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SEDIMENT BOUND TRACE METALS IN THE WHITE  
ROCK CREEK WATERSHED, DALLAS  
AND COLLIN COUNTIES, TEXAS

THESIS

Presented to the Graduate Council of the  
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By

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Areas were sampled in the White Rock Creek Watershed to examine sediment bound trace metal distributions and sorption relationships. A Kruskal-Wallis AOV found significant among area differences for most metals, and SNK-like multiple comparisons were used to group these areas. Kruskal-Wallis AOVs similarly found among area differences for sediment components that bind trace metals (Fe and Mn oxides and organic carbon) and physicochemical conditions that influence metal sorption (particle size and pH). Multiple correlation found numerous relationships among trace metals, sediment components, and physicochemical conditions. Statistical relationships indicate that metal partitioning to various sorption factors is metal specific. White Rock Creek Watershed trace metal concentrations are comparable to those in many urban watersheds.

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## CHAPTER 1

### INTRODUCTION

Aquatic environments receive trace metals from point sources, atmospheric fallout, leachate from landfills, and stormwater runoff. Urban runoff in particular transports alarmingly high trace metal concentrations that commonly exceed chronic water quality criteria (U.S.EPA, 1983).

In the fluvial environment, only a small percentage of the total trace metal load is transported in solution. The greatest percentage is sorbed and transported on sediment. Trace metal concentrations in sediment are usually orders of magnitude greater than concentrations in solution (Gibbs, 1977; Luoma, 1988; Horowitz, 1988; Forstner and Whittmann, 1981). Data from worldwide and local studies (Table 1) reflect the differences between metal concentrations in sediment as compared to concentrations in water.

The highest metal concentrations are usually associated with the finer grained particles which have greater surface areas for sorption. However, sediment studies have shown that metal concentrations are not determined entirely by particle size. In some cases, the highest metal

Table 1. Comparison of metal concentrations in water and sediment.

|    | WATER (mg/l)                      |   |  | SEDIMENT (mg/kg)              |                                 |                              |
|----|-----------------------------------|---|--|-------------------------------|---------------------------------|------------------------------|
|    | Background<br>levels <sup>a</sup> | 1700 U.S.<br>spl. <sup>b</sup><br>(max) | Trinity<br>River <sup>c</sup><br>(max) | Trinity<br>River <sup>d</sup> | Texas<br>90th %ile <sup>e</sup> | Western<br>U.S. <sup>f</sup> |
| Cd | 0.00007                           | .120                                    | 0.01                                   | 1-5                           | 3.0                             | NA                           |
| Cr | 0.00050                           | .112                                    | 0.02                                   | 6-150                         | 72.1                            | 20-210                       |
| Cu | 0.00200                           | .280                                    | 0.03                                   | 3-186                         | 40.0                            | 10-110                       |
| Pb | 0.00020                           | .140                                    | 0.13                                   | 7-57                          | 31.8                            | 9-52                         |
| Zn | 0.01000                           | 1.182                                   | 0.18                                   | 17-503                        | 120.0                           | 49-510                       |

<sup>a</sup>Estimated worldwide background levels (Forstner and Whittmann, 1981).

<sup>b</sup>1700 water samples collected throughout U.S. (Kopp and Kroner, 1968).

<sup>c</sup>Water samples collected from 12 areas in the Trinity River, Texas (IAS and others, 1989).

<sup>d</sup>Metal concentrations measured in bulk sediment from 12 areas in the Trinity River, Texas (IAS and others, 1989).

<sup>e</sup>Texas Water Commission bulk sediment 90th percentile (Davis, 1987).

<sup>f</sup>Metal concentrations in <0.063mm particles from nine western U.S. areas (Severson and others, 1987).

concentrations are found with the silt sized fraction (Helsel and Koltun, 1986; Yorke and others, 1985; Dossis and Warren, 1980). The studies which do find that concentrations increase with smaller particles usually find that the relationship is not proportional (Gibbs, 1977; Feltz, 1980). These results demonstrate that although metal concentrations are strongly dependent upon particle surface area, other factors also influence sediment metal concentrations.

Jenne (1977) reported that the primary influence of the fine grained mineral is that it sorbs other sediment bound



components which are in turn attracting trace metals. According to Luoma and Davis (1983), these additional sediment components most important in attracting trace metals are:

- (1) oxides of Mn and Fe,
- (2) organic matter,  
and to a lesser extent,
- (3) clay minerals and,
- (4) carbonates.

Figure 1 illustrates the relationship between a fine grained mineral and metal binding substrates. The intensity that trace metals bind to each substrate is metal specific and individual metal sorption is probably not dominated by any single substrate (Luoma, 1988; Lee and Jones, 1987; Jenne and Zachara, 1987). Studies by Gibbs (1973), Oakley and others (1981), Luoma and Bryan (1981), and Tessire and Campbell (1988) have demonstrated that substrate binding intensities differ for various trace metals.

In addition to differing binding potentials of substrates, the environment's redox and pH will influence metal sorption and partitioning. Reducing conditions will result in solubilization of Fe and Mn oxides, and changes in pH may influence the speciation of metal ions (Luoma, 1988; Rapin and others, 1983). Table 2 summarizes the physiochemical factors and important sediment components

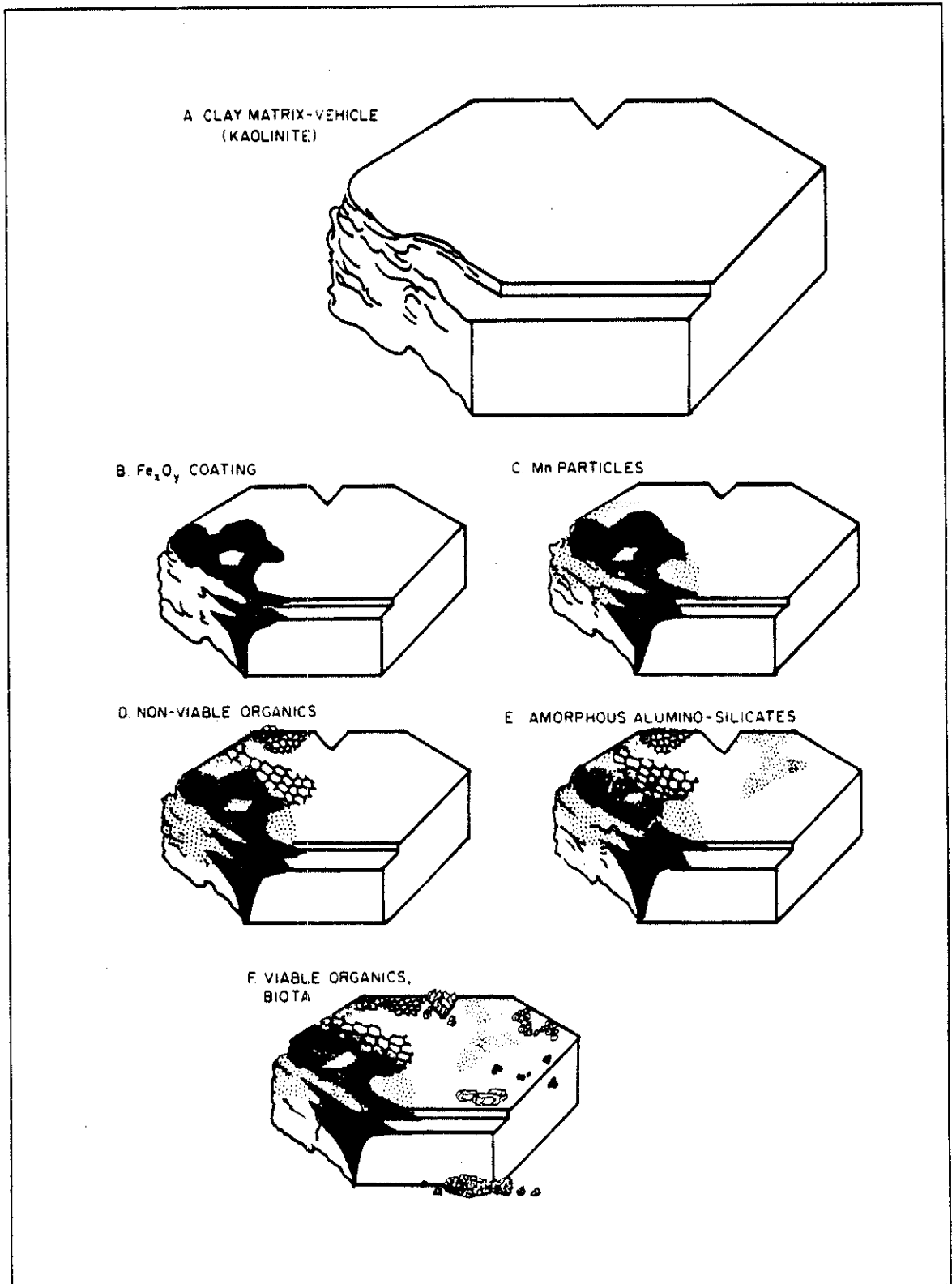


Figure 1. Important metal binding substrates on the surface of a clay mineral (from Jenne, 1977).

that influence trace metal partitioning in aquatic sediments. Factors that are examined in this study are indicated in Table 2.

Table 2. Important factors affecting the partitioning of trace metals in the aquatic environment.

| <u>Sediment Components</u>                 | <u>Physiochemical</u>   |
|--|-------------------------|
| * Oxides of Mn and Fe                      | * Particle size effects |
| * Organic Carbon<br>(and *volatile solids) | * pH                    |
| clay minerals                              | redox potential         |
| carbonates                                 |                         |

---

\* factors examined in this study.

Volatile solid measurements are also included (Table 2) in order to evaluate its frequently observed relationship with organic carbon (Kelly and Hite, 1981; Luoma and Bryan, 1981; Jaffe and Walters, 1977).

A contaminated particle is carried in suspension until its critical settling velocity is reached and then it is deposited as bottom sediment. After deposition, the contaminated particle either remains permanently as bottom sediment or it is resuspended during storms and transported further downstream (Yorke and others, 1985; Lick, 1987). Moderate to intense storms, therefore can alter sediment metal concentrations at specific locations.

Figure 2 illustrates the variables involved in trace metal transport and the relationships among metal sorption factors. As shown on Figure 2, most metals are transported as sorbates with only a minor fraction carried in solution. The trace metals shown in Figure 2 (zinc, lead, chromium, copper, and cadmium) typically occur in urban runoff (U.S.EPA, 1983) and are evaluated in this study.

Currently, three methods are commonly used to indicate how metals are partitioned among the various sediment components. As reported by Horowitz (1988) these methods are: 1) partial chemical extractions 2) physical separation of various phases followed by chemical analysis, and 3) statistical analysis of bulk sediment data (the method used in this study).

Many attempts with selective extractions have been made to quantitatively answer questions about partitioning such as, "How much lead is sorbed to the organic carbon or iron oxide sediment component?" Currently, these questions are problematic because available extraction methods are highly nonspecific (Luoma and Bryan, 1983), and trace element redistribution occurs during extraction (Kheboian and Bauer, 1987). Metal concentrations determined with partial extractions are therefore operationally defined and only the metal's "extractable phase" is being measured (Luoma and Jenne, 1976; Jenne and Zachara, 1987; Horowitz, 1988).

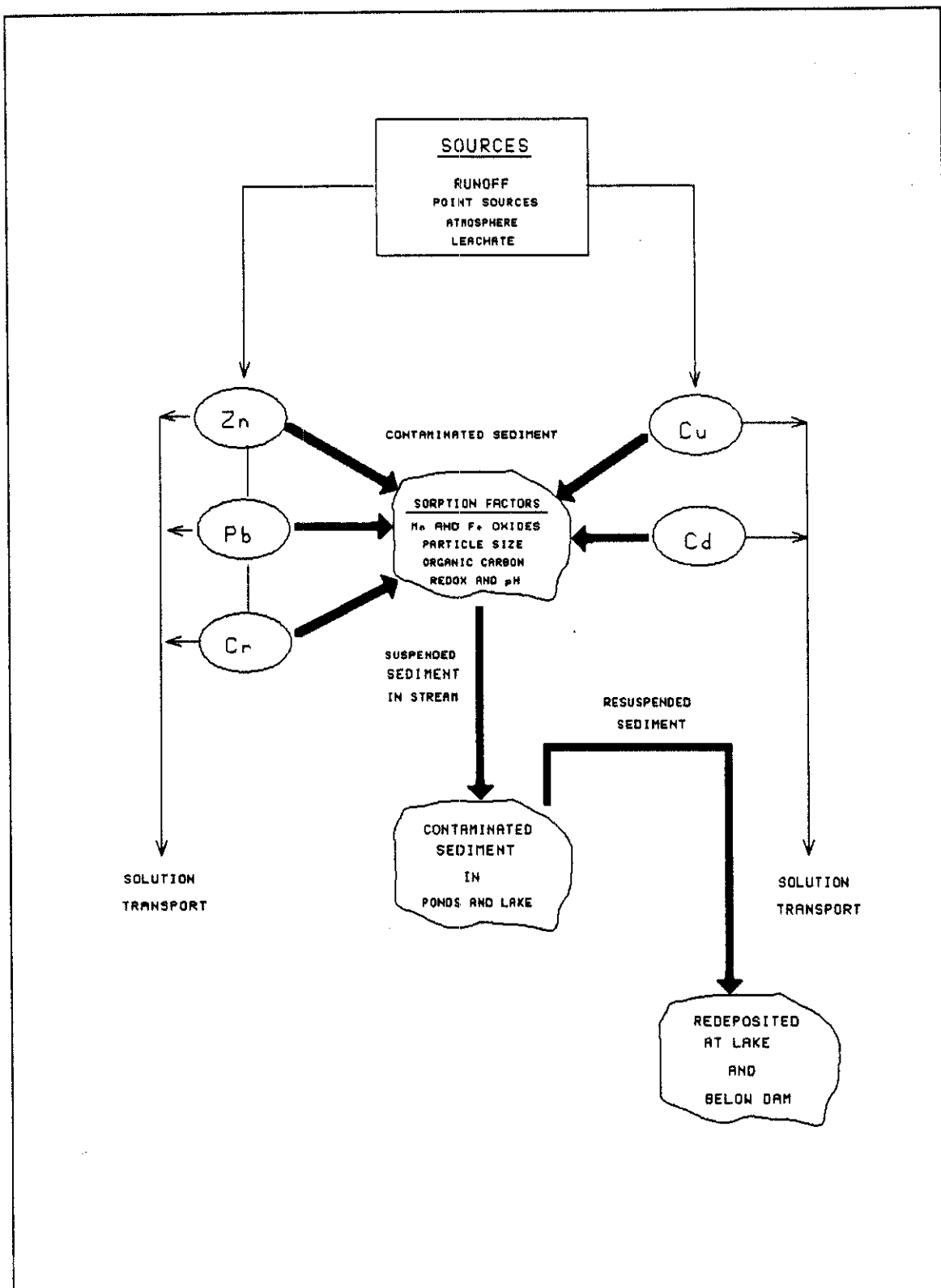


Figure 2. Important factors in transporting, distributing, and partitioning trace metals.

