INFLUENCE OF SIZE-FRACTIONING TECHNIQUES ON CONCENTRATIONS OF SELECTED TRACE METALS IN BOTTOM MATERIALS FROM TWO STREAMS IN NORTHEASTERN OHIO

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IN NORTHEASTERN OHIO

By G. F. Koltun and Dennis R. Helsel

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CONVERSION FACTORS

For the convenience of readers who prefer to use U.S. customary units, conversion factors for metric (International System) units in this report are given below:

Multiply metric units	By	To obtain U.S. customary units
meter (m)	3.281	foot (ft)
square kilometer (km^2)	0.3861	square mile (mi^2)
millimeter (mm)	0.03937	inch (in.)

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ABSTRACT

Identical stream-bottom material samples, when fractioned to the same size by different techniques, may contain significantly different trace-metal concentrations. Precision of techniques also may differ, which could affect the ability to discriminate between size-fractioned bottom-material samples having different metal concentrations.

Bottom-material samples fractioned to less than 0.020 millimeters by means of three common techniques (air elutriation, sieving, and settling) were analyzed for six trace metals to determine whether the technique used to obtain the desired particle-size fraction affects the ability to discriminate between bottom materials having different trace-metal concentrations. In addition, this study attempts to assess whether median trace-metal concentrations in size-fractioned bottom materials of identical origin differ depending on the size-fractioning technique used. Finally, this study evaluates the efficiency of the three size-fractioning techniques in terms of time, expense, and effort involved.

Bottom-material samples were collected at two sites in northeastern Ohio: One is located in an undeveloped forested basin, and the other is located in a basin having a mixture of industrial and surface-mining land uses. The sites were selected for their close physical proximity, similar contributing drainage areas, and the likelihood that trace-metal concentrations in the bottom materials would be significantly different.

Statistically significant differences in the concentrations of trace metals were detected between bottom-material samples collected at the two sites when the samples had been size-fractioned by means of air elutriation or sieving. Statistical analyses of samples that had been size fractioned by settling in native water were not measurably different in any of the six trace metals analyzed.

Results of multiple comparison tests suggest that differences related to size-fractioning technique were evident in median copper, lead, and iron concentrations. Technique-related differences in copper concentrations most likely resulted from contamination of air-elutriated samples by a feed tip on the elutriator apparatus. No technique-related differences were observed in chromium, manganese, or zinc concentrations.

Although air elutriation was the most expensive size-fractioning technique investigated, samples fractioned by this technique appeared to provide a superior level of discrimination between metal concentrations present in the bottom materials of the two sites. Sieving was an adequate lower-cost but more labor-intensive alternative.

INTRODUCTION

Identifying the areal distribution of a chemical constituent in streams is the goal of many chemically oriented studies. This goal is more difficult to achieve when dealing with constituents associated with stream-bottom materials because of the complex array of factors that affect the measured concentrations.

Tne association of metals with sediment particles is a function of many physical and mineralogical factors (Horowitz, 1984). Researchers have demonstrated that the concentrations of trace metals measured in stream-bottom materials may be greatly influenced by the particle-size distribution of the samples (Fitchko and Hutchinson, 1975; Shimp and others, 1971). As a consequence, analysis of sediment trace-metal chemistry without regard for particle-size effects can result in increased data variability. This increased variabilty may complicate data analyses during the reconnaissance phase of a study when approximate areal concentration patterns are being determined.

For many metals, particle-size-related variability may be reduced by isolating and analyzing the size fraction composed of fine silts and clays (Rickert and others, 1977; Helmke and others, 1977). Helsel and Koltun (1986) found that chemical analysis of the <0.020-millimeter (mm) size fraction (clays and fine silts) minimized sample-to-sample variations in certain metal concentrations found within bottom materials of a single stream cross section, and enhanced discrimination between bottom-material samples originating from different depositional basins.

Particle-size distribution is only one of many factors that affect the trace-metal chemistry of bottom materials. As a consequence, additional detailed study, including a determination of metal distribution as a function of particle-size distribution over all class sizes, needs to be performed once areal trends have been established. Chemical analyses of the <0.020-mm fraction, however, may be useful as an initial step during the reconnaissance phase of a study; therefore, the techniques used to obtain the <0.020-mm fraction and their effects (if any) on trace-metal concentrations are also of interest.

Purpose and Scope

The purpose of this report is to present the results of a study to (1) evaluate the influence of three different size-fractioning techniques on the ability to discriminate between bottom-material samples having different trace-metal concentrations; (2) determine if measured metal concentrations differ significantly among samples of like origin that were size fractioned by different techniques; and (3) document the time, cost, and relative effort involved in using each of the size-fractioning techniques. Data in this report are based on bottom-material samples collected in July 1983 at two stream sites in northeastern Ohio (fig. 1). The bottom-material samples were size fractioned and analyzed for six trace metals.

Acknowledgments

The authors wish to thank the owners and manager of Beaver-kettle Farm[1] of East Liverpool, Ohio, for their assistance in providing access and transportation to and from the Pine Run site.

DESCRIPTION OF STUDY SITES

Bottom-material samples were collected at cross sections located on West Fork Stateline Creek near Negley, Ohio, and Pine Run near Fredericktown, Ohio (fig. 1). These sites, which are located in Columbiana County, were chosen for (1) the geologic and physiographic similarities of their contributing drainage, (2) their close physical proximity, and (3) the likelihood that trace-metal concentrations in the stream-bottom materials would differ significantly from site to site.

The 5.0-square-kilometer (km^2) drainage area of the West Fork Stateline Creek site contains surface mines and two chemical-waste handling facilities. In 1977, the U.S. Geological Survey installed a stream gage on Stateline Creek approximately 10 meters below the site at which the bottom-material samples were obtained for this study. Surface-water samples were collected and analyzed for a variety of chemical constituents intermittently from 1977 until 1980, at which time the gage was discontinued. In some of the samples collected during this period, concentrations of total recoverable lead, iron, cadmium, and phenols exceeded maximum allowable instream concentrations stipulated by Ohio water-quality standards (Ohio Environmental Protection Agency, 1978).

