tailor the survey to better achieve its goals. In some cases, it may be desirable to split samples and prepare them by more than one method to produce the best information for different element groups.

In this study, samples of overbank sediment and active stream sediment were collected from part of NTS map area IM on the south coast of Newfoundland (Figure 1). Overbank sediment is normally a fine-grained sediment that deposits as a veneer on the banks adjacent to a stream or river during flooding. An overbank profile reveals the accumulation from many such events. It has been demonstrated in Scandinavia to offer some advantages over conventional active stream sediment as a sample medium (Ottesen et al., 1989), in that, it is more representative of the entire catchment basin and less influenced by industrial and agricultural activity. Two sieve-size fractions were analyzed from both types of samples and two sample preparation techniques were applied to splits of some of the overbank sediment samples. The results are presented and discussed.

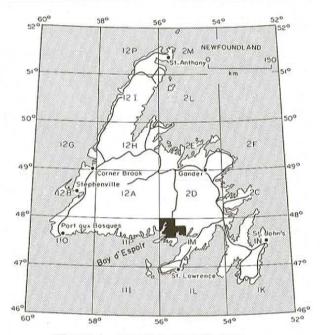


Figure 1. Location of study area.

## REGIONAL GEOLOGY AND GEOCHEMISTRY

Mapping by O'Brien et al. (1981), shows that the northeast-trending Hermitage Bay Fault divides the region

into predominantly volcanic, sedimentary and intrusive rocks of Late Proterozoic age to the southeast of the fault (Avalon Zone), and a region of predominantly clastic sedimentary and volcanic rocks northwest of the fault (Gander and Dunnage zones). Numerous mineral occurrences are known from the IM map area; in particular, map area 1M/13 has over 100 occurrences of Ag, As, Au, Cu, Mo, Pb, Sb and Zn (Brewer and O'Driscoll, 1986).

Regional-scale lake-sediment samples were collected as part of the province-wide lake-sediment survey. A recent publication provides selected element-distribution maps and statistical interpretation for 33 elements, including loss-onignition (Davenport *et al.*, 1990). Anomalously high concentrations in lake sediment of Au, Ag, As, Ba, Cr, Cu, F, Mo, Ni, Pb, Sb, W and Zn were indicated.

### METHODS OF SAMPLE COLLECTION, PREPARATION AND ANALYSES

#### SAMPLE COLLECTION

Wherever possible, paired samples of overbank sediment and active stream sediment were collected from adjacent sites to compare the geochemical responses of the two materials. Seventy-one samples of active sediment and 63 samples of overbank sediment were collected. Duplicate overbank and active sediment samples (site duplicates) were collected at twelve sites. A duplicate overbank sample is a second sample collected at a suitable site within about 100 m of the first. Duplicate samples of active sediment were collected from approximately the same areas of the stream bed.

A sample of overbank sediment was obtained by digging a small pit to expose the overbank profile, which normally ranged from 15 to 50 cm in thickness. A representative sample of the section was then collected from the face in a fashion similar to obtaining a channel sample in bedrock. Active stream sediment was sampled from several sources in the stream bed using a spade. About 10 to 15 L of <5 mm sediment were obtained initially by screening material through a garden sieve. This was further reduced to <300  $\mu$ m by wetsieving on-site, resulting in a final sample weight of about 200 to 300 g.

#### SIEVING, SAMPLE PREPARATION AND ANALYSES

All overbank samples were first oven-dried and sieved and the  $<\!500~\mu\mathrm{m}$  fraction retained. When microscopic examination of the samples indicated that many grains were actually composites of smaller grains, thirteen of the larger samples were split and an additional sample series created. In an attempt to disaggregate the composite grains, the following procedure designed to oxidize the binding organic matter was performed on samples of this subseries.

Approximately 100 to 200 g of sample were placed in a beaker and saturated with distilled water. A solution of 30 percent H<sub>2</sub>O<sub>2</sub> was gradually added while stirring the sample

until effervescence ceased. The mixture was heated slowly to about 70°C and evaporated to dryness. The sediment was then wet-sieved with distilled water into two size fractions, <63 and 63 to  $500 \mu m$ .

The procedure for the other split portion of the subseries, as well as for all remaining overbank samples, consisted of soaking the samples overnight in a very weak (3 percent) solution of  $\rm H_2O_2$  and wet-sieving them into two size fractions as above.

The samples of  $<300~\mu m$  active sediment were drysieved to form two fractions, <63 and  $<180~\mu m$ . No additional sample preparation was performed.

The various size fractions and sample series described above were analyzed by a variety of methods for forty elements. Some elements, notably Ba, Ce, Co, Fe, La, Ni, Sc and Zn were analyzed by more than one method. Of the elements discussed in this report, As, Au, Cr, Mo, Sb, Ta and W were determined by neutron activation analysis and Ag, Mn and Pb by atomic absorption analysis. As a check on accuracy and precision, a standard sample of known composition and a sample duplicate (split) were included in each batch of 20 samples. Details of analytical methods may be found in McConnell and Honarvar (1991).