The third method (statistical method) does not determine cause and effect, or attempt to quantitatively define metal partitioning among specific substrates. The statistical method relies upon analytically determined metal-substrate relationships and evaluates the strength of these relationships with correlation coefficients. For example, statistical analyses will answer questions such as, "Does the concentration of lead significantly correlate with the concentration of organic carbon and what is the strength of the relationship?"

The statistical strength of relationships can reflect the potentials among substrates to attract metals (Luoma and Bryan, 1981). Correlation coefficients must be evaluated cautiously however, and can be misleading if relationships are not examined with regard to recognized sorption mechanisms.

When considering each method's assumptions, the statistical method has several advantages. One advantage in particular is that most laboratories can precisely derive bulk sediment data which are then available to evaluate metal distributions and partitioning.

#### Objectives

Sorption factor variability and differing metal binding intensities require that metal distributions and partitioning relationships be determined on a site

specific basis. This study examines these conditions in the White Rock Creek Watershed. Study objectives are further described as follows:

(1) To examine the downstream changes in trace metal concentrations by sampling 5 areas located from the headwaters of White Rock Creek to the White Rock Lake dam (Figure 3).

(2) To compare the various sorption factors (Table 2) among the 5 areas.

(3) To determine if significant correlations exist between all possible pairs of sorption factors and metals by producing a statistical correlation matrix. This will answer questions such as: "Is the concentration of Pb statistically correlated with organic carbon, or is organic carbon correlated with percent volatile solids?"

(4) To compare White Rock Creek Watershed metal concentrations with background values, concentrations from other sediment studies, and with concentrations considered as elevated by the Texas Water Commission (Davis, 1987) and the U.S. Environmental Protection Agency (EPA, 1974).

(5) Finally, a secondary objective is to provide data that are relevant for future bioavailability studies. Luoma (pers. conv., 1989) recommends that basic measurements required for bioavailability studies should include:

a. measurement of total metals concentrations

- b. metals concentrations determined by 0.5N HCl cold digestion
- c. measurements of major sediment components that affect bioavailability including total organic carbon and concentrations of Fe and Mn.

As shown in Table 2, these recommended measurements, with the exception of the cold HCl digestion for determining metals are evaluated in this study.

#### Optimal Watershed Characteristics

The White Rock Creek Watershed has several physical characteristics that makes it exceptional for investigating sediment trace metal distributions. Some of the more optimal characteristics include:

(1) The watershed does not have complex landuse patterns. It is 85 percent urban, and conditions should be representative of many urban watersheds.

(2) The watershed is incised into only one geologic formation, the Austin Chalk, which consists entirely of alternating calcareous shale, marl and limestone. Water chemistry is therefore not substantially affected by contrasting bedrock lithology. For example, buffering capacity differences between sandstone and carbonate bedrock are not observed and clay mineralogy is not excessively variable.



(3) The watershed is 36.8 km (22.9 mi) long and covers 256.7 km<sup>2</sup> (99.1 mi<sup>2</sup>). Because of the relatively intermediate size, a thorough sampling program can be conducted from the headwaters to White Rock Lake dam.

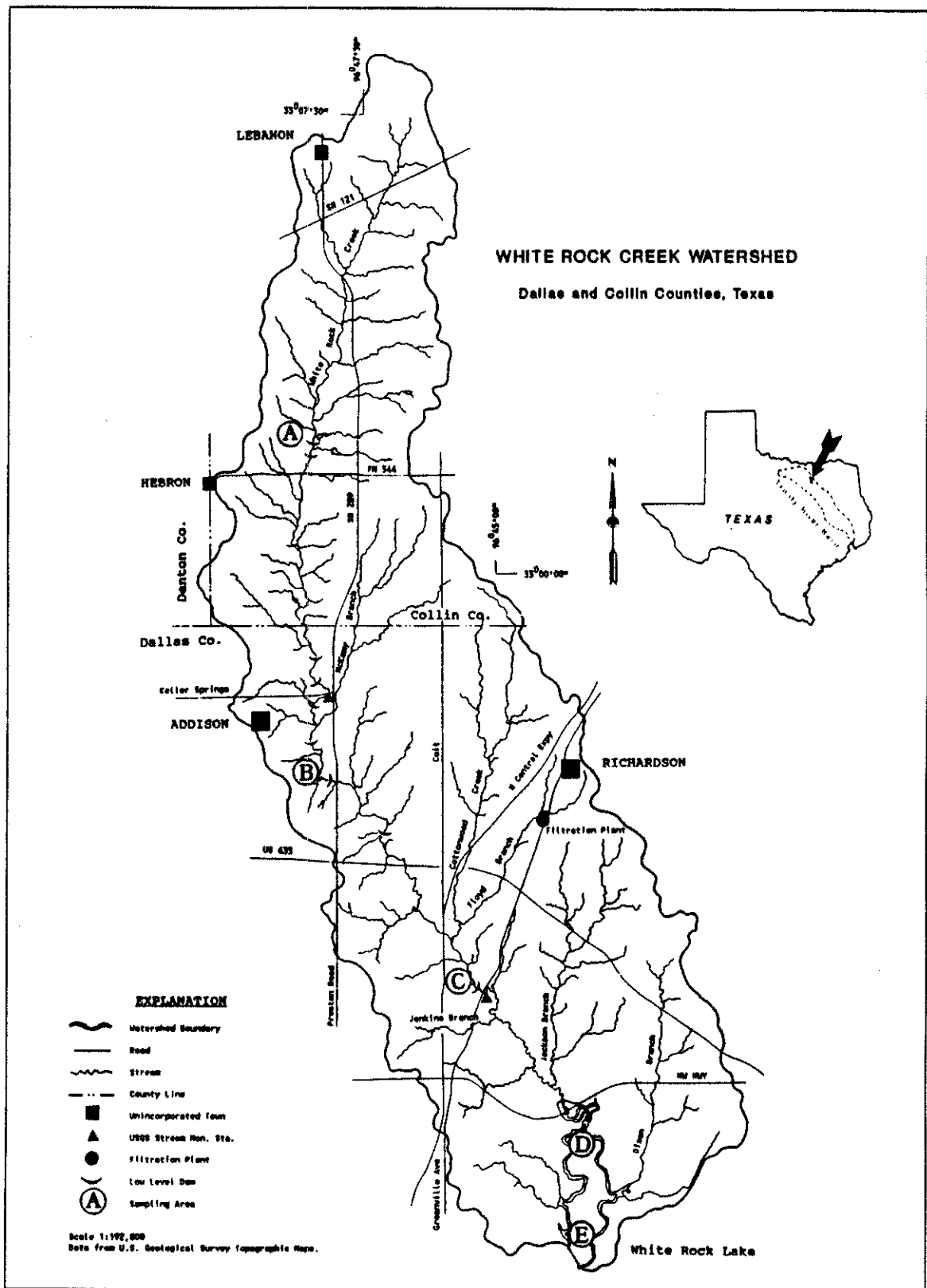


Figure 3. White Rock Creek Watershed.

## CHAPTER 2

### PREVIOUS WORK

A pilot study during June, 1988 evaluated metal concentrations and sediment characteristics at White Rock Lake. Laboratory analyses of fifteen sediment samples collected from various depositional areas determined that metal concentrations are typical of those reported for other urban lakes (Kelly and Hite, 1981). Additionally, it was found that sediment metals have a low degree of variability throughout the lake. Coefficients of variation for lead, cadmium, zinc, and copper ranged from 7.2 to 12.9 percent.

With the exception of the White Rock Lake pilot study, contaminated sediments in the watershed have not been studied. However, there have been studies in reference to:

- (1) Sedimentation surveys at White Rock Lake by the USDA Soil Conservation in 1935, 1956, 1970, 1977, and 1984,
- (2) flood control projects, and
- (3) City of Dallas annual stream water surveys.

Brief summaries of these studies are included in following sections.

## White Rock Creek Watershed Studies

### USDA Soil Conservation Service Sediment Surveys

The USDA Soil Conservation Service (SCS) has conducted extensive White Rock Lake sedimentation surveys in 1935, 1956, 1970, 1977, and 1984 (Arnold and others, 1987). Descriptions of the surveys can be found in Eakin (1939), Marshall and Brown (1939), Ogle (1956), Zodin (1970) and, Arnold and others (1987). The SCS surveys provide considerable information on sediment yield, water yield, and sediment bulk density. Their primary objectives were to determine sedimentation rates, reservoir capacity loss, characteristics of sediment deposition, and the effects of conservation (Zodin, 1970). Because the principal interests dealt with sedimentation rates and reservoir capacity loss, sediment analyses (other than bulk density) were not performed.

The lake has been selectively dredged on at least two occasions. Zodin (1970) reports that in the late 1930's, a "limited section" of the lake was dredged and the spoils were deposited near the Mockingbird Lane overpass west abutment (near sample area D, Figure 3). Mike Byerly, supervisor of White Rock Lake, reports that a few limited areas were dredged during the 1980's (pers. conv., 1988).

### City of Dallas Annual Stream Water Surveys

Since 1971, the City of Dallas Environmental Health Department has monitored water quality in the Dallas area's major streams (Becker, 1987). Out of approximately twenty-five monitored streams, six streams are in the White Rock Watershed and include McKamy Branch, Cottonwood Creek, Floyd Branch, Jackson Branch, White Rock Creek, and Dixon Branch (Figure 3).

Water samples are analyzed for BOD, coliform bacteria, percent oxygen, pH, turbidity, phosphates, and nitrates. Visual evaluations of stream quality are included and biological diversity is briefly noted. Sediment samples however, are not collected.

### Flood Plain Studies

Several engineering studies that describe White Rock Creek's flood plain have been prepared (A.H. Halff and Associates, 1976; Federal Emergency Management Agency, 1982; A.H. Halff and Associates, in press). In the latest study, stream profiles show the locations and vertical dimensions of White Rock Creek's low level dams (A.H. Halff and Associates, in press).

### Geology, Geomorphology, and Soils

The area's geology was first recorded by Roemer (1848) while evaluating the area for German immigration. Following

Roemer's report, were publications by Shumard (1853) and Marcou (1858). Next, the results of R.T. Hill's monumental work at unravelling the region's Cretaceous stratigraphy were published (Hill, 1901). Further geology and geomorphology publications are by Shuler (1918), Allen and Flanigan (1986), Eubank (1965), and St. Clair (1978).

The USDA Soil Conservation Service has published soil surveys covering the watershed's northern section in Collin County (USDA, 1969), and the watershed's southern section in Dallas County (USDA, 1980).

#### Sediment Studies From Other Areas

Research on trace metal partitioning has been "rather sporadic over the last two decades" (Jenne and Zachara, 1987). Also, designs among individual projects have been quite variable; undoubtedly because of the relatively complex interactions between metals and sorption factors. Jenne and Zachara (1987) state that this complexity has led to the "current inability to quantitatively predict metals sorption". They conclude, however, by stating that "recent interest in this area is encouraging, and a (current) review of the factors affecting metal sorption is timely".

Recent and comprehensive reviews of sediment trace metal studies are found in Horowitz (1988), Dickson and others (1987), Feltz (1980), Moore and Ramamoorthy (1984), and Forstner and Whittmann (1981).

Researchers evaluating the various extraction techniques for partitioning are among others, Kheboian and Bauer (1987), Helsel and Koltun (1984), Tessier and Campbell (1988), Tessier and others (1979), Luoma and Jenne (1976), and Guy and others (1978).

With an experimental design similar to the one used in this study, Yorke and others (1985) defined distributions of sediment, trace metals, and organic substances in Pennsylvania's 4921 km<sup>2</sup> (1900 mi<sup>2</sup>) Schuylkill Basin. Although the Schuylkill Basin is considerably larger than the White Rock Creek Watershed which is 256.7 km<sup>2</sup> (99.1 mi<sup>2</sup>), sample areas were similarly distributed from the upper to lower basin sections. Sediment samples were collected at 6 low level dams and statistical analyses were applied to bulk sediment data.

In the Schuylkill Basin study, relationships between particle size and metal concentrations were evaluated with a Wilcoxon signed-rank test. Results indicated that concentrations of copper, lead, zinc and trace organics were significantly higher (LOS = 0.05) in all particles finer than 0.062 mm fraction (coarse silt - clays) than concentrations in the finer than 0.016 mm fraction (fine silt - clays). Their results suggested that the coarse silts adsorb as much or more trace constituents as the very fine silts and clays. As they reported, "the results of the

analyses were unexpected because other investigators have found that the concentrations of trace metals and organic compounds in soils and sediments were associated very closely with the percentage of clay". These results illustrate that particle surface does not singularly control metal sorption. Rather, sorption is determined from interactions among particle surface and sorption factors.

In an Ohio stream sediment study, Helsel and Koltun (1986) also found that the highest metal concentrations were not strictly associated with the finest particles. Metal concentrations were measured after the sediment was fractioned into three size classes of sand ( $<2$  mm - 0.063 mm), coarse silt ( $<0.063$ mm - 0.020 mm), and fine silt and clay ( $<0.020$  mm). At one of the two sample areas, mean concentrations of copper, lead, and zinc were uncommonly higher in the coarse silt size fraction. A parametric AOV found significant differences in mean metal concentrations between and within the two areas for zinc, lead, chromium, manganese, and copper.

Most frequently however, the highest metal concentrations are associated with the finer grained sediments. Striegl (1987) studied trace metal concentrations in a 4.13 ha (10.2 ac) urban lake at Glen Ellyn, Illinois. Sediments were fractioned into sand (0.7 - 0.063 mm) and a silt-clay group ( $<0.063$  mm). Cadmium, copper, iron, lead,



and zinc concentrations were consistently higher in the smallest fraction (silt-clay group).

Researchers that have applied statistical methods for evaluating metal partitioning are Luoma and Bryan (1981), Oakley and others (1981), Gibbs (1977), Jaffe and Walters (1977), and Iskandar and Keeney (1974). Remarkably close correlations were found in the Humber Estuary of northern England between organic carbon and the sediment bound trace metals Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, and Pb (Jaffe and Walters, 1977). Correlation coefficients between organic carbon and individual metals ranged from 0.89 to 0.97. Metal concentrations were highly correlated with the clay-silt sediment fraction ( $r=0.70$  to  $0.92$ ).