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[1]Use of the name Beaverkettle Farm in this report is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.
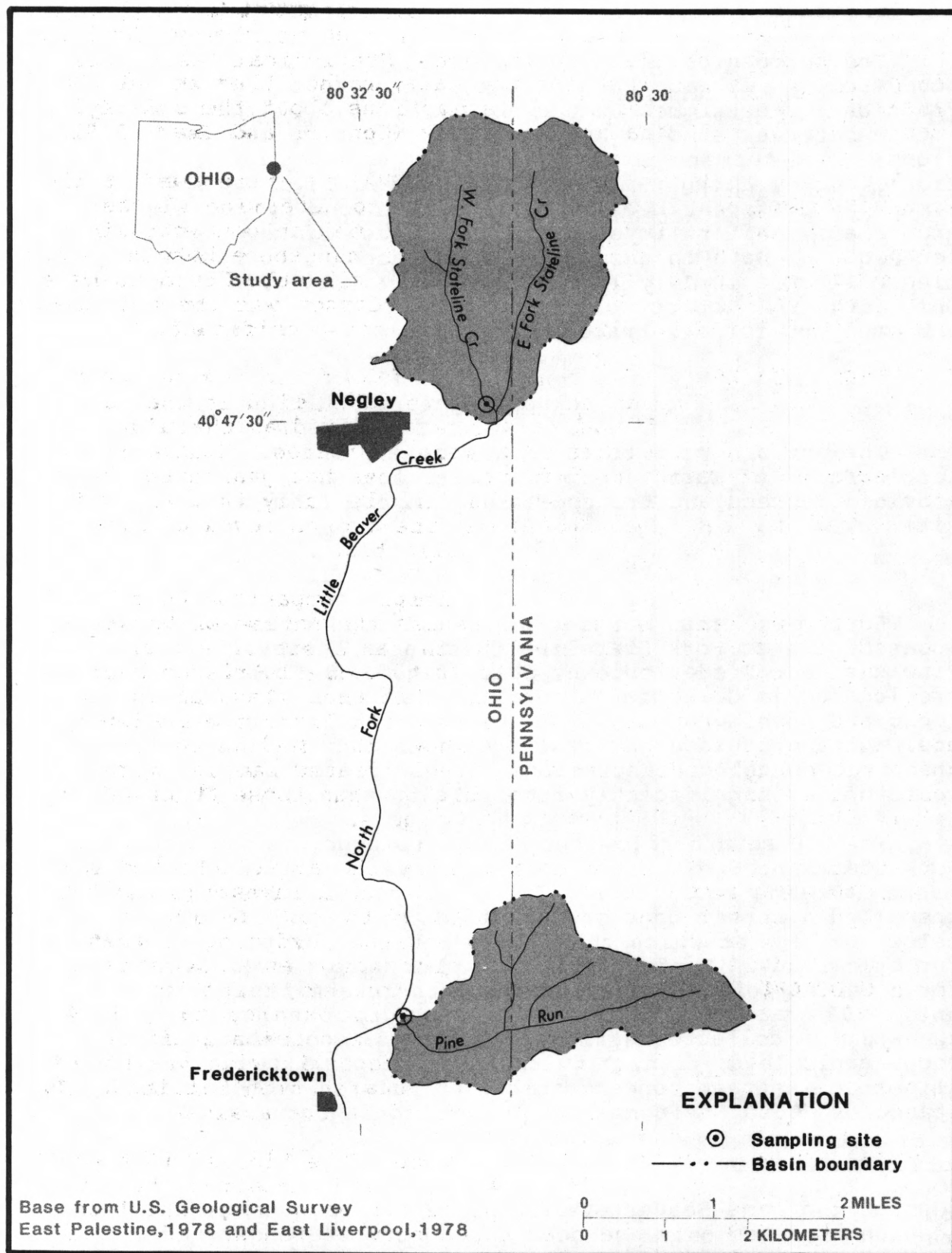
Figure 1.--Locations of sampling sites at West Fork Stateline Creek and Pine Run.

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The 5.16-km^2 drainage area of the Pine Run site is predominantly forested and relatively undisturbed by human activities. Although historical water-quality data were not available for Pine Run, it was surmised that long-term water quality at this site would be better than water quality in the West Fork Stateline Creek subbasin.

Bottom materials, because of their interaction with overlying waters, may act as temporal integrators of water quality (Feltz, 1980). It was therefore expected that the concentration of selected metals in the bottom materials would reflect the differing water-quality history at the two sites.

METHODS OF STUDY

Bottom-Material Sampling and Size-Fractioning Techniques

Bottom-material samples were collected at five randomly selected positions along one cross section established at each site. Randomness was obtained by using a pseudorandom-number generator that produces numbers uniformly distributed between two other bracketing numbers. A tag line was extended across the width of the stream, and the tag-line distances corresponding to the left and right edges of the stream were read. The tag-line distances were then used as bracketing numbers for the uniform random-number generator.

In order to obtain sufficient bottom materials for the chemical analyses, multiple discrete samples were taken at each sampling position. Each discrete sample was collected by entrapping the top 25 to 50 mm of the stream bed in containers made of linear polyethylene. A polyethylene cap was slid over the mouth of the container after it had been inserted into the stream-bed material to prevent loss of fine particles while bringing the sample to the surface. The discrete samples collected at each individual position within the cross section were composited and thoroughly mixed, which resulted in one homogeneous sample per position.