#### RESULTS

#### SIZE FRACTIONS

#### **Distribution Statistics**

One method of assessing the impact of analyzing different size fractions is to compare basic statistics of the element populations. Tables 1 and 2 list the geometric means,  $\log_{10}$  standard deviations and data ranges of most of the elements analyzed. To permit comparisons between the two sample types, data have been included only from the 49 sites that have both overbank- and active stream-sediment samples. The data have been log-transformed to compensate for the positively skewed distributions of these elements. Most have standard deviation/mean ratios >0.5 in the untransformed data.

From Table 1, the mean values of the fine fraction of overbank samples equal or exceed the mean values of the coarse fraction for most elements. Exceptions, e.g., where the mean value in the coarse fraction is significantly greater than in the fine fraction, include Ba, Cr, Rb and Th by ICP analysis.

Comparing the mean values of the <63 and the <180  $\mu m$  fractions of active sediment in Table 2 indicates that the tendency toward enrichment of trace elements in the fine fraction is even more pronounced in this type of sample. Of all elements analyzed only Ba, Mo and Rb and the major element Na have higher mean concentrations in the coarser fraction.

Table 1. Means, standard deviations and data ranges of  $<63 \mu m$  and 63 to 500  $\mu m$  fractions of overbank sediment. Means and ranges in g/t unless otherwise specified (N=49)

	GEOMETRIC MEAN			STANDARD DEVIATION (log)		DATA RANGE		
	<63 μm	63 - 500 μm	<63 μm	63 - 500 μm		<63 μm	63 - 500 μm	
922	0.11	0.05	0.24	0.15		0.05 0.5	0.05	
Ag	0.11 29.50	0.05 26.30	0.34 0.56	0.15		0.05- 0.5 2.4- 402	0.05 - 0.4 $1.7 - 473$	
As	1.66	1.29	0.36	0.38		1- 24	1- 15	
Au (mg/t) Ba	363.00	407.00	0.41	0.29		99- 1237	89- 1286	
Ba1*	324.00	372.00	0.36	0.22		25 - 1400	25- 1400	
Br	34.70	21.90	0.43	0.43		2.2 300	1.7- 216	
Cd	0.25	0.15	0.30	0.32		0.1- 2	0.1 - 1.4	
Ce	72.40	57.50	0.23	0.32		20- 501	19- 312	
	75.90	55.00	0.25			26- 564	5- 360	
Ce1	12.10	12.90	0.44	0.31		1- 287	1- 339	
Co	12.10	12.90	0.48			2- 330		
Co1	58.10	100.00	0.48	0.50		10- 310	2- 420 10- 390	
Cr	7.08	6.17	0.16	0.29		2.8- 18	1.3- 13	
Cs	16.20	12.30	0.38	0.40		1- 134	1- 67	
Cu	4.47	3.89	0.38			1.6- 21.8		
Dy				0.24		The second secon		
Eu (g/)	0.72	0.74 3.02	0.25 0.19	0.24			0.5 - 3.5 $0.67 - 9.48$	
Fe (%)	2.82 3.89	3.55	0.17	0.19		0.54 - 7.78 $0.81 - 8.6$		
Fe1 (%)	21.90		0.09			12- 33		
Ga		21.40		0.11		2- 20	1400.00	
Hf	7.59	4.37	0.22	0.16			1.8- 14	
La	30.90	25.10	0.26	0.22		100 to 10	8- 171	
Lal	29.50 35.50	22.90	0.28 0.19	0.23		5- 297 13.2- 88.4	6- 140 $10- 81.5$	
Li	0.22	36.30 0.17	0.19	0.20		0.1 - 1.6		
Lu		741.00	0.53	0.50		104-13300	0.1 - 1.3 $90 - 12370$	
Mn	692.00 3.02		0.57					
Mo		2.69		0.47		AD 3 (75)		
Na (%) Nb	1.05 9.33	1.10 8.13	0.13 0.24	0.17 0.22		0.4 - 1.7 $2 - 57$	0.34 - 2.18 $2 - 39$	
Ni	15.10	13.50	0.39	0.47		1- 81	1- 69	
Nil	10.20	12.90	0.41	0.44		5- 99	5- 79	
Pb	22.40	14.80	0.32	0.35		7- 192	3- 132	
Rb	77.60	91.20	0.21	0.33		19- 230	27- 250	
Sb	1.02	0.98	0.42	0.45		0.15 - 8.5	0.19 - 6.4	
Sc	10.20	9.55	0.14	0.43		3.5- 19	2.3- 18	
Sm	6.46	5.01	0.14	0.23		1.3- 52	1.4 - 30.2	
Sr	89.10	87.10	0.20	0.23		19- 230	20- 212	
Ta	1.17	0.91	0.26	0.27		0.2 - 7.2	0.2 - 5.2	
Tb	1.02	0.81	0.30	0.26		0.2- 7.2	0.2 - 3.2 $0.2 - 4.6$	
Th2	3.72	6.31	0.60	0.40		0.5- 55	0.2 - 4.0 $0.5 - 41$	
ThI	10.23	8.13	0.24	0.21		4.3 - 52.4	3.6 - 35.3	
U	5.13	3.72	0.36	0.21		1.7 - 73.3	1.1 - 48.7	
v	87.10	83.20	0.36	0.34		25- 252	1.1 - 48.7	
w		1.66		0.26				
Y	1.66 20.00	17.00	0.40 0.27	0.37		0.5- 9.4 6- 115	0.5- 20 6- 80	
Yb	2.88	2.82	0.32	0.24		1- 18	6- 80 1- 12	
Zn	70.80	64.60	0.32			15 - 388	(H)	
Zn Zr	138.00	132.00	0.30	0.27 0.27		100 - 790	9- 258 100- 840	
Z.il	136.00	152.00	0.50	0.27		100- 790	100- 640	