Luoma and Bryan (1981) used statistical analyses similar to those used in this study to indicate relationships among sediment substrates and trace metals. Correlation coefficients were calculated for all possible combinations of substrate and trace metal concentrations measured in bulk sediment. A statistical filtering technique and correlation coefficients were then used to indicate the relative strength of the significant relationships. Their results showed that among substrates, the strongest correlation was between total organic carbon and extractable Fe ( $p < 0.001$ ,  $r=0.72$ ). The correlation between particle size and Fe was significant ( $p < 0.005$ ,  $r=0.47$ ), and the

correlation between particle size and organic carbon was also significant ( $p < 0.001$ ,  $r = 0.65$ ). Correlations between individual trace metals and substrates are illustrated in Figure 4 where arrow widths signify the strength of the statistical relationships. As shown in Figure 4, statistical relationships reflect various substrate potentials for binding specific metals.

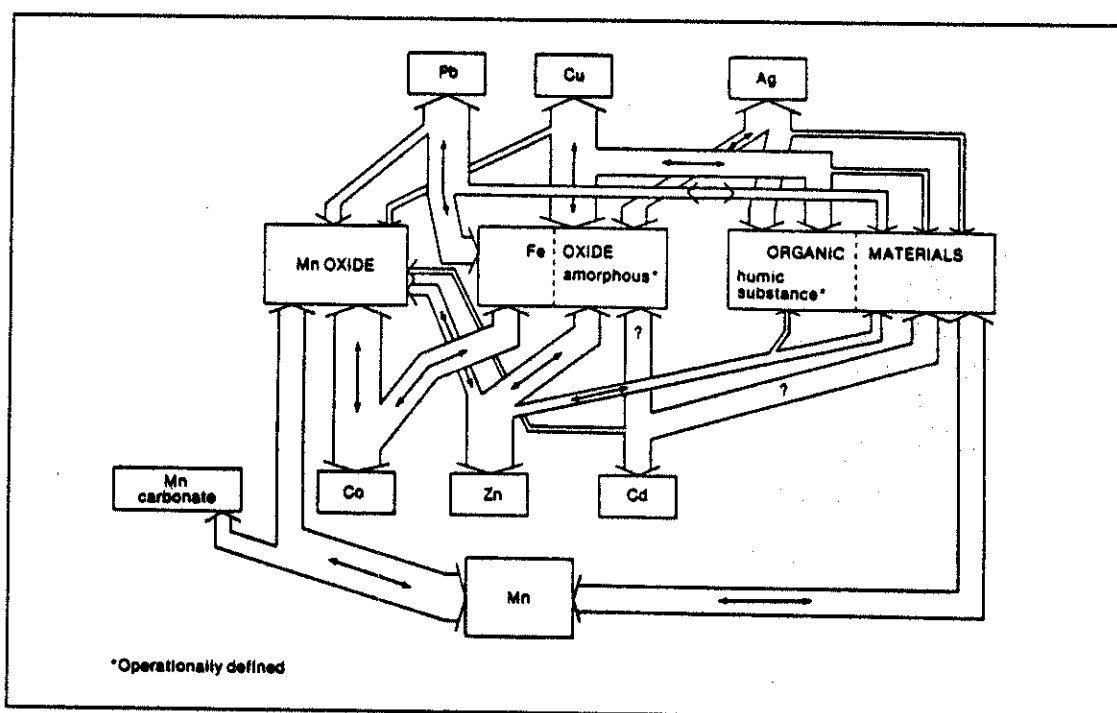


Figure 4. Partitioning of trace metals among binding components important in oxidized sediments, as indicated by statistical associations (from Luoma and Bryan, 1981).

Sediment studies reporting metal concentrations that can be compared with White Rock Creek Watershed concentrations are by: Kopp and Kroner (1968), Iskandar and Keeney (1974), Striegl (1987), Kelly and Hite (1981), and

Forstner and Whittmann (1981). More locally, several studies contain sediment metal data for comparison. A recent study by the University of North Texas and the University of Texas at Dallas contains sediment metal data from twelve stations in the Trinity River (IAS and others, 1989). Other local sources include the Texas Water Commission statewide monitoring database (Davis, 1987) and the U.S. EPA data for EPA Region VI (U.S. EPA, 1974).

## CHAPTER 3

### STUDY AREA

The following sections describe the White Rock Creek Watershed and contain additional information on White Rock Creek and White Rock Lake.

#### Watershed Description

The White Rock Creek Watershed is located in north central Texas between latitudes  $33^{\circ} 10'$  and  $32^{\circ} 45'$ , and longitudes  $96^{\circ} 40'$  and  $96^{\circ} 50'$  (Figure 3). As shown by the inset on Figure 3, the watershed is a subbasin of the major Trinity River Basin. The watershed drains the extreme southwestern corner of Collin County and the north central part of Dallas County.

Including the lake's surface area, the watershed covers  $256.7 \text{ km}^2$  ( $99.1 \text{ mi}^2$ ), is 36.8 km (22.9 mi) long, and averages 6.4 km (4 mi) wide. White Rock Creek has five major tributaries (Figure 3), with Cottonwood Creek draining the largest area and Floyd Branch draining the smallest area (Table 3).

White Rock Creek originates near the township of Lebanon and flows southerly across southeastern

Collin County. A photograph in Appendix A shows White Rock Creek near the Dallas-Collin County line at Frankford Road. Mean sea level at the headwaters is 224.9 m (738 ft) and decreases to 127.1 m (417 ft) in the stream channel behind the dam. The total relief is 97.8 m (321 ft) and is illustrated in a longitudinal profile (Figure 5).

Ten-low level dams have been constructed in White Rock Creek to serve as golf cart bridges and for aesthetic purposes (Figure 3). The dams vary in size with vertical heights from 0.30-3.66 m (1-12 ft). Photographs in Appendix A show low level dams at Areas A, B, and C.

Table 3. Drainage area of White Rock Creek and its major tributaries (sources: FEMA, 1982; Zodin, 1970).

| <u>Stream</u>                        | <u>Drainage Area</u>  |                       |
|--------------------------------------|-----------------------|-----------------------|
|                                      | <u>km<sup>2</sup></u> | <u>mi<sup>2</sup></u> |
| White Rock Creek (at spillway)       | 256.7                 | 99.1                  |
| Dixon Branch                         | 17.4                  | 6.7                   |
| Jackson Branch                       | 18.4                  | 7.1                   |
| Cottonwood Creek (above Floyd Branch | 22.5                  | 8.7                   |
| Floyd Branch (at Cottonwood Creek)   | 10.6                  | 4.1                   |
| McKamy Branch                        | 17.9                  | 6.9                   |

### Geology

The watershed is incised entirely into the upper Cretaceous age Austin Chalk Formation. Outcrops of Austin Chalk exhibit "white rock" surfaces, the characteristic from which the creek and lake were named.

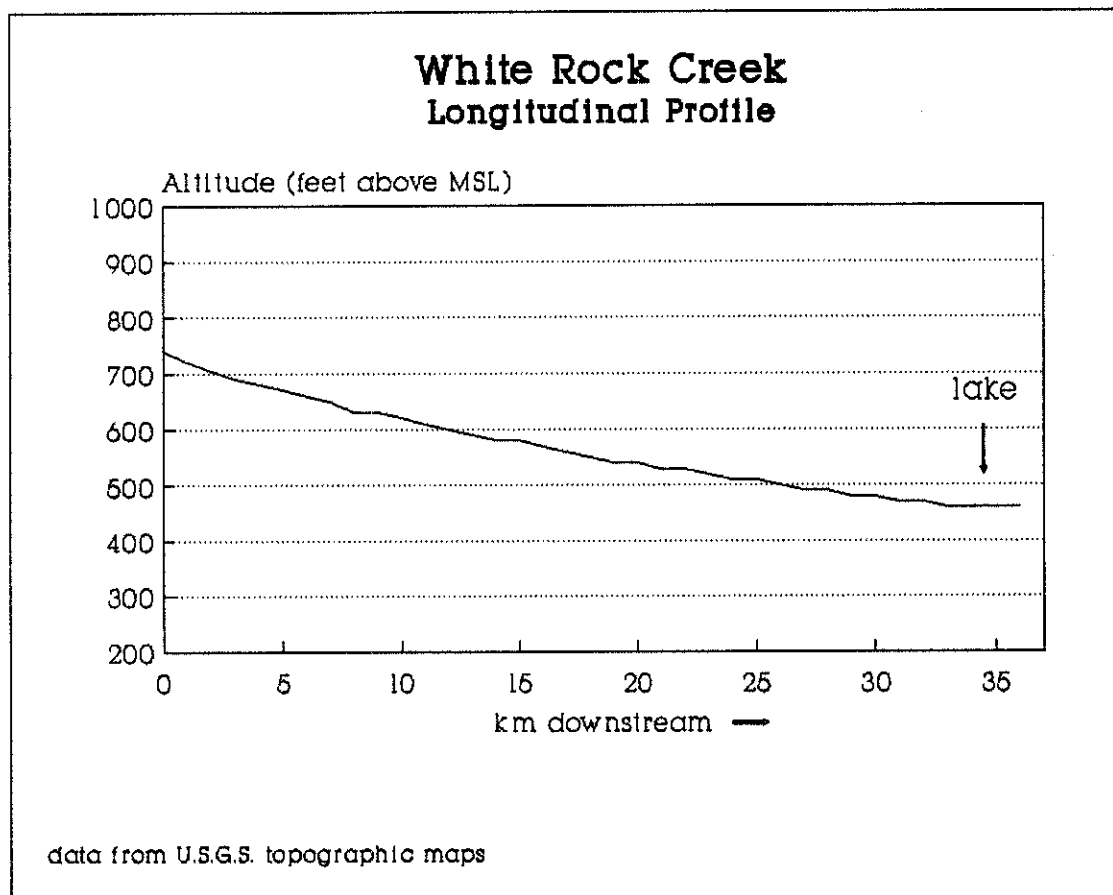


Figure 5. White Rock Creek Longitudinal Profile.

The Austin Chalk Formation consists of 122 m (400 ft) of interbedded calcareous shale, marl and chalky limestone. The upper 1/3 consists predominantly of chalk, the middle 1/3 contains more shale and marl, and the lower 1/3 is again predominantly chalk (Norton, 1965). A correlation chart (Figure 6) shows the Austin Chalk Formation in relation to the overlying Taylor Marl Formation and the underlying Eagle Ford Shale Formation. Figure 6 also illustrates how the lithology defines the upper, middle, and lower units.

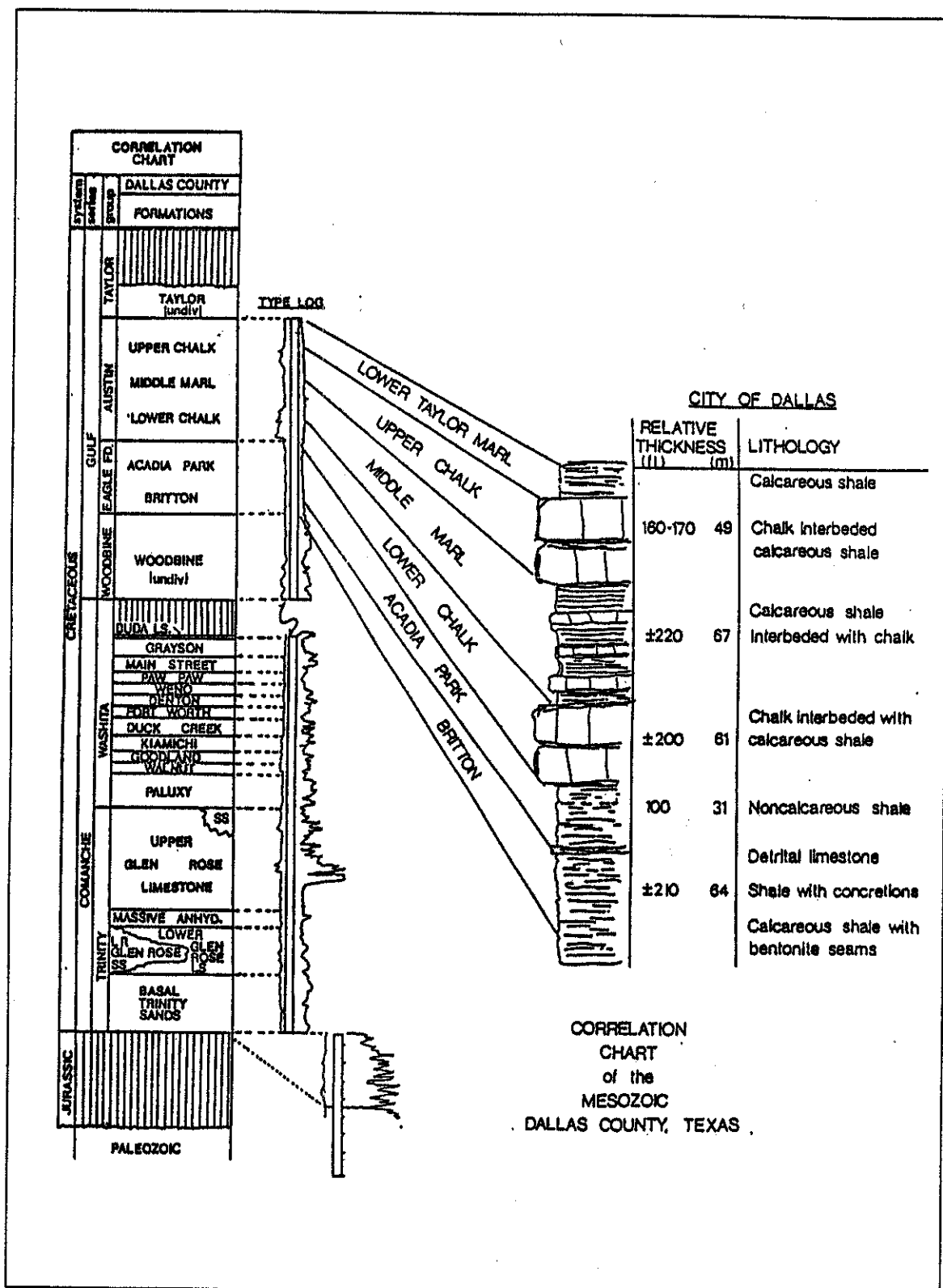


Figure 6. Correlation chart. (source: Allen & Flanigan, 1986 as adapted from Foster, 1965).

A photograph in Appendix A shows a quarry cut in the lower Austin Chalk unit. At this location in watershed's headwater region, massive chalk beds with thin intervening shale beds can be seen.