Samples collected at each of five randomly selected positions within two cross-sections (one located on Stateline Creek and one located on Pine Run) were individually size fractioned. Each sample was sieved in the field to remove particles larger than 2 mm. The samples were further size fractioned by means of settling in native stream water (after additional sieving to 0.25 mm), sieving, or air elutriation so that only particles less than 0.020 mm in diameter remained. As a result, a total of 30 size-fractioned bottom-material subsamples were available for chemical analyses (three size-fractioned subsamples from each of five positions randomly selected from within one cross-section located on two separate streams). Figure 2 is a schematic of the sample-collection and size-fractioning processes followed at each individual position within a cross-section.

COLLECT AND COMPOSITE MULTIPLE DISCRETE
SAMPLES TO FORM A HOMOGENEOUS MIXTURE
FOR A SINGLE CROSS—SECTION POSITION

SIEVE TO
<2 mm

SIEVE TO
<0.25 mm

AIR ELUTRIATION

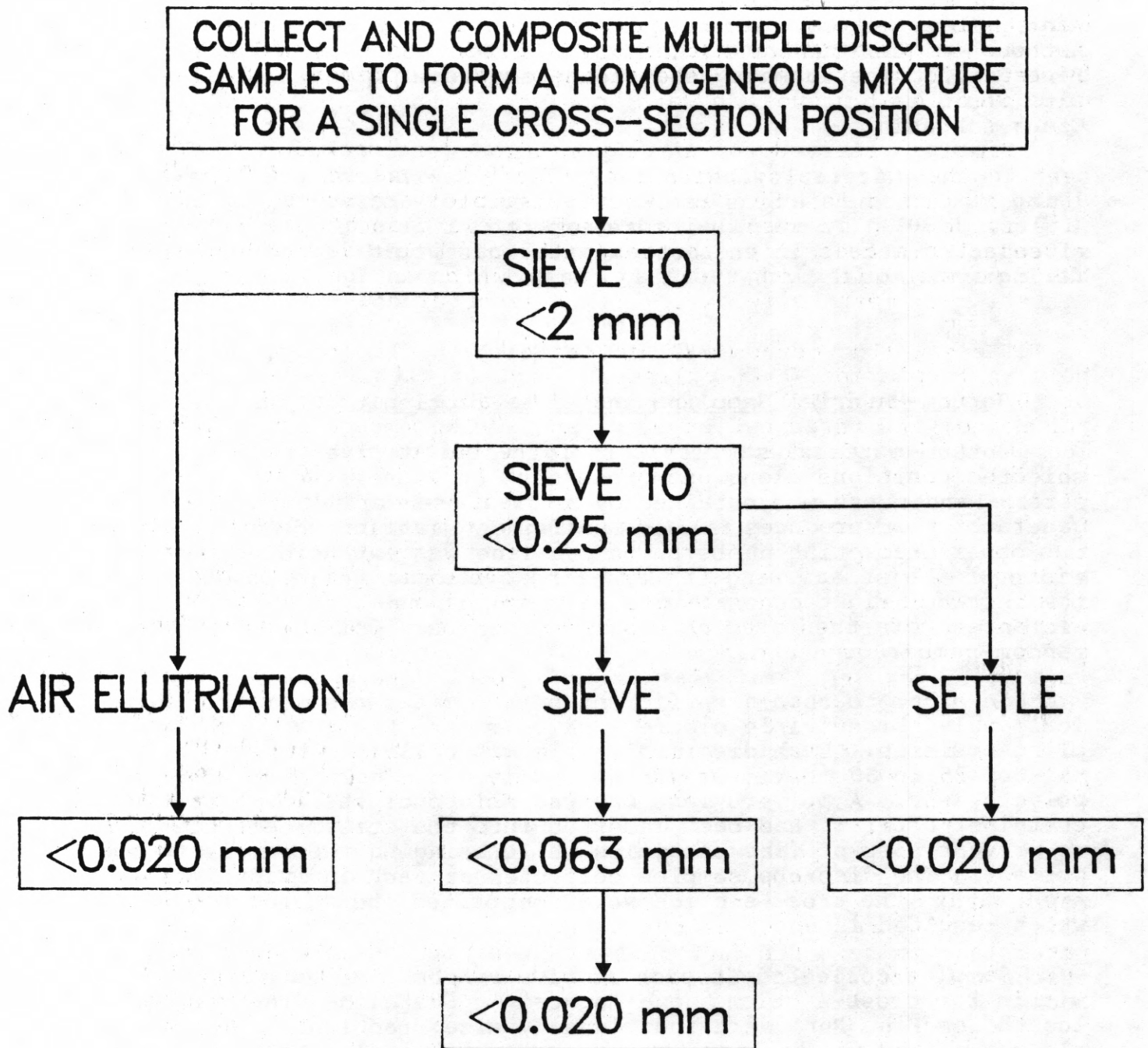SIEVE

SETTLE

<0.020 mm

<0.063 mm

<0.020 mm

<0.020 mm

Figure 2.--Schematic representation of sample–collection and size–fractioning procedures
performed at each cross–section position.

All sieving was done on site by means of a series of linear polyethylene sieves. Linear polyethylene sieves were chosen to prevent possible contamination of the bottom-material samples by metallic constituents used in the screens, frames, and solders of conventional sieves.

Samples reserved for air elutriation were packed in ice and sent to the U.S. Geological Survey office in Madison, Wisconsin. Air elutriation is a process whereby samples are freeze dried and size fractioned by means of a microparticle classifier. The microparticle classifier separates the particles according to their terminal settling velocity, from which an equivalent Stokesian-diameter size distribution is obtained.

The settling method was performed in the laboratory using polyvinyl chloride (PVC) cylinders (fig. 3) with inside diameters of 50.8 mm. Each PVC cylinder had removable stoppers at the top and bottom and three outlet ports spaced 100 mm apart along the length of the cylinder. Portions of the bottom-material sample, which had been previously sieved in the field to 0.25 mm, were placed in the PVC cylinder, and sufficient stream water was added to the cylinder so that the water-sediment mixture level was above the top outlet port. The cylinder was then stoppered and agitated to disperse the bottom materials. The cylinder was agitated for about 3 minutes by repeated tilting such as used for a bottom-withdrawal tube (Guy, 1969). Immediately after agitating the sediment-water mixture, the PVC cylinder was placed vertically in a rack, and the top outlet port was opened to establish the initial reference fluid level.