<sup>\*</sup> In instances where an element is analyzed by more than one method, '1' indicates neutron activation, and '2' indicates ICP.

#### Site Duplicates

A good measure of the significance and reliability of data can be obtained by determining correlations among site duplicates. Scatterplots of data from 12 pairs of site duplicates are presented in Figure 2. Data include Ag, Au, Mo, Sb, Ta, and W in <63 and 63 to 500  $\mu$ m fractions of overbank sediment and in <63 and <180  $\mu$ m fractions of active sediment.

Of the six elements plotted, Ag shows the most

pronounced variation with size fraction. Of the four silver plots, only the  $<63~\mu m$  overbank fraction shows any correlation (0.64)-16 of 24 analyses have detectable Ag. Of the remaining 70 analyses, only two have detectable levels of Ag.

The effect of size fraction (and sample type) is also pronounced for Au. The scatterplot of Au in the  $<63~\mu m$  overbank samples is the only one of the four plots with good correlation (0.54). Five of the nine sample pairs analyzed that have detectable Au in both splits are in this fraction. The

**Table 2.** Means, standard deviations and data ranges of  $< 63 \mu m$  and  $< 180 \mu m$  fractions of active stream sediment. Means and ranges in g/t unless otherwise specified (N=49)

	GEOMETRIC MEAN		STAN	DARD ION (log)	DATA R	DATA RANGE		
	<63 μm	<180 μm	<63 μm	<180 μm	<63 μm	<180 μm		
Ag	0.06	0.05	0.17	0.09	0.05- 0.3	0.05- 0.2		
As	26.90	19.50	0.57	0.58	1.9- 557	2.2- 575		
Au (mg/t)	2.14	1.51	0.49	0.46	1- 80	1- 536		
Ba	372.00	417.00	0.21	0.22	85 - 1430	76- 1619		
Ba1*	363.00	479.00	0.35	0.28	25- 1700	25- 2000		
Br	11.80	5.75	0.40	0.34	0.2 - 91.8	1- 34		
Cd	0.23	0.18	0.26	0.27	0.1- 0.9	0.1 - 0.9		
Ce	87.10	55.00	0.27	0.16	17- 557	21- 148		
Ce1	89.10	52.50	0.29	0.19	13- 553	14- 170		
Co	12.00	9.77	0.36	0.39	2- 57	1- 63		
Col	15.90	12.30	0.39	0.39	2- 71	2- 82		
Cr	186.00	166.00	0.42	0.41	10 - 700	10- 1500		
Cs	5.25	5.37	0.19	0.17	0.8- 15	2.1- 13		
Cu	15.10	10.00	0.34	0.38	2- 70	1- 60		
Dy	5.50	4.17	0.21	0.19	1.7- 18.1	2.1 - 13.4		
Eu	0.66	0.60	0.22	0.17	0.5- 2.5	0.5 - 1.9		
Fe (%)	2.09	1.74	0.18	0.21	0.65 - 4.21	0.42 - 4.63		
Fe1 (%)	4.17	3.31	0.18	0.22	1- 10	0.65 - 9.1		
Ga	20.40	20.00	0.10	0.11	14- 35	9- 34		
Hf	22.40	5.75	0.48	0.23	0.5- 281	1.8- 28		
La	37.20	24.60	0.26	0.15	7- 283	9- 71		
Lal	37.20	22.90	0.27	0.15	7- 256	8- 62		
Li	35.50	34.70	0.17	0.17	17.8 - 87.7	17.3 - 77.9		
Lu	0.40	0.30	0.40	0.35	0.1 - 3.3	0.1 - 1.5		
Mn	692.00	525.00	0.40	0.43	137 - 10210	123 - 11620		
Mo	1.20	1.35	0.45	0.43	0.5- 15	0.5- 15		
Na (%)	1.38	1.41	0.16	0.16	0.23 - 2.25	0.34 - 2.65		
Nb	11.20	9.55	0.36	0.25	1- 90	2- 49		
Ni	28.80	15.90	0.34	0.41	4- 135	1- 96		
Nil	22.40	15.50	0.51	0.42	5- 130	5- 85		
Pb	16.60	13.80	0.29	0.25	3- 125	5- 123		
Rb	91.20	97.70	0.21	0.20	26- 300	29- 310		
Sb	1.23	1.02	0.36	0.38	0.18-6.6	0.2 - 5.8		
Sc	12.30	10.50	0.17	0.21	5.3 - 32.8	2.8 - 30.1		
Sm	8.13	5.25	0.24	0.14	1.6 - 28.9	1.9 - 13.9		
Sr	115.00	110.00	0.25	0.25	23 - 344	18- 316		
Ta	2.14	1.38	0.34	0.24	0.2- 18	0.2 - 7.6		
Tb	1.32	0.89	0.32	0.21	0.2 - 5.5	0.2 - 2.9		
Th2	3.39	3.16	0.70	0.57	0.5- 194	0.5- 65		
Thl	13.20	8.32	0.34	0.24	3.9- 190	3.2 - 58.8		
U	5.37	2.95	0.32	0.20	1.7- 40.1	1- 10		
V	89.10	79.40	0.24	0.26	16- 295	10- 256		
W	2.57	2.51	0.46	0.49	0.5- 27	0.5 - 124		
Y	25.10	20.40	0.26	0.21	8- 121	9- 68		
Yb	5.50	3.24	0.29	0.27	1- 28	1- 11		
Zn	81.30	67.60	0.23	0.23	24 - 258	28- 260		
Zr	776.00	170.00	0.64	0.34	100-13000	100- 1300		