Beginning at the headwaters, White Rock Creek is incised in the lower chalk unit. As the creek flows southward toward the lake it cuts across progressively higher chalk units. At its confluence with the lake, the creek is incised into the upper unit. Bedrock is commonly exposed along many reaches of the creek because of thin to non-existent sediment deposits (Appendix A, photograph). Upstream from low level dams, bottom sediments accumulate as a result of flow velocity decreases. The amount and characteristics of sediment that accumulates is controlled by pool size (Yorke and others, 1985) and available sediment source. Figure 7 shows particle size distributions at the three stream areas (Areas A, B, and C, Figure 3) and reveals that Area A has considerably more clay and less sand than Areas B and C.

Beginning at Addison, the watershed's lower reaches have thin Quaternary sand and gravel deposits adjacent to the creek's channel. The substantial width of these alluvial deposits and the presence of coarse gravels and sands indicates that the creek was previously much larger with higher flow rates. The sand and gravel have been quarried at 12 sites located along White Rock Creek (St. Clair 1978).



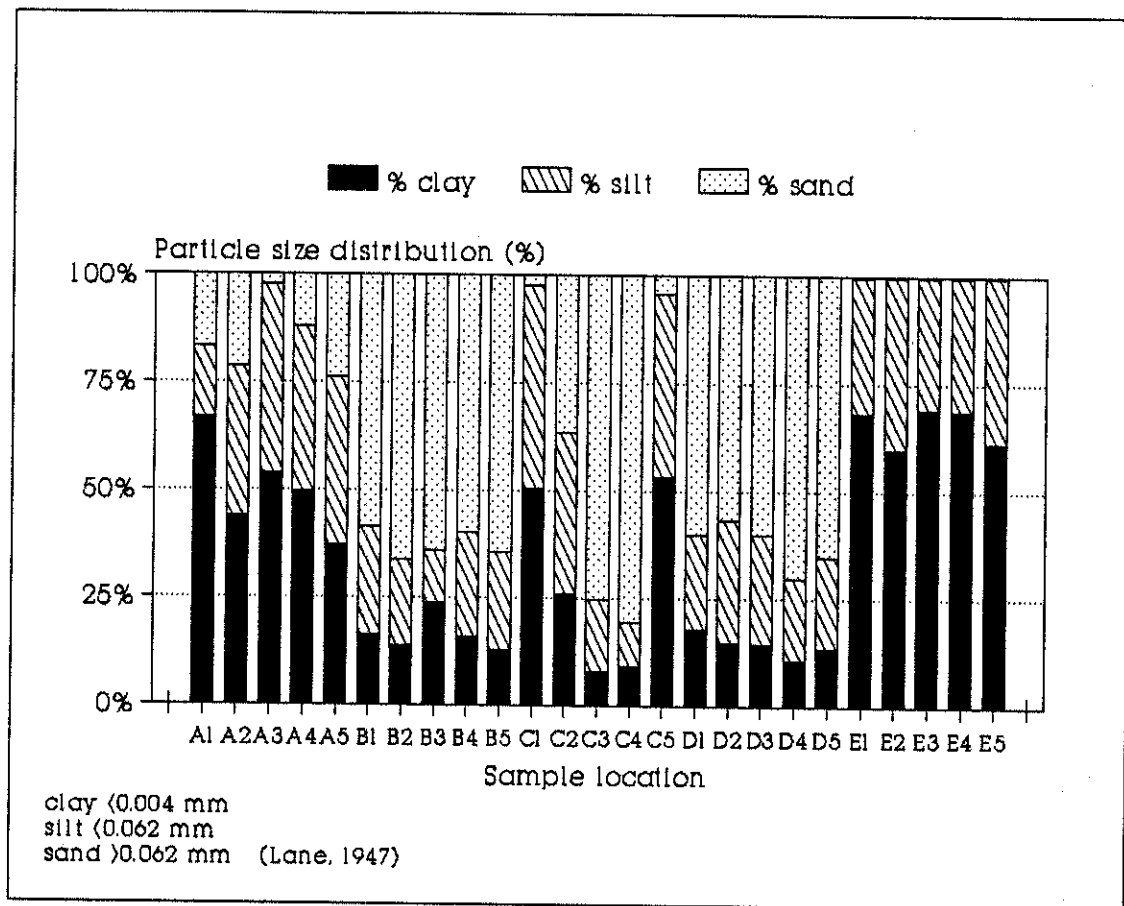


Figure 7. Particle size distributions.

### Soils

Major soils that have formed on the Austin Chalk are in the Houston Black-Austin map unit (USDA-SCS, 1980). Within this map unit, are the Austin and Houston Black soils which together cover approximately 54% of the watershed. The remaining area is covered by several minor soils. The few areas which are not urban continue to use the Houston Black-Austin unit for farming and pasture land. A photograph in

Appendix A shows an agricultural area in the watershed's upper section.

The Austin soils are formed on gently sloping to sloping surfaces, on ridges and knolls, and upland side slopes (USDA-SCS, 1980). In many upland areas, the soil is noticeably thin, and the Austin Chalk bedrock is exposed at the surface (Appendix A, photograph). To depths of 25.4 cm (10 in) the Austin soils are generally dark grayish brown silty clay. Underneath this layer and to a depth of 81.3 cm (32 in) the soils are typically brown silty clay. Underlying this layer, the soils are white and consist of platy chalk.

The Houston Black soils are moderately well drained and are found on nearly level to gently sloping surfaces and in valleys. The Houston Black soils are thicker than the Austin soils and usually less eroded because of occurrence on lower topographic relief. The Houston Black soil's upper layer to a depth of 132 cm (52 in), is very dark gray to black clay. Underneath this layer, and to a depth of 177.8 cm (70 in) the soil is dark grayish brown clay (USDA-SCS, 1980).

#### Reservoir Description

White Rock Lake was built in 1910 to serve as a municipal water source for Dallas, Texas. However, from 1930 to 1953 after other area lakes were built, White Rock Lake was used only for recreation. In 1953 to 1957 during the drought years, the lake was used to supplement municipal

water supplies. In April and May of 1957 heavy rains restored the region's water levels and the lake has since been used for recreation. A photograph in Appendix A shows the lake as viewed from the east.

The dam is located 16.5 km (10.3 mi) upstream from the confluence of White Rock Creek with the Trinity River (Zodin, 1970). The dam is 640 m (2,100 ft) long, is earth filled, and trends northwest-southeast (Figure 8 and Appendix A, photograph). Maximum height above White Rock Creek is 12.2 m (40 ft), maximum base width is 65.2 m (214 ft), and crown width is 6.1 m (20 ft). The lake side of the dam is covered with concrete and the downstream side is covered with gravel and soil.

The spillway is constructed of concrete and is located at the southeastern end of the dam (Figure 8 and Appendix A, photograph). The spillway's length is 137.1 m (450 ft) and has a long broad concrete apron on its downstream section. Originally, the spillway had a cat-walk, piers, and channels that could raise the lake's elevation when flash boards were inserted into the channels. In 1966, the City of Dallas removed the cat-walk and piers and recapped the surface with concrete.

Prior to the 1966 renovation of the dam, the lake's original elevation was 139.43 m (457.45 ft) above mean sea level (MSL). After the 1966 renovation, the lake's elevation increased 0.18 m (0.58 ft) to 139.61 m (458.03 ft).

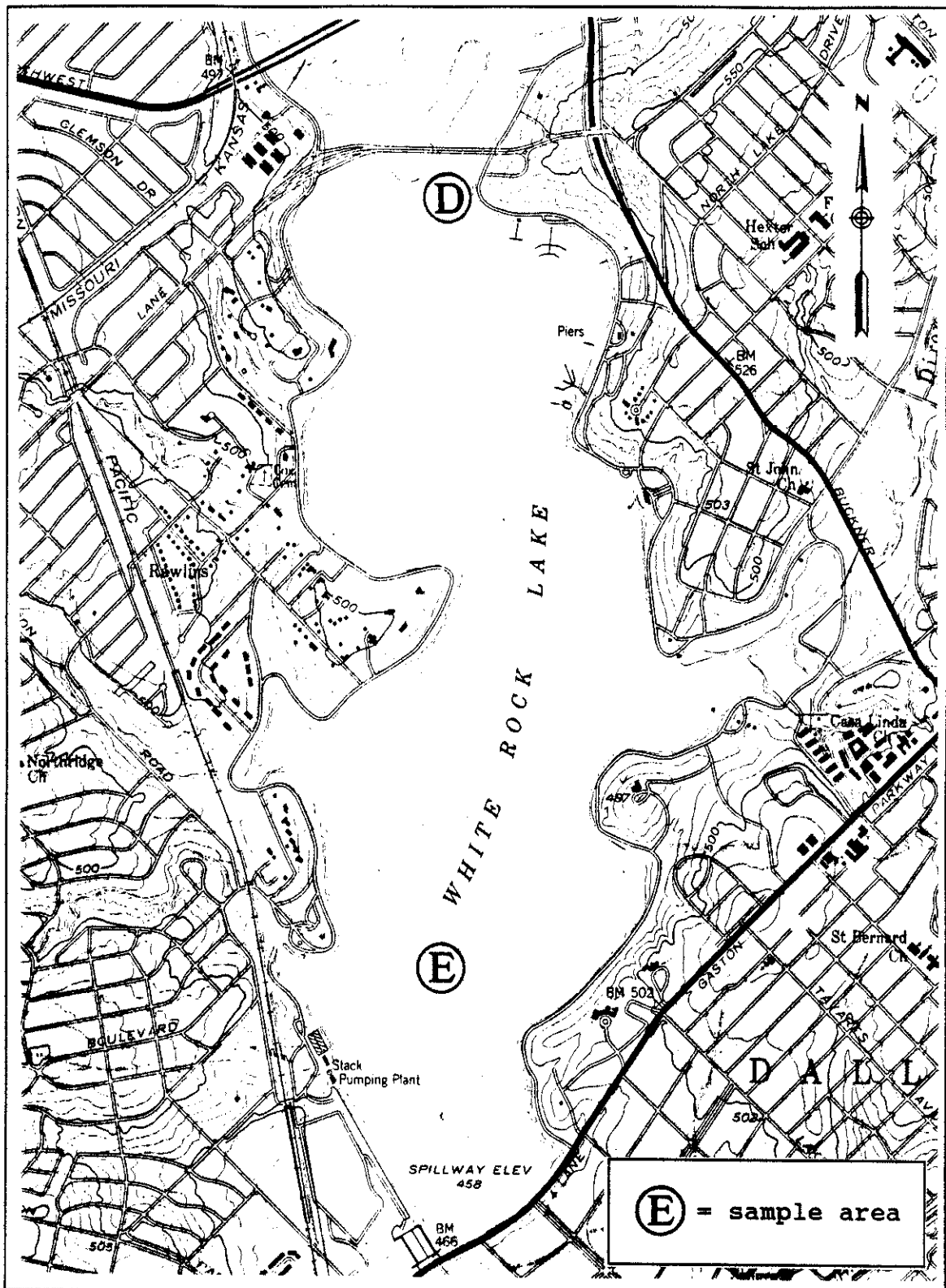


Figure 8. Map of White Rock Lake. (USGS quadrangle map: White Rock Lake, Texas. scale 1:24000).

Zodin (1970) reports that the lake's original surface area was 507.5 ha (1,254 acres) and the capacity was 22.41 km<sup>3</sup> (18,158 acre-feet). Arnold and others (1987) report that siltation has decreased the surface area by 14.5 percent to a current surface area of 434.2 ha (1072.9 ac) and lake capacity has greatly decreased by 44 percent to 12.60 km<sup>3</sup> (10,211 acre-feet). The deepest water measured during a pilot study was 5.0 m (16.5 ft), and average depths were 1.68 m (5.5 ft). The shallowest sampling depths are at the deltas and are typically less than 0.15 m (0.5 ft).

Because of shallow depths throughout the lake, depositional environments are either deltaic or bottomset beds (after Lane, 1953). The vast majority of sediments are found in bottomset beds which consist of very fine grained organic clays and silts.

Tributaries directly entering the lake (Figure 8) have built small deltas with sediment transported during floods. In comparison to bottomset beds which are mostly organic-rich clay with some fine silt, deltaic sediments typically contain less than 25 percent clay with more than 75 percent silt and fine sand. The deltaic process has formed low lying areas that support wetland grasses and cattails (Appendix A, photograph).

## CHAPTER 4

### METHODS

Methods for field sampling, laboratory analysis, and statistical analyses are described in the following sections.

#### Field Methods

On July 31, 1989 samples were collected at 5 areas located from the watershed's headwaters to the lower section of White Rock Lake. At each of the 5 areas, 5 randomly located bottom sediment samples were collected which produced a total of 25 samples. Figure 9 illustrates the experimental design and sample locations.