The theory behind the use of the PVC cylinders is analogous to that for bottom-withdrawal tubes (Guy, 1969). Stokes's Law was used to compute the settling time for particles 0.020 mm in diameter to fall 100 mm. After waiting the computed time, the next lower outlet port was opened and an aliquot withdrawn. Two such withdrawals are made from each cylinder. The process was repeated until sufficient volume of sample had been collected. This new mixture, which contained water and bottom-material particles smaller than 0.020 mm, was centrifuged to consolidate the bottom-material particles, and the overlying water was decanted.

Laboratory Analyses of Trace-Metal Concentrations

The clay and fine silt fraction (<0.020 mm) obtained by means of each of the size-fractioning techniques were analyzed for total recoverable chromium, copper, iron, manganese, lead, and zinc at the U.S. Geological Survey's central laboratory in Doraville, Georgia. The extraction procedure was performed on the bottom materials, and the extractant was analyzed by means of an atomic-absorption spectrophotometer using techniques described by Skougstad and others (1979). The metal concentration data are shown in table 5 at the back of this report.
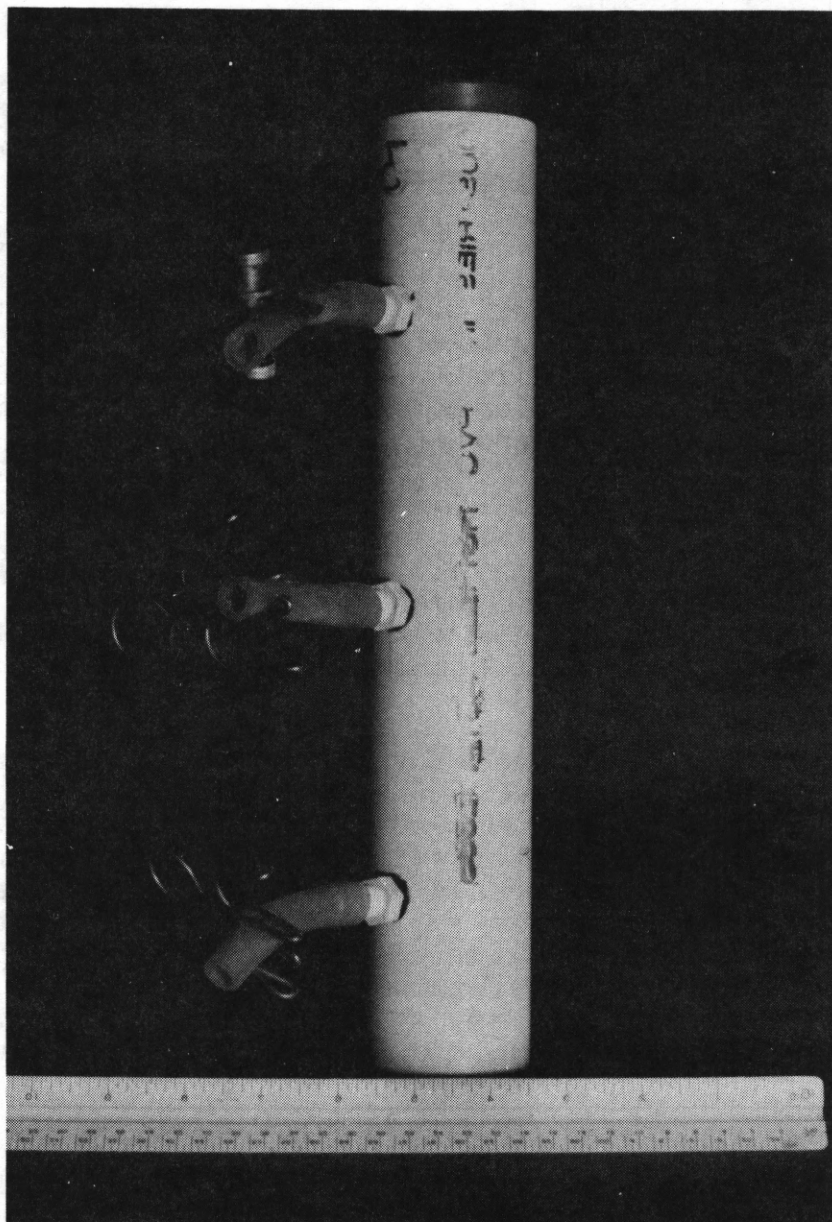
Figure 3.--Settling apparatus.

PARTICLE-SIZE DISTRIBUTIONS IN FRACTIONED BOTTOM MATERIALS

The distribution of particle sizes in unfractioned bottom materials is known to affect the measured concentrations of many trace metals (Horowitz, 1984). It is possible that the distribution of particle sizes in fractioned bottom-material samples also would have some smaller, but measurable, effect on the concentrations of trace metals.

The distribution of particle sizes in bottom materials fractioned by sieving, settling, and air elutriation was determined for three bottom-material samples collected from a central Ohio stream. Particle-size diameter was plotted against the percentage of particles in the sample finer than the stated size (fig. 4). The plots suggest that differences in the particle-size distributions may occur among the size-fractioning techniques. Specifically, the air-elutriation technique may result in a particle-size distribution with a larger median grain size than that produced by sieving or settling. If this is true, it may be because air-elutriated samples are subject to less mechanical breakage. An insufficient number of analyses were made to statistically evaluate the differences observed between the distributions.

STATISTICAL ANALYSES

In the following discussion, differences in median metal concentrations that are measured in samples collected from the Pine Run and Stateline Creek sites are called "between-site" differences. Differences in median metal concentrations that are associated with a size-fractioning technique and are independent of any between-site effects are called "between-technique" differences. The alpha level (the probability of rejecting a true null hypothesis) chosen for tests of significance in this report was 0.05.