<sup>\*</sup> In instances where an element is analyzed by more than one method, '1' indicates neutron activation, and '2' indicates ICP.

The correlation of 0.30 in the <180  $\mu$ m active sediment fraction may be misleading as it is strongly influenced by a single sample with detectable Au in both splits.

The effect on Mo is more equivocal than in the cases of Ag and Au. Scatterplots of both fractions of overbank sediment are very similar; both fractions have 7 of 12 sample pairs having detectable Mo in both splits. The plot of Mo in  $<63~\mu m$  active sediment indicates this fraction has the poorest correlation of the four and the fewest pairs (2) with detectable Mo in both splits. The  $<180~\mu m$  fraction of active sediment has the best correlation (0.69) although it has fewer

pairs (5) with detectable Mo in both splits than do either of the overbank fractions.

The scatterplots of antimony (Sb) have the strongest correlations of the six elements plotted and appear unaffected by variation in size fraction. The correlations are excellent in all four plots, being slightly stronger in the active sediment fractions (0.99) than in the overbank fractions (0.96).

The scatterplots of Ta show two influences at work. The fine fractions of both sample types are enriched relative to the coarse fractions. The active sediment samples are enriched

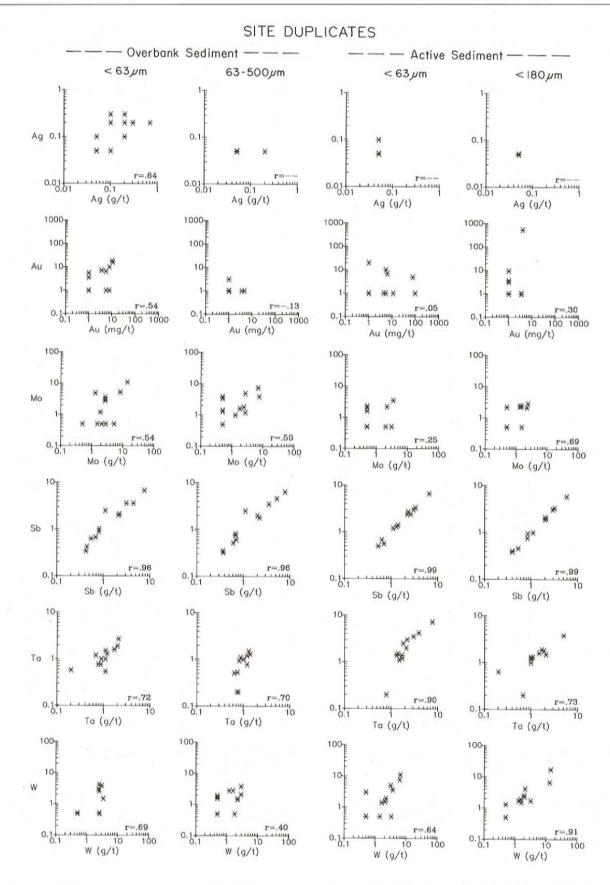


Figure 2. Scatterplots of 12 pairs of site duplicates of Ag, Au, Mo, Sb, Ta, and W in  $< 63 \mu m$  and 63 to  $500 \mu m$  fractions of overbank sediment and in  $< 63 \mu m$  and  $< 180 \mu m$  fractions of active sediment (r=correlation coefficient of  $\log_{10}$  data).