The five sample areas (designated Areas A-E) were selected for the following reasons:

- (1) to examine downstream patterns in trace metals and sorption factors,
- (2) to evaluate trace metal concentrations below the confluences of major tributaries,
- (3) and because thin to non-existent sedimentary deposits are found in many reaches of White Rock Creek, it is necessary to locate the three stream areas slightly upstream

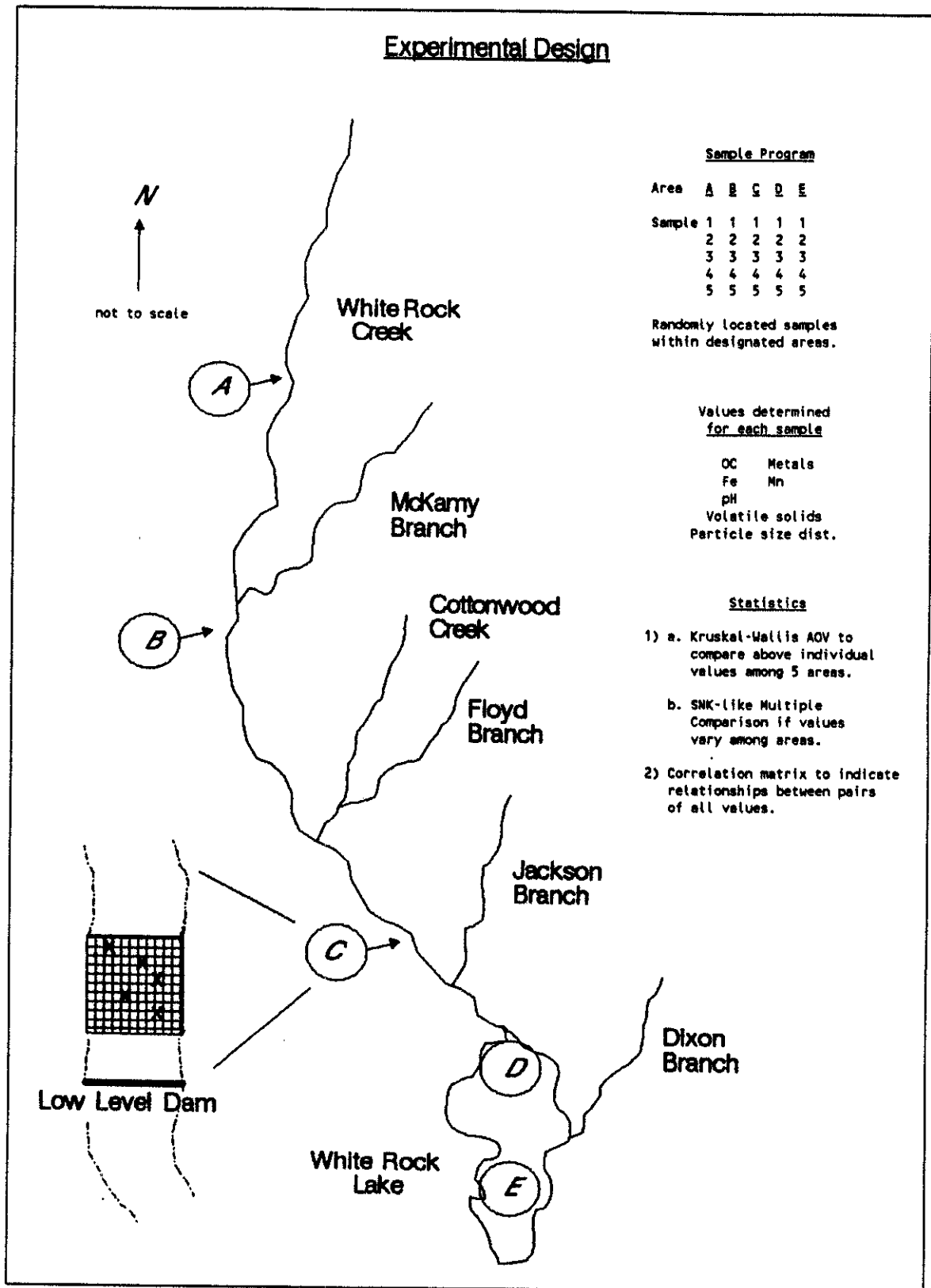


Figure 9. Experimental Design.

from low level dams (Figures 3 and 9, Areas A,B, and C). The greater amounts of sediment and relatively uniform distributions above the dams ensured that samples could be randomly collected.

At areas A,B, and C, stream widths were measured for calculating imaginary 10 X 10 grids with equal area cells (Figure 9). The stream width at each area was 15.2 m (50 ft), therefore each cell was equal to 1.52 m X 1.52 m (5 ft X 5 ft). At each area, five numbers were drawn from a random number table to determine which cells would be sampled. The selected cells were located in the field by measuring from a corner of the imaginary grid.

At the two lake areas (Figure 9, Areas D and E), sample locations were similarly selected by using 10 X 10 imaginary grids with overall grid dimensions of 15.2 m X 15.2 m (50 ft X 50 ft). Sample locations were determined by measuring from a buoy that was anchored at a grid corner.

Sample collection and preservation followed recommendations by Plumb (1981) and the OWDC (1978) for dredged and wet stored samples. In summary, each sample was dredged from the stream or lake bottom with a Petite Ponar dredge and wet sieved to <2.0 mm into a plastic 15.14 l (4 gal) bucket. The sediment was hand stirred to obtain a representative homogenous slurry and poured into a glass jar. After a pH measurement was recorded, the jar was sealed, labelled, and stored in an ice cooler.



Prior to collecting each sample, the sampling equipment was washed with stream water to prevent contamination between samples. The samples were continuously refrigerated at approximately 4°C and only removed briefly during lab analyses.

#### Laboratory and QA-QC Methods

All laboratory analyses were conducted at the University of North Texas during August, 1989 except for pH measurements which were determined during sample collection. Table 4 summarizes the various analytical methods and gives references for detailed procedures. The sections following Table 4 summarize the various test methods.

Laboratory precision for each test was determined by duplicating samples A1, C2, and E5 (designated as A1D, C2D, and E5D). The precision values from these duplicates were averaged to provide a precision value for the specific test. Additional QA-QC procedures included matrix spikes and blanks which are described in the following methods summaries.

#### Percent Volatile Solids

Percent volatile solids was determined by the difference in the weight of a dried sample (105°C) compared to the sample's weight after ignition in a muffle furnace at 400°C for 6h (Jaffe and Walter, 1974; Luoma and Bryan, 1981).

Luoma and Bryan (1981) found that ashing at 400°C for 6h rather than 500°C for 1h provided a very high correlation between volatile solids and organic carbon. Ashing at the higher temperature removed approximately 3 percent of the sediment weight which was not carbon.

Percent volatile solids is calculated with the following formula:

% volatile solids =

$$\frac{105^{\circ}\text{C dried sed. wt} - 400^{\circ}\text{C dried sed. wt.}}{105^{\circ}\text{C dried sed. wt}} * 100$$

Duplicate samples A1D, C2D, and E5D were included to indicate laboratory precision.

Table 4. Summary of analyses and methods.

| <u>ANALYSIS</u>                                     | <u>METHOD</u>  |
|---|--|
| % Volatile Solids<br>(organic matter)               | Weight loss of dried sample after ignition in furnace at 400° C for 6 hrs. (Jaffe and Walters, 1974) |
| Particle Size                                       | Pipet Method and Sieving (OWDC, 1978)  |
| % Organic Carbon                                    | Modified Walkley-Black titration (Black, 1986; Gaudette and others, 1974; Jaffee and Walters, 1977)  |
| Metals (mg/kg)<br>Pb, Cu, Zn, Cd,<br>Cr, Mn, and Fe | Atomic Absorption (AA)<br>(Plumb, 1981)  |
| pH  | Orion pH meter (Orion manual)  |

### Particle Size Distribution

The broad range of particle sizes within individual samples usually requires more than one method to determine size distributions. Following OWDC (1978) procedures, the pipet and sieving methods were used to determine the size classes shown in Table 5.

Table 5. Particle Size Classes (after Lane, 1947).

| <u>Material</u> | <u>Size</u>      |
|-----------------|------------------|
| Sand            | 0.062 - 2.0 mm   |
| Silt            | 0.004 - 0.062 mm |
| Clay            | <0.004 mm        |

Laboratory precision was determined by averaging the precision from duplicate samples A1D, C2D, and E5D.

### Percent Organic Carbon

Organic carbon was determined with a modified Walkley-Black titration method following procedures similar to those of Jaffe and Walter (1977). Gaudette and others (1974) demonstrated that the Walkley-Black method provides organic carbon values comparable to values from a carbon analyzer. In their study, LECO carbon analyzer values were compared with titration values for samples collected from several diverse sedimentary environments. Regression analysis from thirty-three samples revealed a correlation coefficient of 0.989 and an intercept of 0.00.

The Walkley-Black method is also well adapted for carbon analysis when sediments contain high amounts of carbonate (Walkley, 1946). White Rock Creek sediments often have greater than 50 percent detrital limestone grains ( $\text{CaCO}_3$ ) and early attempts at using a carbon analyzer found that all limestone grains were not completely removed prior to combustion. Further comparisons also demonstrated that the Walkley-Black laboratory precision was higher than precision obtained with the carbon analyzer. The titration method was therefore the preferred method for carbon analysis.

The Walkley-Black method is based on a dichromate ( $\text{Cr}_2\text{O}_7^{2-}$ ) oxidation of organic matter to  $\text{CO}_2$  in a sulfuric acid medium. Following the reaction with the acid and dichromate, the amount of remaining dichromate is determined by titrating with ferrous sulfate ( $\text{FeSO}_4$ ). The amount of dichromate used in the reaction is assumed to be proportionate to the sample's organic carbon (Black, 1986).

Jaffe and Walter (1977) modified the Walkley-Black procedure by using a reaction time of 45 minutes and found that organic carbon values compared closely, but slightly lower than dry combustion values. Similarly, a reaction time of 45 minutes was used in this study. Percent carbon was calculated with the following formula:

$$\% \text{ carbon} = \frac{[(N1 * V1) - (N2 * V2)] * 0.364}{\text{net dry wt of sample}}$$

where: N1 = normality of  $K_2Cr_2O_7$   
 N2 = normality of  $FeSO_4$  \* N2 = N1 (V1/V2)  
 V1 = volume of  $K_2Cr_2O_7$  (usually 0.9 to 1.1)  
 V2 = volume of  $FeSO_4$   
 net dry wt. = 2.0 g

As required in the above formula, a blank solution is titrated in order to determine the  $Cr_2O_7^{2-}$  normality. QA-QC procedures included the analysis of three duplicates, A1D, C2D, and E5D.

### Metal Analysis

Sample handling, storage, and digestion procedures for metals follow recommendations by Plumb (1981). The digestion procedure requires a 1:3 v/v  $HNO_3$ -HCl mixture (aqua regia) to extract total metal concentrations. The digestate was analyzed on a Perkin-Elmer Model 2380 atomic absorption spectrophotometer (AA) and total metal concentrations were calculated for cadmium, chromium, copper, lead, zinc, manganese, and iron. Detection limits for each metal are included on individual analysis sheets in Appendix B. Concentrations are reported in mg/kg of sediment and high iron concentrations are occasionally reported in percent of total sediment.

For each sample's digestate, 3 concentrations were read in ppm from the AA and averaged. Samples A1D, C2D, and E5D were duplicate samples for evaluating analytical precision.

The digestion method and percent recoveries were evaluated with three matrix spike samples. Known concentrations of each metal were spiked into samples A1, C2, and E5 and designated A1MS, C2MS, and E5MS. Spiking was conducted prior to digestion and indicated the digestion method's efficiency (percent recovery) and indicated if matrix interference was occurring during AA analysis. The concentration of a spiked sample should equal the sum of the known spike concentration and the concentration read from the AA for the unspiked sample. For example, percent spike recovery is calculated for A1MS as follows:

$$\% \text{ spike recovery} = \frac{\text{A1MS recovery (ppm)}}{(\text{A1 conc.} + \text{A1MS added conc.})} * 100$$

where: A1MS recovery = concentration from 3 AA readings  
 A1 conc. = avg. concentration for sample A1  
 A1MS added conc. = the known concentration added to sample A1MS. In this case, the added concentration for all metals (\*except Fe) is 1.00 ppm.

\* high concentrations of Fe results in high matrix interference on the AA and percent recoveries were not tested.

Finally, in order to provide additional quality control, two blank samples containing only aqua regia were analyzed at the end of each metal analysis. The blanks were handled identically as the actual samples beginning with digestion on the hot plate. Table 6 summarizes all samples analyzed for each metal.

Table 6. Summary of samples used in metal analyses.

| <u>Sample name</u>            | <u>No. of samples</u> | <u>Description</u>        |
|-------------------------------|-----------------------|---------------------------|
| Blanks                        | 2                     | aqua regia only           |
| A1-E5                         | 25                    | field samples             |
| A1D, C2D, E5D                 | 3                     | duplicate lab samples     |
| A1MS, C2MS, E5MS              | <u>3</u>              | A1, C2, E5 spiked samples |
| Total samples<br>(each metal) | 33                    |                           |

### pH Measurements

pH was measured with an Orion pH meter while collecting individual samples in the field.

### Statistical Methods

Data from the five areas were evaluated for homoscedasticity with Bartlett's test (Zar, 1984) and normal distributions with the SAS univariate normal procedure (SAS Institute, 1985). Several of the variances among areas were found to be heteroscedastic (Bartlett's test with Chi square correction, 0.05 LOS), and several values were non-normally distributed within areas (Shiparo-Wilks test, 0.05 LOS). Therefore, in order to keep the statistical analyses consistent, Kruskal-Wallis nonparametric AOVs and SNK-like multiple comparisons were used exclusively to determine among area differences. Zar (1984) reports that the Kruskal-Wallis test has 95 percent the power of a parametric AOV, and when parametric assumptions are deviated from severely, the nonparametric test may in fact be more powerful.

The sampling program was designed with random sampling and equal sample sizes in order to meet requirements for AOV and multiple comparison testing. The sampling design also follows Model I AOV requirements where samples are randomly located at non-randomly selected areas (Zar, 1984). The following summarizes the statistical analyses:

- (1)a. A Kruskal-Wallis nonparametric AOV determines if significant differences exist among areas for metal concentrations and sorption factors listed in Table 7, and
- b. where significant differences are indicated, a SNK-like Multiple Comparison is used to separate areas into distinct groupings.
- (2) Finally, a multiple correlation matrix is calculated to evaluate the relationships between all possible pairs of variables listed in Table 7.

Table 7. Variables analyzed with the AOV and correlation matrix.

|                 |    |        |
|-----------------|----|--------|
| Organic carbon  | Pb | Cr     |
| Volatile solids | Cu | % clay |
| Mn              | Zn | % silt |
| Fe              | Cd | % sand |

The statistical design is summarized in Figure 9 and the statistical analyses are briefly discussed in the following sections.



### Kruskal-Wallis AOV and SNK-like Multiple Comparisons

The following null hypotheses were evaluated with a nonparametric Kruskal-Wallis AOV.

Ho: There is no difference in lead concentrations among the five sampled areas.

Ho: There is no difference in copper concentrations among the five sampled areas.

Ho: There is no difference in chromium concentrations among the five sampled areas.

Ho: There is no difference in zinc concentrations among the five sampled areas.

Ho: There is no difference in cadmium concentrations among the five sampled areas.

Ho: There is no difference in iron concentrations among the five sampled areas.

Ho: There is no difference in manganese concentrations among the five sampled areas.

Ho: There is no difference in organic carbon values among the five sampled areas.

Ho: There is no difference in total volatile solids values among the five sampled areas.

Ho: There is no difference in values of percent clay for the five sampled areas.

Ho: There is no difference in values of percent silt for the five sampled areas.

Ho: There is no difference in values of percent sand for the five sampled areas.

The above null hypotheses which were rejected at the 0.05 level of significance (LOS) were further evaluated with a nonparametric SNK-like multiple comparison test. The multiple comparisons were calculated on the Quattro personal computer spreadsheet program using formulas from Zar (1984).

#### Correlation Matrix

A multiple correlation matrix was produced on SAS (SAS Institute, 1985) for determining significant relationships between all paired combinations of variables (Table 7). The correlation matrix provides a Pearson correlation coefficient and a significance probability for each pair. Correlations were considered significant with probabilities  $\leq 0.05$ .

## CHAPTER 5

### RESULTS AND DISCUSSION

The beginning of this section summarizes the complete data set followed by the Kruskal-Wallis AOV results which indicated differences among the five areas. After these sections, partitioning relationships determined from multiple correlations are discussed. The last section of the results compares White Rock Creek metal concentrations with concentrations from other areas.