The metal-concentration data associated with each size-fractioning technique were statistically analyzed individually and as a group in order to address the following questions:

1. When metals data for size-fractioned bottom materials are combined and analyzed as a group, are there statistically detectable between-site differences in one or more of the median metal concentrations? Are there detectable between-technique differences in the median concentrations of one or more metals? If there are, how do concentrations associated with each technique compare with each other?

2. For an individual size-fractioning technique, are there detectable between-site differences in the median metal concentrations?
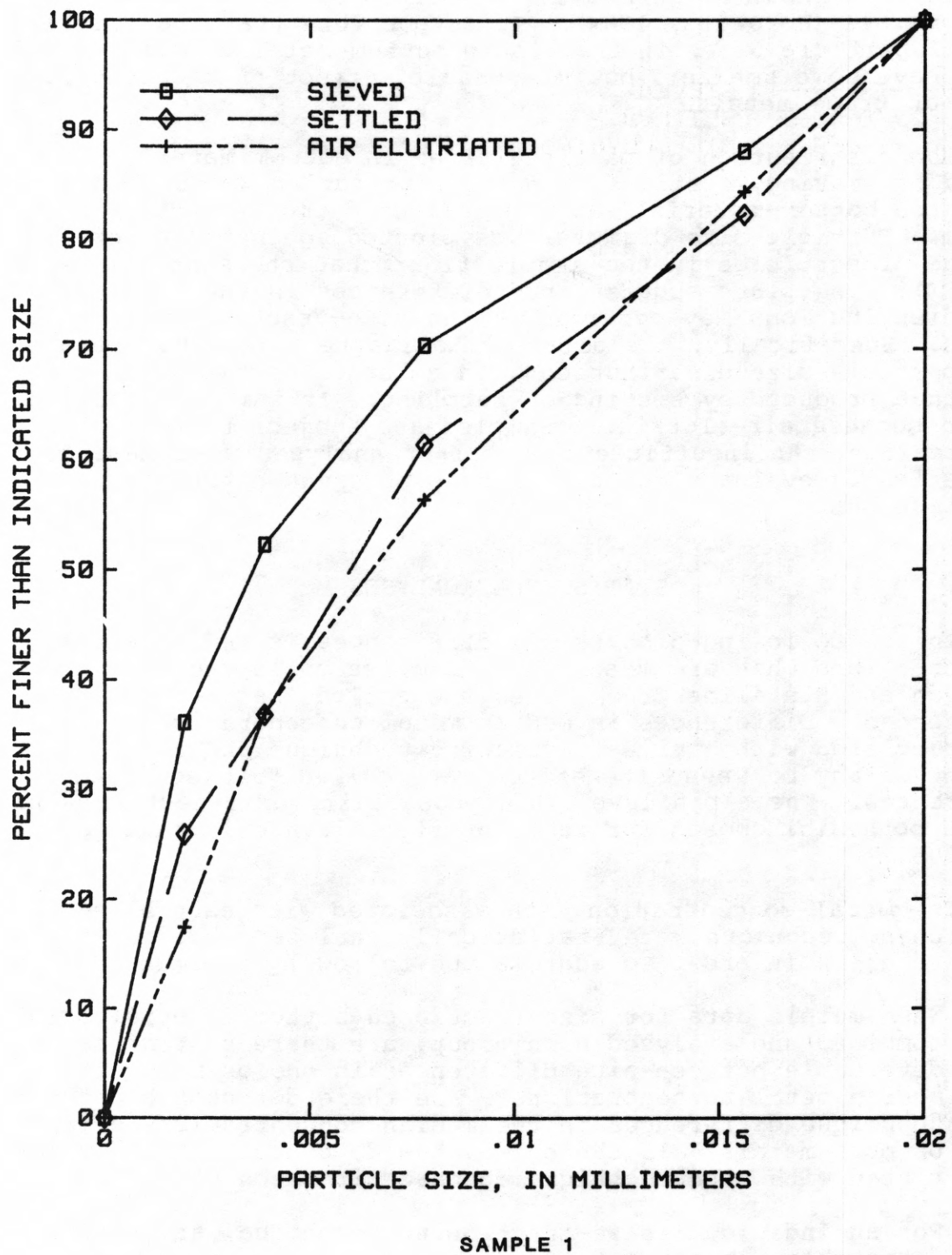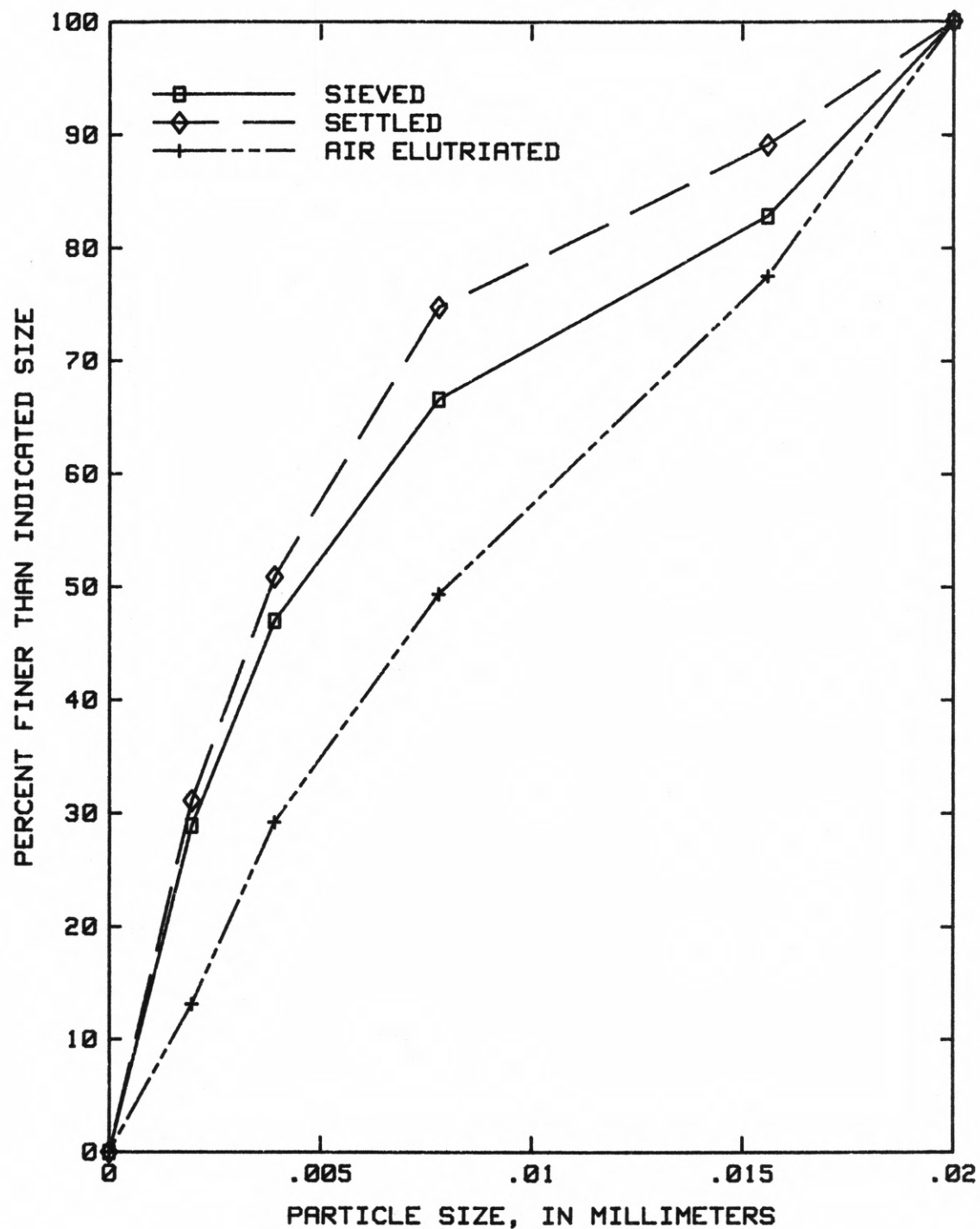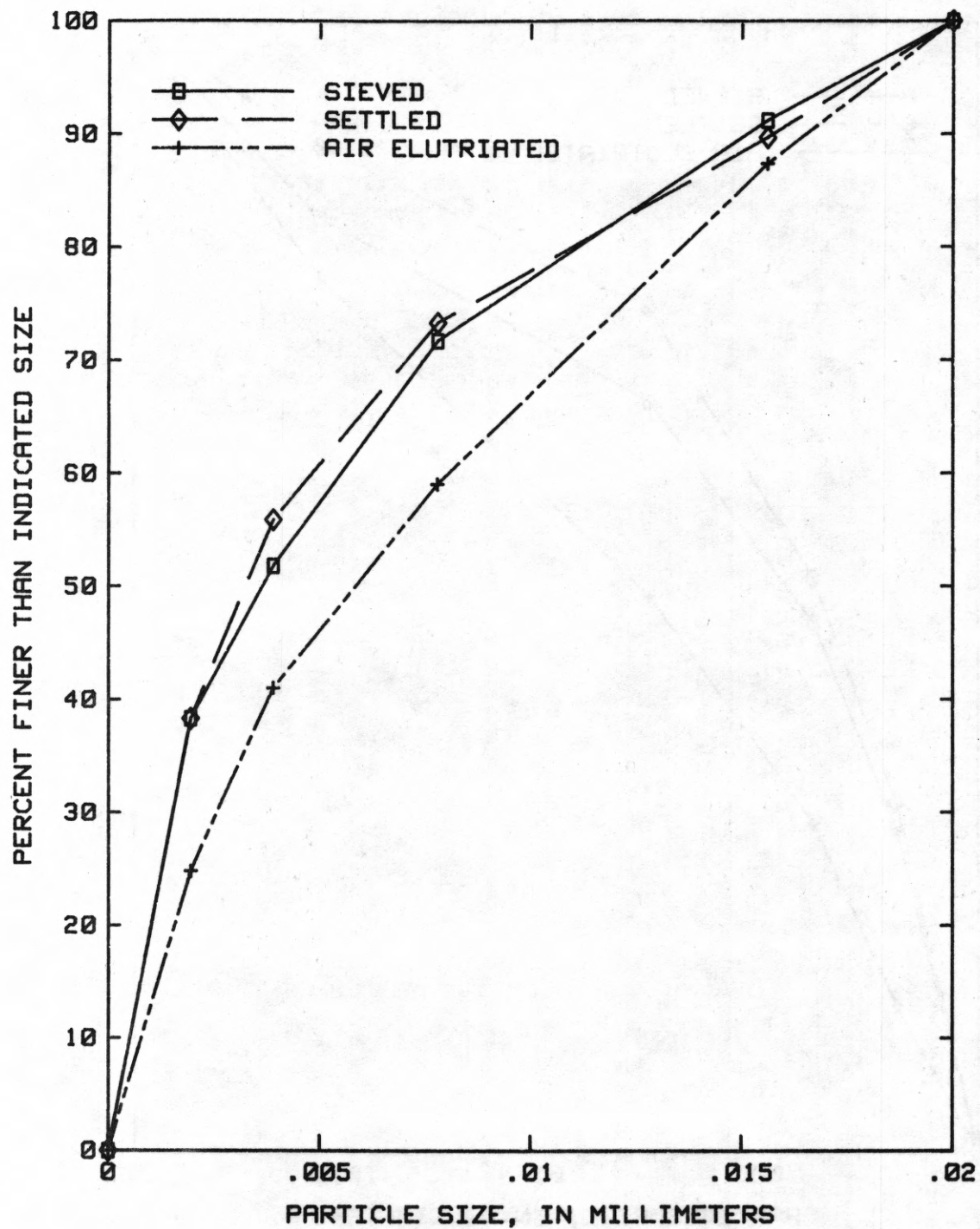
Figure 4.--Particle-size diameter and percentage of particles finer than indicated size.