Table 3. Means, standard deviations and data ranges of 13 pairs of sample splits of overbank sediment prepared by normal preparation and by 30 percent H<sub>2</sub>O<sub>2</sub> oxidation. Means and ranges in g/t unless otherwise specified

	GEOMETRIC MEAN			STANDARD DEVIATION (log)		DATA RANGE		
	Normal Prep.	H₂O₂ Prep.	Normal Prep.	H <sub>2</sub> O <sub>2</sub> Prep.		Normal Prep.	H <sub>2</sub> O <sub>2</sub> Prep.	
Ag	0.08	0.06	0.32	0.13		0.05- 0.3	0.05 - 0.1	
As	29.50	49.00	0.49	0.50		2.6 - 99.4	5.2 - 177	
Au (mg/t)	2.45	4.68	0.53	0.43	7	1- 24	1- 14	
Ba	363.00	468.00	0.07	0.07		246- 441	321 - 577	
Ba1*	372.00	490.00	0.15	0.12		130 - 500	210- 640	
Br	21.90	1.66	0.28	0.41		10 - 94.8	0.2 - 7.3	
Cd	0.19	0.22	0.25	0.22		0.1 - 0.7	0.1 - 0.7	
Ce	77.60	87.10	0.13	0.26		53- 135	14- 156	
Ce1	81.30	100.00	0.14	0.12		55 - 130	71 - 160	
Co	13.20	10.50	0.13	0.12		8- 27	6- 18	
Co1	14.50	12.00	0.13	0.16		10- 29	5.5- 21	
Cr	105.00	129.00	0.23	0.17		30- 190	54- 200	
Cs	6.31	8.13	0.15	0.18		4.3 - 17	5.1- 26	
Cu	23.40	22.40	0.17	0.11		12- 45	16- 34	
Dy	3.89	4.68	0.11	0.11		2.9 - 6.8	3.3 - 8.3	
Eu	0.85	0.93	0.23	0.24		0.5 - 1.9	0.5 - 2.1	
Fe (%)	2.51	3.24	0.11	0.12		1.82 - 3.71	2.1 - 4.85	
Fe1 (%)	3.39	4.68	0.10	0.12		2.4 - 5.1	2.8 - 7.3	
Ga	19.50	26.30	0.06	0.08		15- 25	19 – 35	
Hf	9.12	9.55	0.16	0.12		4.8- 20	6.2 – 16	
La	33.90	41.70	0.14	0.12		22 - 65	27 – 72	
Lal	31.60	39.80	0.13	0.12		22 - 60	28- 64	
Li	32.40	41.70	0.13	0.13		19.9- 52.1	25.4- 71.2	
Lu	0.21	0.27	0.28	0.31		0.1 - 0.43	0.1 - 0.59	
Mn	617.00	372.00	0.54	0.36		89- 2520	79 - 908	
Мо	1.91	2.04	0.46	0.54		0.5 - 10	0.5- 20	
Na (%)	1.20	1.32	0.08	0.07		0.8 - 1.6	0.92 - 1.6	
Nb	8.71	11.00	0.15	0.14		6- 17	6- 20	
Ni	22.90	22.90	0.27	0.22		8- 58	8- 48	
Ni1	10.70	19.50	0.38	0.42		5- 40	5- 59	
Pb	17.40	31.60	0.21	0.16		9- 34	14 - 64	
Rb	81.30	112.00	0.16	0.17		55- 200	62 - 310	
Sb	1.78	2.45	0.44	0.44		0.44 - 7.8	0.53 - 11.5	
Sc	9.77	12.30	0.04	0.05		8.8- 13	11- 15	
Sm	7.08	8.91	0.11	0.11		4.7 - 10.8	6.3 - 13.1	
Sr	97.70	102.00	0.17	0.17		51- 230	59- 233	
Ta	1.17	1.62	0.16	0.18		0.68- 2.7	0.82 - 3.9	
Tb	1.05	1.32	0.09	0.10		0.81 - 1.6	0.95 - 2.2	
Th2	5.62	5.25	0.42	0.48		0.5 - 18	0.5- 13	
Th1	10.70	14.10	0.17	0.15		5.5 - 20.8	7.2 - 24.9	
U	4.57	5.13	0.25	0.25		1.9- 15	2.3- 19	
V	75.90	104.70	0.08	0.08		56- 114	75 - 139	
W	2.09	7.00	0.37	0.29		0.5- 5.5	0.5 - 5.2	
Y	16.60	19.10	0.12	0.12		11- 28	13- 32	
Yb	3.63	3.55	0.06	0.08		2.6- 4.6	2.3 - 4.8	
Zn	66.10	75.90	0.17	0.15		37 - 134	42 - 144	
Zr	170.00	275.00	0.36	0.44		100- 600	100- 1000	

<sup>\*</sup> In instances where an element is analyzed by more than one method, '1' indicates neutron activation, and '2' indicates ICP.

relative to the overbank samples. In both types, correlations are better in the fine fractions.