#### Summary Of Data Set

Excluding blanks and matrix spike samples, the complete data set has twenty-five observations for each variable (5 samples collected from 5 areas). The data set is statistically summarized in Table 8 and laboratory QA-QC values are included. Coefficients of variation (CV) range from 23.73 to 83.42 and indicate that considerable variation occurs throughout the watershed. Sand percentages display the highest variation (CV=83.42), and clay percentages are also high (CV=64.54).

Table 8. Statistical summary for five areas inclusive.

| Variable   | N  | Minimum | Maximum  | Mean     | Std Dev | CV <sup>a</sup> | Prec. <sup>b</sup> | MSrec(%) <sup>c</sup> |
|------------|----|---------|----------|----------|---------|-----------------|--------------------|-----------------------|
| Zn (mg/kg) | 25 | 76.20   | 157.60   | 115.57   | 27.43   | 23.73           | 99.92              | 112.58                |
| Pb (mg/kg) | 25 | 12.20   | 46.80    | 24.34    | 11.42   | 46.89           | 99.84              | 94.63                 |
| Cr (mg/kg) | 25 | 10.70   | 30.50    | 19.53    | 6.81    | 34.87           | 99.95              | 90.91                 |
| Cu (mg/kg) | 25 | 11.70   | 31.94    | 18.27    | 5.75    | 31.45           | 99.96              | 88.24                 |
| Cd (mg/kg) | 25 | 1.50    | 5.00     | 2.36     | 0.70    | 29.61           | 99.72              | 89.14                 |
| Fe (mg/kg) | 25 | 7187.90 | 18748.20 | 13572.41 | 3828.95 | 28.21           | 99.89              | -                     |
| Mn (mg/kg) | 25 | 318.20  | 1007.10  | 512.68   | 217.53  | 42.43           | 99.96              | 98.45                 |
| VS (%)     | 25 | 1.28    | 6.41     | 4.02     | 1.87    | 46.50           | 99.94              | -                     |
| CLAY (%)   | 25 | 7.97    | 69.13    | 35.15    | 22.69   | 64.54           | 99.95              | -                     |
| SILT (%)   | 25 | 10.18   | 46.95    | 28.68    | 10.45   | 36.44           | 99.94              | -                     |
| SAND (%)   | 25 | 0.10    | 80.54    | 36.16    | 30.17   | 83.42           | 99.74              | -                     |
| OC (%)     | 25 | 0.29    | 1.00     | 0.64     | 0.21    | 32.80           | 99.94              | -                     |

<sup>a</sup>Coefficient of variation<sup>b</sup>Average laboratory precision<sup>c</sup>Metals matrix spike recovery

The relatively high CVs were beneficial for statistically analyzing among area differences. For most factors, significant among area differences were detected with the Kruskal-Wallis AOV which indicates that variation is occurring among sample areas rather than sporadically throughout the sample program.

Laboratory precision ranged from 99.72 to 99.96% and average matrix spike recovery for metals ranged from 88.24 to 112.58% (Table 8).

Attached in Appendix B is a summary of the data set values followed by laboratory data sheets. All measurements, including those for blanks, matrix spikes, and duplicate samples are included with the laboratory data sheets. Similar to Table 8, statistical summaries for each of the five areas are attached in Appendix C.

### Kruskal-Wallis Among Area Comparisons

Statistical results show that the Kruskal-Wallis AOV and the SNK-like multiple comparisons were efficient at determining among area differences and defining distinct groupings. Significant among group differences were detected for all variables except for copper and silt (Table 9).

Table 9. Results from Kruskal-Wallis AOV and SNK-like multiple comparisons among areas.

| <u>Variable</u> | <u>Chi-Square Appx.</u> | <u>DF</u> | <u>p&gt;Chi-Square</u> | <u>SNK<sup>a</sup></u> |
|-----------------|-------------------------|-----------|------------------------|------------------------|
| Zn              | 14.16                   | 4         | 0.007                  | E > AC > BD            |
| Pb              | 17.91                   | 4         | 0.001                  | E > DCBA               |
| Cr              | 17.05                   | 4         | 0.002                  | E > A > CBD            |
| Cu              | 6.151                   | 4         | 0.188 n.s.             |                        |
| Cd              | 14.42                   | 4         | 0.006                  | CEDB > A               |
| Fe              | 21.56                   | 4         | 0.0002                 | E > AC > B > D         |
| Mn              | 15.86                   | 4         | 0.003                  | E > CDAB               |
| VS              | 16.14                   | 4         | 0.003                  | AECB > D               |
| OC              | 11.88                   | 4         | 0.018                  | EABC > D               |
| Clay            | 16.53                   | 4         | 0.002                  | E > A > CBD            |
| Silt            | 7.42                    | 4         | 0.115 n.s.             |                        |
| Sand            | 16.42                   | 4         | 0.002                  | DBCA > E               |

<sup>a</sup>Accepted level of significance  $\leq 0.05$

The ability of the Kruskal-Wallis AOV to detect numerous significant differences reflects nonparametric statistical power when variance heterogeneity is considerable. A cursory comparison between the Kruskal-Wallis AOV and a parametric AOV revealed that the parametric AOV could not determine among area differences for copper ( $p=0.32$ ), cadmium ( $p=0.27$ ), and silt ( $p=0.085$ ). Similarly,

the Kruskal-Wallis AOV did not find copper and silt significantly different, but the Kruskal-Wallis AOV did find cadmium significantly different ( $p=0.006$ ). Variance heterogeneity at Area C in particular, decreased the parametric AOV's efficiency.

Variance heterogeneity as evaluated with Bartlett's test, occasional non-normal distributions, and the comparison with the parametric AOV demonstrated that the Kruskal-Wallis AOV was the appropriate and most efficient statistical test.

#### Metals Compared Among Areas

The Kruskal-Wallis AOV detected significant differences among the five areas for zinc, lead, and chromium, and the SNK-like multiple comparison defined groupings as shown in Table 9. Copper is the only trace metal not significantly different among areas (Kruskal-Wallis AOV,  $p=0.188$ ).

Graphs were constructed to further evaluate metal distributions (Figure 10). The graphs and statistical results together reveal a trend of high metal concentrations at White Rock Lake (Area E). Zinc, chromium, and lead concentrations are statistically higher at the lake than at the other areas (Table 9 and Figure 10). Cadmium is statistically different among areas but the SNK-like comparison shows only two distinct groups with Areas C,E,D,B significantly greater than Area A. The highest grouping

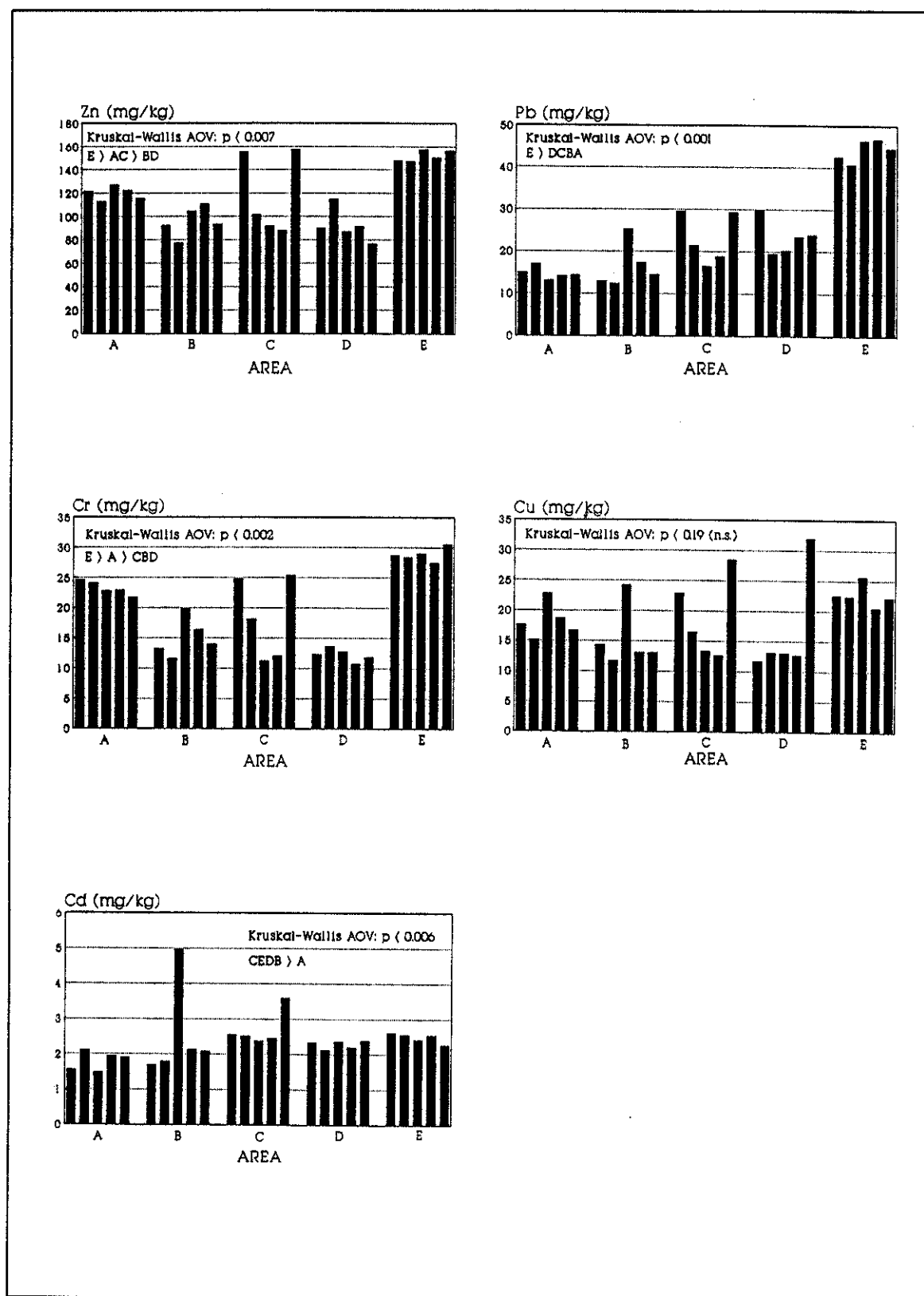


Figure 10. Bar graphs of metal concentrations.

contains Area E and graphs show that cadmium concentrations are moderately high at Area E. Although copper is not significantly different among areas, Figure 10 strongly suggests that copper concentrations follow the trend where elevated concentrations are found at White Rock Lake (Area E).

The SNK-like comparisons and graphs also reveal a trend of elevated metal concentrations at Area A which is located near the creek's headwaters. This spatial trend exhibited where elevated concentrations are found at both Areas E and A demonstrates that metal concentrations do not gradually increase or decrease downstream. The trend also reveals that sediment metal concentrations are not determined by increasing areal drainage. Area A for example, receives the watershed's smallest runoff volume and Area E is influenced by the watershed's greatest runoff volume (Figure 3).

Upon closer inspection of the individual metals, zinc concentrations closely follow the trend where concentrations are elevated at both Areas E and A. Zinc concentrations are highest at Area E, secondly at areas A and C, and lowest at Areas B and D (SNK-like comparison,  $E > AC > BD$ ).

Table 9 and Figure 10 reveal that chromium also follows the trend where highest concentrations are found at Area E and secondly at Area A (SNK-like comparison,  $E > A > CBD$ ). Copper appears to follow the same trend (Figure 10), even



though the Kruskal-Wallis did not detect significant among area differences for copper (Kruskal-Wallis,  $p < 0.19$ ).

Figure 10 shows that lead and cadmium concentrations do not follow the trend of high concentrations at both Areas E and A. The graph for lead concentrations suggests a subtle downstream increase, but the SNK-like comparison could not detect differences between Areas A,B,C, and D. Area E was statistically higher than these four areas (Table 9).

Cadmium was significantly different among areas (Kruskal-Wallis AOV,  $p=0.006$ ), and the SNK-like comparison determined Areas C,E,D, and B were in a single group which was higher than Area A. With the exclusion of sample B3 (Figure 10), cadmium concentrations are less variable than the other metals. Although not statistically tested, sample B3 appears to be an outlier, however its value did not restrict the AOV and multiple comparison tests.

#### Sorption Factors Compared Among Areas

Sorption factors were significantly different among areas for all factors except for silt (Table 9). Silt distributions were extremely variable (Figure 7) and indicates why the Kruskal-Wallis AOV did not detect significant differences among areas (Kruskal-Wallis AOV,  $p=0.115$ ).

The watershed's sediment pH values vary only within a narrow range of 6.9 to 7.1, and this slight variation was

observed both within and among all areas. pH values were therefore excluded from further statistical analyses.

In addition to statistical analyses, graphs were constructed to evaluate sorption factors (Figure 11). A review of the graphs and the statistical analyses (Table 9) again reveal a general trend of high values at Area E and secondly at Area A. In the case of sand, its pattern is reversed because of sand's frequently observed negative correlations with other sorption factors and metal concentrations.

The comparable spatial trends exhibited by both sorption factors and metal concentrations (Figures 10 and 11) indicates that metal concentrations are principally determined by substrate availability for metal sorption. For example, zinc, lead, and chromium were significantly highest at Area E which is also the area where the highest amounts of iron, manganese, and clay were available for sorption. Accordingly, percent sand was lowest at Area E.