SAMPLE 2

Figure 4.--Particle-size diameter and percentage of particles finer than
indicated size (Continued).

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Figure 4.--Particle-size diameter and percentage of particles finer than

indicated size (Continued).

Concentration data for the three size-fractioning techniques were combined and the concentrations for each metal were individually ranked from smallest to largest. Statistical tests were then performed on the ranks of the data, rather than on the data themselves. By doing this, few assumptions about the underlying distribution of the data are necessary (Conover and Iman, 1981).

A two-way analysis of variance (ANOVA) was performed on the ranks of the combined concentration data to determine whether there were significant differences in the median concentrations of copper, chromium, lead, iron, zinc, or manganese between sites. Lead, chromium, iron, zinc, and manganese all were found to have significantly different concentrations. Copper was the only metal for which between-site differences were not significant.

Significant between-technique differences in median copper, iron, and lead concentrations also were identified in the combined ANOVA. No significant differences in median chromium, manganese, or zinc concentrations were indicated. Nonsigificance of between-technique differences does not guarantee that median concentrations are equal, but implies only that any differences are too small to discern at the chosen level of significance with the amount of data collected.

The Ryan-Einot-Gabriel-Welsh multiple comparison test (SAS Institute, 1982) was used to assess the nature of between-technique differences identified in the analysis of variance. The median concentration of copper in the air-elutriated samples was found to be significantly higher than that measured in the sieved and settled samples. Table 1, which lists the median metal concentrations at each site, shows that median copper concentrations observed with the air-elutriated samples were approximately three to nine times higher than those observed with the other size-fractioning techniques.

These results strongly suggest that the air-elutriated samples somehow were contaminated. An unrelated investigation conducted by the Water Chemistry Section of the U.S. Geological Survey's Office of the Southeastern Regional Hydrologist identified a metallic feed tip on the air elutriator as a potential source of copper, zinc, cadmium, and nickel contamination (A. J. Horowitz, U.S. Geological Survey, written commun., 1984). Copper, zinc, and cadmium contamination was eliminated in that investigation by replacing the metallic feed tip with one made of nylon; however, some residual nickel contamination remained. No evidence of zinc contamination was found in our analyses.

Lead and iron concentrations tended to be highest in sieved bottom-material samples, followed by settled and air-elutriated samples in that order. Significant differences in median lead and iron concentrations were observed between the sieved and air-elutriated bottom-material samples. Settled samples generally

Table 1.--<u>Median metal concentrations in bottom material</u>

[All concentrations represent total recoverable, in micrograms per gram of bottom material]

Sample type	Site[1]	Chro-mium	Cop-per	Iron	Lead	Man-ganese	Zinc
Sieved	P	10	20	26,000	40	1,200	170
	S	20	30	64,000	70	4,400	270
Settled	P	20	10	28,000	30	970	130
	S	20	30	53,000	50	2,700	200
Air elutri-ated	P	10	90	15,000	30	1,000	180
	S	20	80	37,000	40	4,100	200

[1]P, Pine Run near Fredericktown, Ohio; S, West Fork Stateline Creek near Negley, Ohio

exhibited median concentrations somewhere between those observed in sieved and air-elutriated samples, and could not be declared significantly different from either. Table 2 illustrates these relationships.

For each individual size-fractioning technique, a Kruskal-Wallis test was run to assess between-site differences in median metal concentrations. Between-site differences were found to be significant for five of the six metals analyzed in samples that were either sieved or air elutriated. No significant between-site differences were detected in samples that were size fractioned by settling. Significant between-site differences in the median concentrations of copper, iron, lead, manganese, and zinc were identified in sieved bottom-material samples. Median copper, chromium, iron, lead, and manganese concentrations were found to differ significantly between sites in the air-elutriated samples.

The probabilities that median metal concentrations were equal at the two sites were computed in the Kruskal-Wallis test for samples fractioned by each of the individual techniques. Table 3 reports the resulting probability of site medians being equal. Small probabilities indicate that median concentrations at the two sites are not likely to be equal.

Table 3 shows that the probability of air-elutriated sample medians being equal was generally lower than that associated with either the sieved or settled samples. This implies that the air-elutriated samples discriminated between concentrations at the two sites with a higher degree of certainty.

Statistical tests that assessed differences in median copper concentrations for the air-elutriated samples have questionable validity due to the likelihood that the air-elutriated samples were contaminated with copper; however, the tests for between-technique differences in median copper concentrations are still valid.

COSTS AND EASE OF APPLICATION OF SIZE-FRACTIONING TECHNIQUES

Quality of data is paramount; however, processing costs and ease of use also are important factors in the selection of a size-fractioning technique. Total costs associated with a size-fractioning technique are a function of the time spent processing a sample and any additional costs for special size-fractioning procedures. Table 4 shows a breakdown by technique of the average time spent processing samples and the fixed costs associated with special size-fractioning procedures. The average processing times include all steps necessary to prepare the samples for transport

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Table 2.--Results of Ryan-Einot Gabriel Welsh multiple F test

[For a given metal, sample types having common letters were not significantly different.]