Tungsten differs the most from the other elements plotted. Of the four plots, the <180  $\mu$ m fraction of active sediment has the best correlation (0.91) and the most samples with detectable W in both splits.

### SAMPLE PREPARATION BY PEROXIDE TREATMENT

Basic statistics are provided in Table 3 to permit comparison of the effects of two different sample preparation methods on the distribution characteristics of the data populations. Data from thirteen samples that were split and

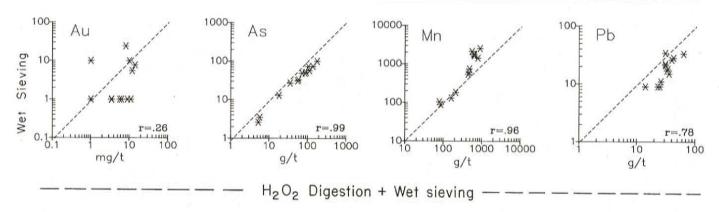


Figure 3. Scatterplots of Au, As, Mn and Pb in 13 pairs of sample splits of  $< 63 \mu m$  overbank samples comparing wet-sieved preparation with preparation by 30 percent  $H_2O_2$  oxidation followed by wet-sieving.

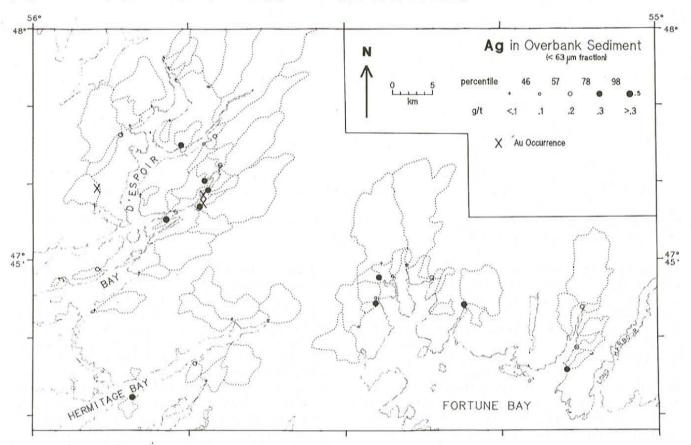


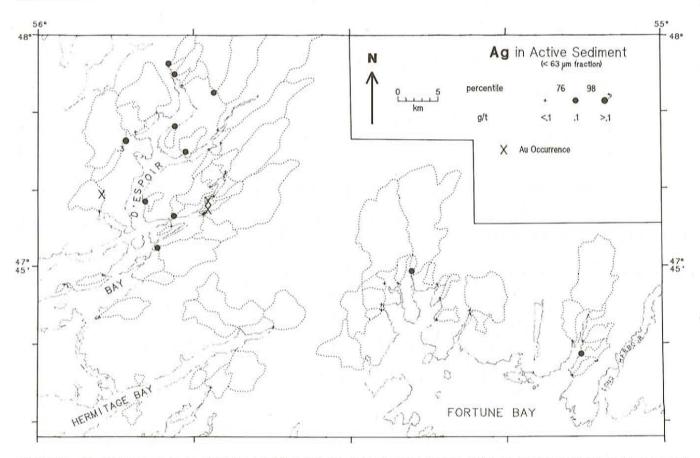
Figure 4. Distribution of Ag in < 63  $\mu$ m (<250 mesh) fraction of overbank sediment in Bay d'Espoir area. Sampled catchment basins are enclosed by dotted lines.

prepared as described previously are compiled. For most elements, mean values in the samples prepared by 30 percent  $H_2O_2$  digestion are higher than in the normally prepared samples. Values in this group are 39 to 80 percent higher for the elements As, Au, total iron (Fel), Ga, Pb, Rb, Sb and Ta. For two elements, the reverse situation is true. Bromine is virtually eliminated and Mn values are much lower in the samples oxidized by  $H_2O_2$ . The scatterplots in Figure 3 graphically show the differences in the analytical results from the two preparation methods for Au, As, Mn and Pb.

#### DATA DISTRIBUTION MAPS

Symbol maps showing the distribution of values for two elements, Ag and Au, are included to illustrate the effects on distribution patterns of different size fractions and sample types.