The Kruskal-Wallis AOV determined significance for both organic carbon and volatile solids, but considerable within area variance restricted the SNK-like comparison to defining only two distinct groups for each variable (Figure 11 and Table 9). Again, Areas E and A were in the highest group for both organic carbon and volatile solids which indicates that organic carbon is available for metal sorption. A review of

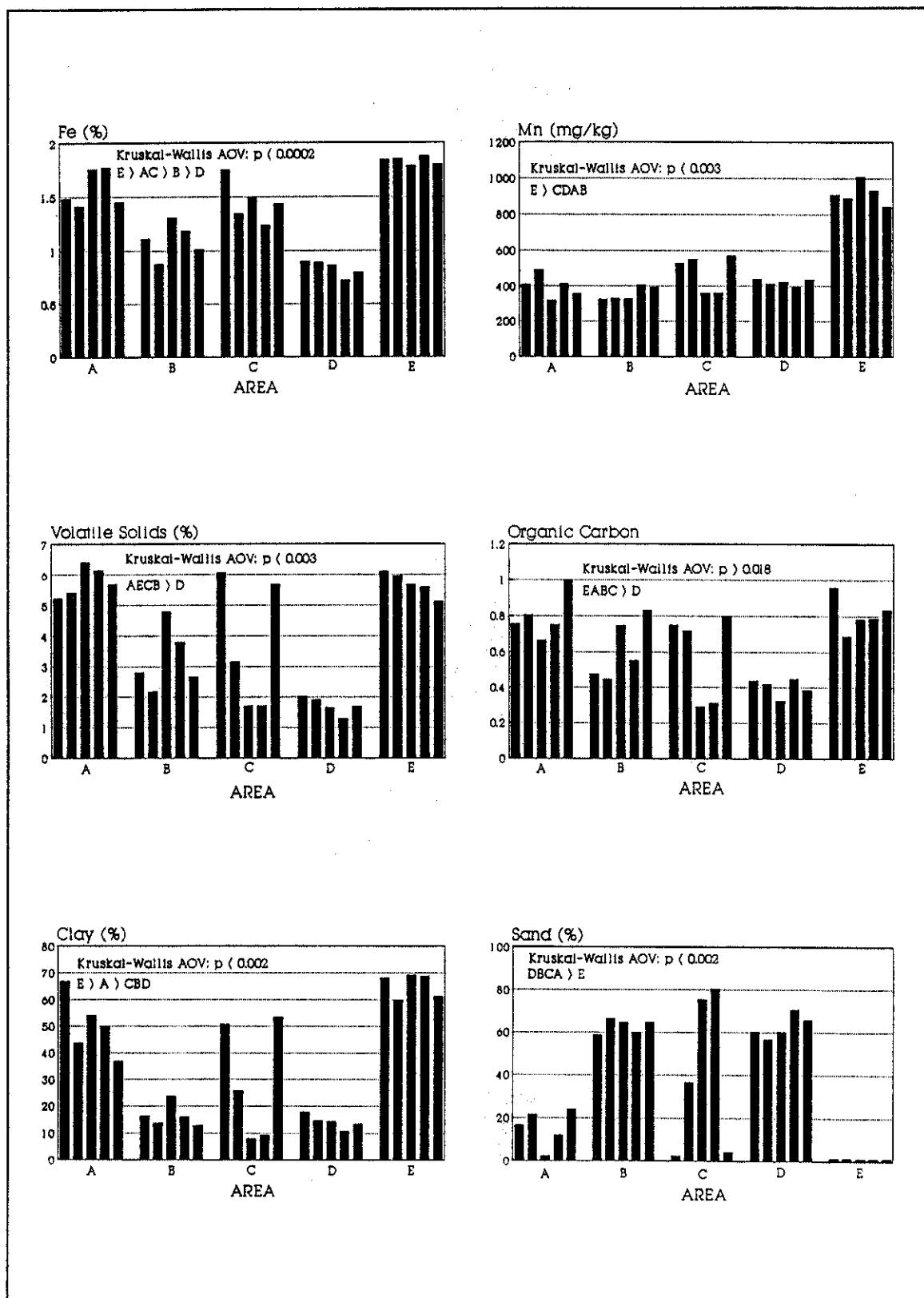


Figure 11. Bar graphs of sorption factor values.

the SNK-like comparisons in Table 9 shows that the distributions of organic carbon and volatile solids were somewhat similar and suggests a correlation between the two variables.

Upon closer inspection of iron, its concentrations closely followed the general trend where the highest concentrations are observed at Area E and secondly at Area A. The SNK-like comparison again found that the delta area (Area D) is low in iron as it generally is for trace metal concentrations.

Manganese concentrations are highest at Area E, however Area A was not statistically different from Areas C,D, and B (Table 9). As shown on Figure 11, manganese concentrations are fairly constant in the stream, but considerably elevated at Area E.

#### Relationships Among Variables

In order to determine significant relationships among variables, all data (n=25) were entered into the SAS multiple correlation procedure (SAS Institute, 1985). This first section describes the relationships between individual metals and sorption factors. The following section describes relationships among sorption factors.

### Correlations Between Metals and Sorption Factors

The correlation matrix (Table 10) indicates numerous significant relationships between metals and sorption factors. The strongest correlations are between percent clay and chromium ( $r=0.96$ ), and percent sand and zinc ( $r= -0.91$ ). These relationships are also inversely similar with strong correlations between sand and chromium ( $r= -0.95$ ) and clay and zinc ( $r=0.89$ ).

An evaluation of individual metals shows that zinc is significantly correlated with all sorption factors (Table 10). The strongest relationship with zinc is a negative correlation with percent sand, and this relationship is illustrated in Figure 12. Zinc and clay ( $r=0.89$ ) and zinc and iron ( $r=0.83$ ) are also highly correlated (Table 10 and Figure 12). Significant correlations are listed according to decreasing correlation coefficients in Table 11. Zinc was significantly correlated with organic carbon ( $p=0.0001$ ,  $r=0.68$ ), but the relationship is not as strong as with the other sorption factors.

Lead is significantly correlated with manganese, clay, sand, and iron (Table 10). The strongest relationship for lead is with manganese ( $r=0.92$ ), and this relationship is illustrated in Figure 12. The lead:manganese relationship is also reflected when comparing Figures 10 and 11 (bar charts) which show lead and manganese are particularly high at Area E. Table 11 shows all significant correlations with

Table 10. Correlation matrix of relationships between individual metals and sorption factors. The upper value is the correlation coefficient and the lower value is the probability that the correlation coefficient equals zero. Statistical significance at  $p \leq 0.05$  denoted with (\*).

|      | Zn                  | Pb                  | Cr                  | Cu                  | Cd                 |
|------|---------------------|---------------------|---------------------|---------------------|--------------------|
| Fe   | 0.83075<br>0.0001*  | 0.51211<br>0.0089*  | 0.88212<br>0.0001*  | 0.47064<br>0.0176*  | 0.07477<br>0.7224  |
| Mn   | 0.75172<br>0.0001*  | 0.92930<br>0.0001*  | 0.74315<br>0.0001*  | 0.46337<br>0.0197*  | 0.15754<br>0.4520  |
| VS   | 0.83453<br>0.0001*  | 0.38049<br>0.0606   | 0.92365<br>0.0001*  | 0.55507<br>0.0040*  | 0.11631<br>0.5798  |
| OC   | 0.68602<br>0.0001*  | 0.35085<br>0.0855   | 0.80900<br>0.0001*  | 0.41806<br>0.0376*  | 0.14676<br>0.4839  |
| Clay | 0.89067<br>0.0001*  | 0.61125<br>0.0012*  | 0.96133<br>0.0001*  | 0.57364<br>0.0027*  | 0.01349<br>0.9490  |
| Silt | 0.69360<br>0.0001*  | 0.27642<br>0.1810   | 0.65146<br>0.0004*  | 0.40171<br>0.0465*  | -0.11917<br>0.5705 |
| Sand | -0.91012<br>0.0001* | -0.55546<br>0.0040* | -0.94864<br>0.0001* | -0.57058<br>0.0029* | 0.03113<br>0.8826  |

Table 11. Significant correlations between individual metals and sorption factors in order of decreasing correlation coefficients. All are significant at LOS  $\leq 0.05$ .

Zn: Sand > Clay > Volatile Solids > Fe > Mn > Silt > OC  
Pb: Mn > Clay > Sand > Fe  
Cr: Clay > Sand > Volatile Solids > Fe > OC > Mn > Silt  
Cu: Clay > Sand > Volatile Solids > Fe > Mn > OC > Silt  
Cd: no significant correlations

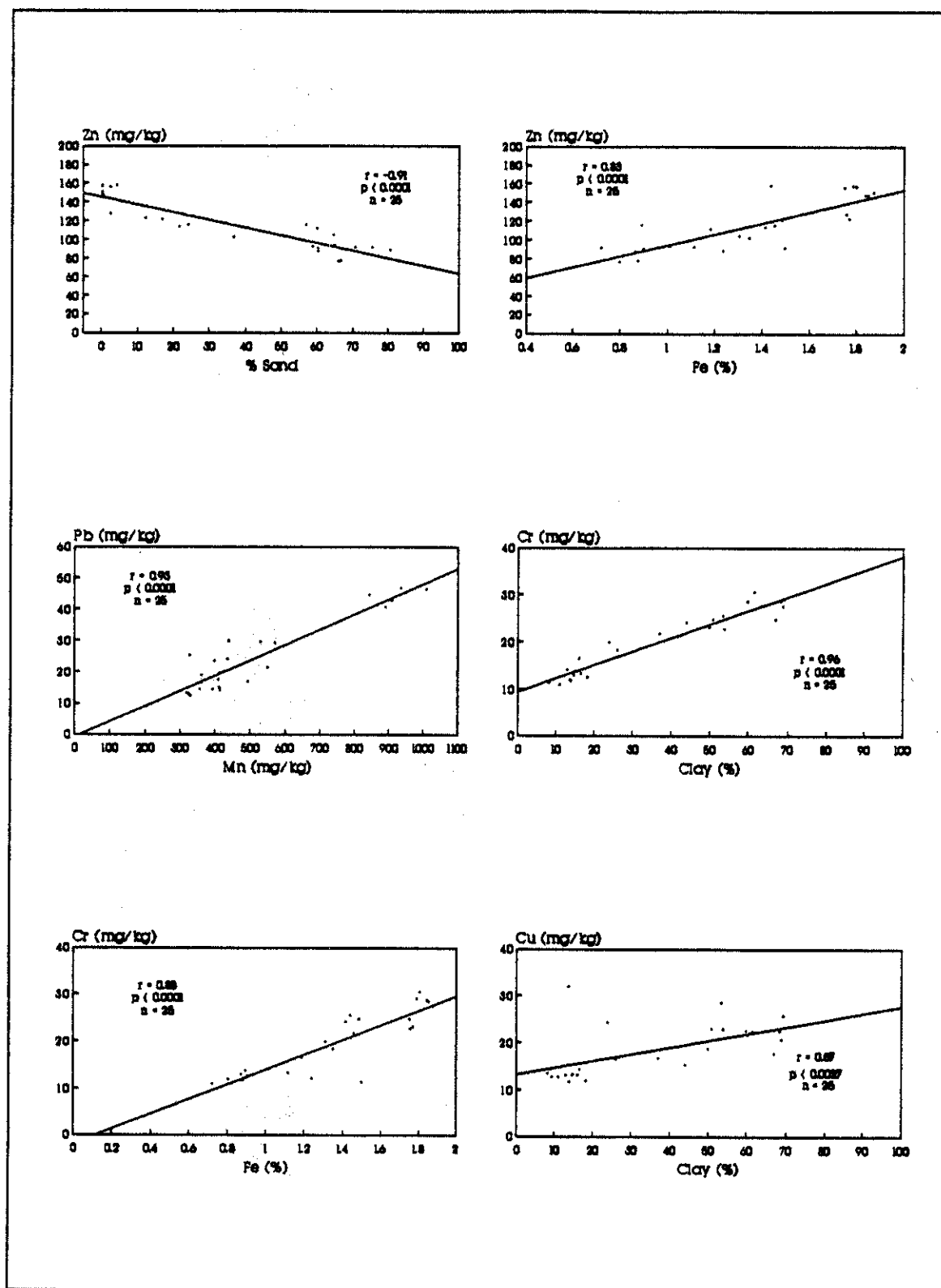


Figure 12. Various correlations between metals and sorption factors.

lead in order of decreasing correlation coefficients. Lead was not correlated with organic carbon, volatile solids, or silt.

Similar to zinc, chromium is significantly correlated with all sorption factors (Table 10). The correlations with clay ( $r=0.96$ ) and sand ( $r=-0.95$ ) are the strongest correlations observed between metals and sorption factors. Correlation coefficients also indicate that overall, chromium is more closely related to the sorption factors than any of the other four metals (Table 10). Table 11 shows the significant correlations with chromium in order of decreasing correlation coefficients, and Figure 12 illustrates the significant relationships between chromium and clay, and chromium and iron.

Copper is also significantly correlated with all of the sorption factors but the strength of these relationships are comparatively weak (Table 10). The highest correlation coefficient is between copper and clay ( $r=0.57$ ). However, as shown in Figure 12 if three outlying points were excluded, the relationship between copper and clay would be relatively strong. Table 11 shows the significant correlations for copper in order of decreasing correlation coefficients.

Cadmium was not significantly correlated with any sorption factor even though the Kruskal-Wallis AOV indicated significant differences among areas. Cadmium concentrations



were near AA detection limits which decreased the resolution of these data.

Table 11 reveals that metal concentrations are more closely correlated with percent clay and percent sand than any other variables. Zinc and chromium concentrations especially appear predictable with linear regression when particle size distribution is known. Lead concentration is also correlated with percent clay, but a regression would be somewhat indefinite as suggested by the relatively low correlation coefficient ( $r=0.61$ ). However, if manganese concentrations were known, lead could be predicted confidently ( $r=0.93$ ). Copper is most highly correlated with clay ( $r=0.57$ ), but the relatively low correlation coefficient suggests that a regression would also be indefinite. Additional data for copper and clay might reveal that the three outlying points in Figure 12 are not strongly representative of the copper:clay relationship and would improve the correlation.

A multiple regression procedure was used to further examine the predictability of metal concentrations with sorption factors (SAS Institute, 1985). Regression coefficients indicated that various combinations of sorption factors were not appreciably more capable than highly correlated single factors for predicting metal concentrations.

### Correlations Among Sorption Factors

Correlation is exceptionally high among sorption factors as demonstrated by significant correlations among all possible pairs (Table 12).

Table 12 shows that iron is most strongly correlated with volatile solids ( $r=0.87$ ) and closely followed by a correlation with percent clay ( $r=0.86$ ). The relationship between iron and clay is illustrated in Figure 13 and the relationship's strength suggests that sediment iron concentrations are predictable by knowing clay percentages. Iron also has a fairly strong negative correlation with percent sand ( $r=-0.85$ ). Correlations between iron and all sorption factors are shown in Table 13 in order of decreasing correlation coefficients.

Manganese is most highly correlated with clay ( $r=0.72$ ), but this correlation is not as strong as the relationship observed between iron and clay. The relationship between manganese and clay would allow only tentative predictions of manganese concentrations if clay percentages were known. The clay:manganese correlation is shown in Figure 13, and Table 12 shows the correlations between manganese and all sorption factors. Significant correlations with manganese in order of decreasing correlation coefficients are shown in Table 13.