Sample type	Metals					
	Chromium	Copper	Iron	Lead	Manganese	Zinc
Sieved------------	A	A	A	A	A	A
Settled-----------	A	A	AB	AB	A	A
Air elutriated----	A	B	B	B	A	A

Table 3.--Probability that median metal concentrations are equal at the Pine Run and West Fork Stateline Creek sites

Metal	Sample type		
	Sieved	Settled	Air elutriated
Chromium-----------	0.166	0.699	0.050
Copper-------------	.046	.131	.041
Iron---------------	.047	.075	.009
Lead---------------	.019	.192	.015
Manganese----------	.008	.094	.007
Zinc--------------	.008	.072	.205

Table 4.--Mean sample-processing times and costs associated with special fractioning procedures

[NA, not applicable]

Size-fractioning technique	Mean processing time (minutes)	Costs for special fractioning procedures (dollars)
Sieving---------------	47	NA
Settling-------------	57	NA
Air elutriation-------	9	30*

*Price as of May 1984

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to a laboratory for chemical analysis. Several factors, such as the particle-size distribution and the amount of detritus present in the bottom materials, may greatly influence the processing times.

Sieving is less expensive than settling at a given hourly rate of pay, assuming that the processing times given in table 4 are representative (or are at least in the proper ratio) and that equipment costs are not considered. For the processing times and fixed costs given in table 4, the settling and sieving techniques are less expensive than the air-elutriation technique at hourly rates of pay less than $37.50 and $47.37, respectively.

The size-fractioning techniques differed in their ease of use. Field processing of bottom-material samples was easiest for samples size fractioned by air elutriation. The air-elutriation procedure requires a minimal amount of sample preparation, and as an added benefit, the particle-size distribution of the samples can be easily determined at the same time (at an additional cost). There is a tradeoff between the relative ease of using the air-elutriation technique and its cost. However, if a particle-size analysis of the bottom materials is desired, this technique becomes more cost effective. The settling and sieving techniques were roughly equal in their ease of application, although one technique may be easier to use than the other depending on the physical nature of the bottom materials.

SUMMARY AND CONCLUSIONS

Bottom-material samples were collected from two streams located in northeastern Ohio. The <0.020-mm fraction obtained by each of three size-fractioning techniques (sieving, settling, and air elutriation) was analyzed for total recoverable chromium, copper, lead, iron, zinc, and manganese.

An analysis of variance of the combined concentration data indicated that the median concentration of chromium, iron, lead, zinc, and manganese differed significantly between sites. Between-site differences in copper concentrations, if present, may have been obscured by copper contamination in the air-elutriated samples.

Statistically significant between-technique differences in median copper, iron, and lead concentrations also were identified in the combined ANOVA. No significant between-technique differences in median chromium, manganese, or zinc concentrations were indicated.

Median concentrations of copper in air-elutriated samples were found to differ significantly from those observed in sieved and settled samples. The observed differences are probably caused by contamination of air-elutriated samples by a metalic feed tip on the elutriator apparatus.

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Lead and iron concentrations tended to be highest in sieved samples, followed by settled and air-elutriated samples, in that order. Significant differences in median lead and iron concentrations were observed between the sieved and air-elutriated bottom-material samples. Settled samples generally exhibited median concentrations somewhere between those observed in sieved and air-elutriated samples, and were not significantly different from either.

Samples size fractioned by sieving or air elutriation displayed statistically significant between-site differences in the median concentrations of five of the six metals. No significant differences in median metal concentrations were observed between sites in samples that had been size fractioned by settling.

Probability values computed in the Kruskal-Wallis test suggest that the air-elutriated samples discriminated between concentrations at the two sites with a higher degree of certainty than either sieved or settled samples. This result, however, cannot be interpreted as meaning that air-elutriated samples will always provide a superior level of discrimination. Further investigation is required to test that hypothesis. Samples fractioned by sieving also provided an adequate level of discrimination, however, the samples fractioned by settling did not.

The particle-size distributions of bottom materials fractioned by sieving, settling, and air elutriation were determined for three samples collected from nine central Ohio streams. Graphical analyses suggest that the three size-fractioning techniques may not produce equivalent particle-size distributions. This has not been statistically supported, however.

Under the conditions of this experiment, air elutriation was the easiest to apply and most costly of the three size-fractioning techniques. The sieving and settling techniques were roughly equal in their ease of application; sieving was the least costly of the two alternatives.

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Table 5.--<u>Metal-concentration data from the Pine Run and West Fork Stateline Creek sites</u>

Site	Cross-section position	Frac-tioning method	Metal concentrations, in micrograms per gram of bottom material					
			Chromium	Copper	Iron	Lead	Manganese	Zinc
Pine Run near Fredericktown, Ohio--------	1	SV	10	20	21,000	40	1,200	150
		ST	20	20	28,000	50	1,300	170
		AE	10	90	17,000	30	1,000	180
	2	SV	20	20	33,000	40	1,300	170
		ST	20	10	28,000	30	930	100
		AE	10	110	15,000	30	1,100	200
	3	SV	10	10	20,000	40	820	110
		ST	10	9	14,000	20	810	110
		AE	10	80	12,000	40	1,000	170
	4	SV	20	20	66,000	40	1,200	170
		ST	20	20	33,000	50	3,800	250
		AE	10	110	14,000	30	1,000	190
	5	SV	10	20	26,000	40	1,200	170
		ST	10	10	18,000	30	970	130
		AE	10	90	18,000	30	1,000	180
West Fork Stateline Creek near Negley, Ohio----	1	SV	20	40	72,000	60	4,600	270
		ST	20	30	84,000	60	2,700	270
		AE	10	60	37,000	40	4,100	200
	2	SV	50	50	64,000	130	3,800	490
		ST	20	20	30,000	50	1,500	200
		AE	20	70	43,000	40	2,600	220
	3	SV	10	20	45,000	70	4,400	190
		ST	20	30	53,000	50	4,400	200
		AE	20	90	43,000	60	6,600	240
	4	SV	20	20	43,000	40	4,700	200
		ST	8	9	22,000	20	1,000	72
		AE	10	80	30,000	40	5,400	190
	5	SV	20	30	74,000	80	1,800	280
		ST	20	30	79,000	60	3,800	280
		AE	20	80	37,000	40	1,600	170

SV = sieved
ST = settled
AE = air elutriated