Silver was selected because it shows highly contrasting analytical responses in different size fractions; gold also has higher concentrations in the fine fractions of both media (Figure 2 and Tables 2 and 3). Figures 4 to 6 display the



**Figure 5.** Distribution of Ag in < 63  $\mu$ m (< 250 mesh) fraction of active stream sediment in Bay d'Espoir area. Sampled catchment basins are enclosed by dotted lines.

distribution of Ag in <63  $\mu$ m overbank, <63  $\mu$ m active sediment and <180  $\mu$ m active sediment, respectively. The patterns are clearly varied. The map of Ag in <63  $\mu$ m overbank sediment (Figure 4) contains the most samples with detectable Ag. Most of the highest values cluster around the two gold occurrences east of Bay d'Espoir. In contrast, most of the high Ag values in <63  $\mu$ m active sediment are located to the west and north of Bay d'Espoir (Figure 5). The distribution of Ag in the <180  $\mu$ m (<80 mesh) fraction of active sediment (Figure 6) is included to illustrate the almost complete lack of geochemical response in the fraction that is usually analyzed in stream-sediment surveys.

Figures 7 and 8 depict the distribution of Au in <63  $\mu$ m overbank and <63  $\mu$ m active sediment. For sites with duplicate samples, the mean (geometric) value of gold is plotted. The highest Au in the overbank samples (24 mg/t) is from the only catchment basin that actually contains known mineralization. The two gold occurrences to the east of Bay d'Espoir are very close to sampled basins and may be reflected by the sample of active sediment having 8.1 mg/t Au.

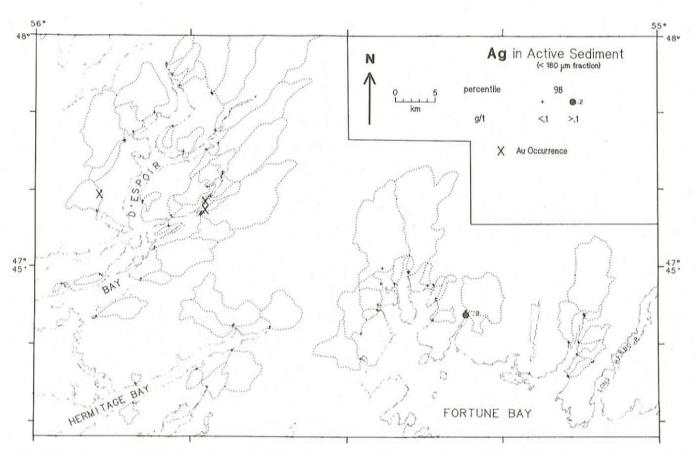
#### DISCUSSION

#### SIZE FRACTIONS

The effect of different size fractions on geochemical response varies widely by element. For most elements

analyzed in this study, the fine fractions of both overbank and active sediment have higher concentrations than the corresponding coarse fractions (Tables 1 and 2). Other factors being equal, it is advantageous to have higher concentrations when interpreting data as these improve the signal-to-noise ratio by increasing the contrast with background and by improving the precision of analyses. This is particularly true of elements for which many samples have values at or near the detection limit. Elements for which detection limits are a problem and which yield higher values in the fine fraction of overbank sediment include Ag, Au, Cd, Hf, Mo and Ta. As noted above, the tendency toward element enrichment in the fine fraction is even more pronounced in active sediment than in overbank sediment.

Scatterplots of site duplicates (Figure 2) of fine and coarse fractions serve to reinforce the desirability of selecting the fine fraction for analysis of most elements, particularly for overbank sediment. Correlation among site duplicates is generally stronger in the fine fraction of overbank; silver is the most extreme example. Only Ag data from the fine fraction of overbank sediment has any correlation (r=0.64). Most samples of other fractions are at or below the 0.1 g/t detection limit. Similarly for gold, of all the fractions, only the fine fraction of overbank shows a significant correlation among its duplicates. However, for some elements, particularly Cr, Mo and W, the <180  $\mu$ m fraction of active



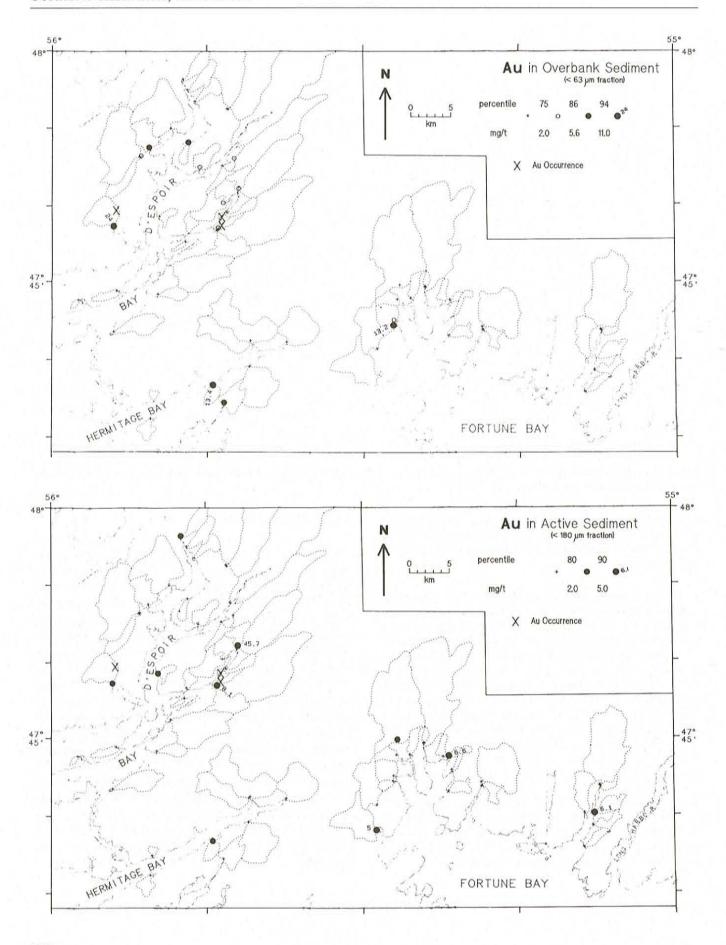
**Figure 6.** Distribution of Ag in <180  $\mu$ m (<80 mesh) fraction of active stream sediment in Bay d'Espoir area. Sampled catchment basins are enclosed by dotted lines.

sediment has significantly stronger correlations than does the  $<63~\mu m$  fraction suggesting that these elements occur in minerals that are more homogeneously distributed in the coarse fraction.

An important point to remember when comparing correlations of site duplicates of active sediment with site duplicates of overbank sediment, is the dissimilar nature of the two sample types. Samples of active sediment represent a 'snapshot' of sediment composition at a given time. The sediment composition at a given site following changes in water levels, Eh conditions, sediment sources or transport conditions may be quite different. Moreover, the method of collecting active sediment, whereby it is a composite of several sites within a small area, would tend to increase the homogeneity and similarity of site duplicates. However, for overbank samples there is little or no expectation of composition change with changing stream conditions as the sample already is a composite of multiple flooding episodes over a long period of time. A single flooding event would not substantially change the composition of the overbank profile. Thus, although the correlations of active sediment duplicates may appear better, when the time factor is considered it makes the comparisons uncertain.

#### SAMPLE PREPARATION

Basic statistics are presented in Table 3, tabulating the analytical results of two sample-preparation procedures applied to 13 pairs of sample splits of overbank sediment. Of the two methods, the 30 percent H2O2 oxidation procedure resulted in considerably higher concentration levels in the fine fraction for many trace elements including gold. Not illustrated, but present, was a corresponding drop in the relative concentration levels in the coarse fraction. This enrichment trend likely results from the fact that many mineral phases that are enriched in trace elements in sediments are finer grained than the more resistate and abrasion-resistant quartz and feldspar grains. If this fine material is cemented into composite grains, they would remain in the coarse fraction with the quartz and feldspar during normal sieving. By disaggregating these composite grains with a process such as H2O2 oxidation prior to analysis, the resulting data should have improved the analytical precision and signal-to-noise ratios. This step will reinforce the effect already initiated by analyzing the fine fraction rather than the coarse.



#### CONCLUSIONS

These observations are made on a modest-sized dataset from only one area and should be regarded as tentative, until confirmed by similar results from elsewhere. Nonetheless, certain conclusions and recommendations are warranted.

There appears to be no single, best sieve size to use for preparing overbank or active stream sediment for analysis, as results vary according to the element. However, analytical precision and concentration levels may be increased for most elements by analyzing the  $<63~\mu m$  fraction of overbank sediment rather than a coarser fraction. In overbank sediment, this effect is most pronounced for Ag, Au, Br, Cd, Ce, Cu, Hf, Pb, Sm, Ta and U. The same conclusion applies, in general, to active stream sediment, where contrast between fine and coarse fractions is even stronger. The elements most enhanced in the fine fraction of active sediment are As, Au, Br, Ce, Cu, Hf, La, Ni, Sm, Ta, Tb, U and Yb. However, for Ba in both sample types, for Cr in overbank sediment, and for Mo and W in active sediment, the information provided by the coarser fraction appears superior.

A sample preparation procedure that separates composite mineral grains, such as oxidation by 30 percent  $H_2O_2$  prior to sieving, can enhance data precision for many elements by increasing the concentration levels in the fine fraction. One side effect of the method, however, is the virtual elimination of Br and organic matter.

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**Figure 7** (top of page 298). Distribution of Au in <63  $\mu m$  (<250 mesh) fraction of overbank sediment in Bay d'Espoir area. Sampled catchment basins are enclosed by dotted lines.

**Figure 8** (bottom of page 298). Distribution of Au in <180  $\mu$ m (<80 mesh) fraction of active stream sediment in Bay d'Espoir area. Sampled catchment basins are enclosed by dotted lines.