Table 12. Correlation matrix of relationships between all paired combinations of sorption factors. The upper value is the correlation coefficient and the lower value is the probability that the correlation coefficient equals zero. Statistical significance at  $p \leq 0.05$  denoted with (\*).

|      | Mn                 | VS                 | Clay               | Silt               | Sand                | OC                  |
|------|--------------------|--------------------|--------------------|--------------------|---------------------|---------------------|
| Fe   | 0.63123<br>0.0007* | 0.86736<br>0.0001* | 0.85627<br>0.0001* | 0.59271<br>0.0018* | -0.84929<br>0.0001* | 0.65017<br>0.0004*  |
| Mn   | _____              | 0.49172<br>0.0125* | 0.72633<br>0.0001* | 0.39849<br>0.0485* | -0.68429<br>0.0002* | 0.46846<br>0.0182*  |
| VS   | _____              | _____              | 0.89088<br>0.0001* | 0.68797<br>0.0001* | -0.90832<br>0.0001* | 0.83320<br>0.0001*  |
| Clay | _____              | _____              | _____              | 0.60340<br>0.0014* | -0.96107<br>0.0001* | 0.73693<br>0.0001*  |
| Silt | _____              | _____              | _____              | _____              | -0.80024<br>0.0001* | 0.57470<br>0.0027*  |
| Sand | _____              | _____              | _____              | _____              | _____               | -0.75331<br>0.0001* |

Table 13. Significant correlations among sorption factors in order of decreasing correlation coefficients. All are significant at  $LOS \leq 0.05$ .

|                   |  |
|-------------------|--|
| Fe:               | Volatile Solids > Clay > Sand > OC > Mn > Silt |
| Mn:               | Clay > Sand > Fe > Volatile Solids > OC > Silt |
| VS <sup>a</sup> : | Sand > Clay > Fe > OC > Silt > Mn              |
| OC <sup>b</sup> : | Volatile Solids > Sand > Clay > Fe > Silt > Mn |
| Clay:             | Sand > Volatile Solids > Fe > OC > Mn > Silt   |
| Silt:             | Sand > Volatile Solids > Clay > Fe > OC > Mn   |
| Sand:             | Clay > Volatile Solids > Fe > Silt > OC > MN   |

<sup>a</sup>VS = Volatile Solids

<sup>b</sup>OC = Organic Carbon

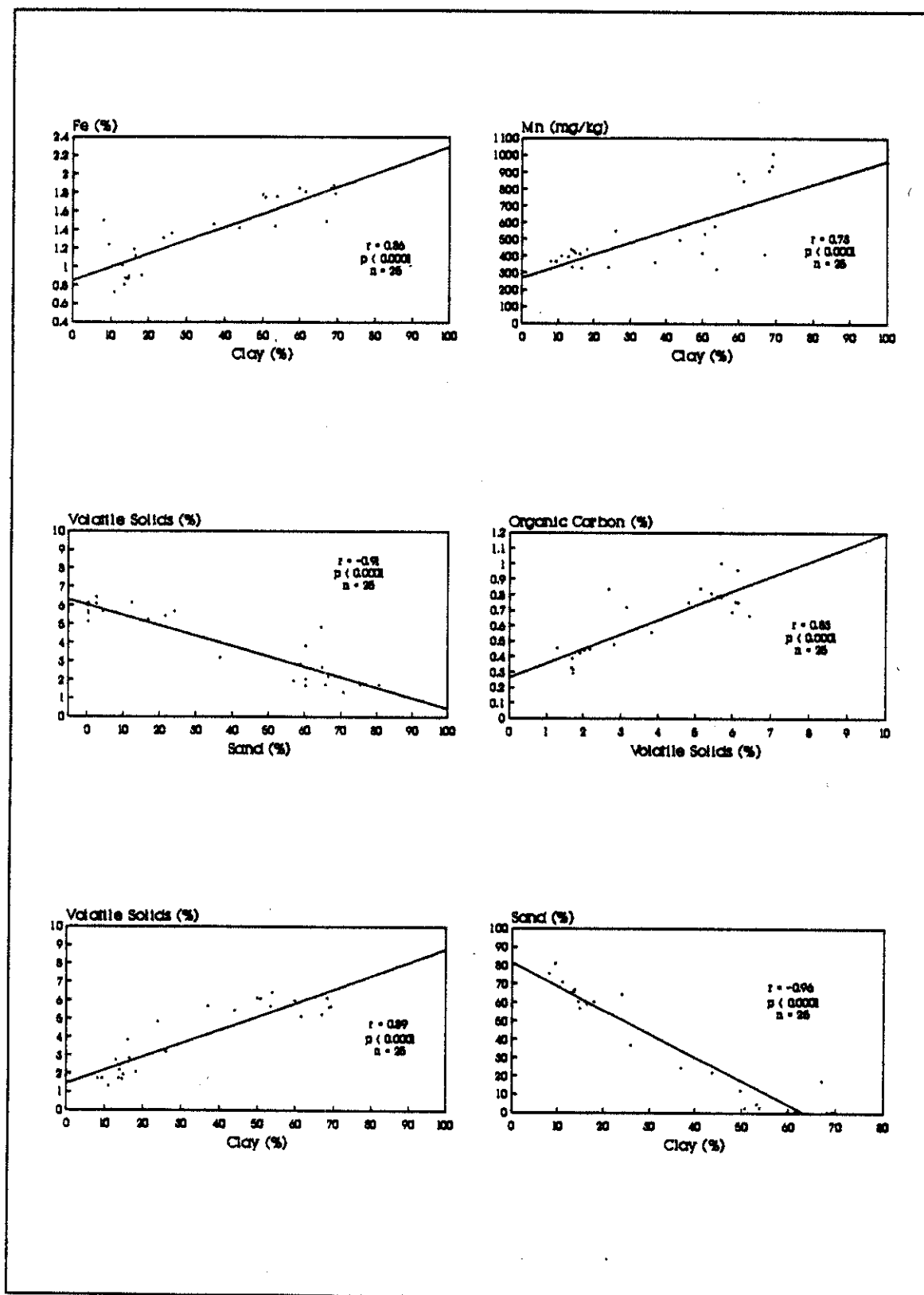


Figure 13. Various correlations among sorption factors.

Volatile solids has a strong negative correlation with percent sand ( $r=-0.91$ ) and conversely, a strong positive correlation with percent clay ( $r=0.89$ ). The correlation between volatile solids and sand is shown in Figure 13 which suggests that either factor could be confidently predicted by knowing the value of the other. Table 13 shows correlations between volatile solids and all sorption factors in order of decreasing correlation coefficients. Of particular interest is the relationship between volatile solids and organic carbon. Previous studies have frequently found strong correlations between volatile solids and organic carbon (Jaffe and Walter, 1977; Luoma and Bryan, 1983; and Gaudette and others 1974). In this study volatile solids and organic carbon are also closely correlated ( $r=0.83$ ) and the relationship is shown in Figure 13.

Percent clay was most highly correlated with percent sand ( $r=-0.96$ ). Other variables strongly correlated with clay were volatile solids ( $r=0.89$ ) and iron ( $r=0.86$ ). Figure 13 shows the close relationship between percent clay and volatile solids. Table 13 shows correlations between clay and the other sorption factors according to decreasing correlation coefficients. As previously described, strong correlations with percent clay suggest that values for sand, volatile solids, and iron are predictable by knowing the sediment's percent clay.

Because of the strong relationship between clay and sand (Figure 13), most variables that are positively correlated with clay are negatively correlated with sand. Volatile solids and iron, for example have strong negative correlations as described above. Correlations between sand and the other sorption factors in order of decreasing correlation coefficients are shown in Table 13.

Correlation coefficients between percent silt and the other sorption factors are comparatively low (Table 12). The highest correlation for silt is with percent sand ( $r=-0.80$ ), and secondly with volatile solids ( $r=0.69$ ). Because of relatively low correlation coefficients and because the Kruskal-Wallis AOV could not detect among area differences for silt, a prediction of other variables with percent silt would be indefinite.

In summary, high correlation coefficients found between many sorption factors suggests that several factors can be confidently predicted with simple linear regression. Volatile solids, percent clay, and percent sand in particular are strongly correlated with many sorption factors (Tables 12 and 13). Volatile solids correlations between iron ( $r=0.87$ ) and organic carbon ( $r=0.83$ ) suggests these factors can be closely estimated simply by measuring volatile solids. Percent clay correlations with volatile solids ( $r=0.89$ ), iron ( $r=0.86$ ), organic carbon ( $r=0.74$ ), and

manganese ( $r=0.73$ ) suggests that these values can be estimated after conducting particle size analysis. Sand percentages determined also from particle size analysis could be used for predicting volatile solids ( $r= -0.91$ ) and iron ( $r= -0.85$ ).

#### Comparative Metal Concentrations

Although federal or state agencies have not developed sediment quality criteria, comparisons can be made among the watershed's metal concentrations and concentrations from local and nationwide studies (Table 14). Sediment size fractions used in measuring respective metal concentrations are listed in Table 14 because of the particle size to metal concentration relationship. In most cases where bulk sediment analysis is reported, the sediment was either ground to fine particles before analysis, or the excessively coarse particles were selectively removed in the field. In this manner, the term "bulk sediment" generally indicates sand to clay particles and excludes large gravel size particles.

A general review of Table 14 reveals two areas which have anomalously high metal concentrations. In particular, the study of the contaminated Montana stream (Moore and others, 1989) found concentrations that are well above the other areas. Secondly, the Illinois study (Striegl, 1988)

also reported noticeably high lead and copper concentrations. Remaining areas have concentrations which appear to fall within a discernable range, regardless of the particle size analyzed.

Table 14. Comparative metal concentrations reported in either ranges or mean mg/kg concentrations.

| <u>Area</u>                | <u>Sediment Fraction</u> | <u>Zinc</u>           | <u>Lead</u>          | <u>Chromium</u>     | <u>Copper</u>         | <u>Cadmium</u>     |
|----------------------------|--------------------------|-----------------------|----------------------|---------------------|-----------------------|--------------------|
| This study                 | bulk(<2mm)               | 115.6 ( $\pm 27.43$ ) | 24.3 ( $\pm 11.42$ ) | 19.5 ( $\pm 6.81$ ) | 18.3 ( $\pm 5.75$ )   | 2.4 ( $\pm 0.70$ ) |
| Trinity River <sup>a</sup> | bulk                     | 17-503                | 7-57                 | 6-150               | 3-186                 | 1-5                |
| TWC 90th % <sup>b</sup>    | bulk                     | 120                   | 31.8                 | 72.1                | 40.0                  | 3.0                |
| EPA region VI <sup>c</sup> | bulk                     | 75                    | 50                   | 100                 | 50                    | 2                  |
| Western U.S. <sup>d</sup>  | <0.063mm                 | 49-510                | 9-52                 | 20-210              | 0-110                 | -                  |
| Illinois <sup>e</sup>      | bulk                     | 112.7 ( $\pm 65.6$ )  | <57                  | <21.6               | 42.0 ( $\pm 56.0$ )   | <1.04              |
| Illinois <sup>f</sup>      | <0.762mm                 | 210                   | 1,590                | -                   | 250                   | -                  |
| Pennsylvania <sup>g</sup>  | 2-.25mm                  | 240                   | 120                  | 10                  | 40                    | -                  |
| Ohio <sup>h</sup>          | 2-.063mm                 | 200                   | 41                   | 12                  | 64                    | -                  |
| Missouri <sup>i</sup>      | 0.25-0.15mm              | 31.1 ( $\pm 2.02$ )   | 32.9 ( $\pm 1.91$ )  | -                   | 10.6 ( $\pm 0.23$ )   | 0.4 ( $\pm 0.03$ ) |
| Montana <sup>j</sup>       | bulk                     | 1,200 ( $\pm 990$ )   | 318 ( $\pm 344$ )    | -                   | 1,160 ( $\pm 1,180$ ) | 7.8 ( $\pm 4.5$ )  |
| U.S. soils <sup>k</sup>    | bulk                     | 48 ( $\pm 1.95$ )     | 16 ( $\pm 1.86$ )    | 37 ( $\pm 2.37$ )   | 17 ( $\pm 2.44$ )     | -                  |

<sup>a</sup>IAS and others (1989). Study of the Trinity River, Texas.

<sup>b</sup>Davis (1987). TWC 90th percentile values. Concentrations considered elevated above these values.

<sup>c</sup>EPA (1974). Proposed guidelines for dredged sediments.

<sup>d</sup>Severson and others (1987). Sediment from nine western U.S. areas (n=108).

<sup>e</sup>Kelly and Hite (1981). Study of 63 Illinois Lakes. Bulk sediment ground to <0.1 mm.

<sup>f</sup>Striegl (1988). Sediment from a Chicago, Illinois small urban lake.

<sup>g</sup>Yorke and others (1985). Sediment from Fairmount Pool, Lower Schuylkill River, Pennsylvania.

<sup>h</sup>Helsel and Koltun (1986). Sediment from Cuyahoga River in Cleveland, Ohio.

<sup>i</sup>Mantei and Coonrod (1989). Missouri stream adjacent to a sanitary landfill.

<sup>j</sup>Moore and others (1989). Contaminated Clark Fork River.

<sup>k</sup>Shacklette and Boerngen (1984). Concentrations in uncontaminated U.S. soils (n=1,318 sites).



When comparing areas with the White Rock Creek Watershed, the nearest area is the Trinity River which was recently studied by the University of North Texas and the University of Texas at Dallas (IAS and others, 1989). White Rock Creek is a major tributary to the Trinity River, and the confluence is 16.5 km (10.3 mi) below White Rock Lake dam (section 3.1.3). Several sampling stations on the Trinity River were below this confluence and therefore are influenced by metal transport through the White Rock Creek Watershed. A comparison between the two areas reveals that the watershed's mean metal concentrations are centered within the Trinity River's range of concentrations.

The Texas Water Commission (TWC) in Austin maintains a historical data base which shows statewide 90th percentile concentrations (Davis, 1977). If a sediment's metal concentration exceeds the 90th percentile level, the TWC considers it an elevated concentration. Upon inspection of White Rock Creek metals, mean zinc, lead, and cadmium concentrations are slightly below TWC levels and chromium and copper concentrations are well below TWC levels (Table 14).

The U.S. Environmental Protection Agency (U.S.EPA, 1974) published acceptability guidelines for dredged sediment in EPA Region VI. An interagency comparison between EPA guidelines and TWC 90th percentile levels shows slight

differences with neither agency consistently reporting higher or lower concentrations. A comparison between the watershed's metals with the EPA guidelines shows that mean zinc and cadmium concentrations are slightly elevated, whereas, lead, chromium, and copper are below EPA guidelines.

The U.S. Department of the Interior Geological Survey analyzed sediment from sites in California, Nevada, Montana, Utah, Wyoming, Arizona, and Texas (Severson and others, 1987). Sediment samples were laboratory sieved to  $<0.063\text{mm}$  (silt and finer) and analyzed for metals. A comparison with these concentrations shows that White Rock Creek metals are generally lower than the range reported in the western U.S. study. However, when comparing concentrations between the two studies, the different particle sizes analyzed must be recognized. In the western U.S. study, only the  $<0.063\text{mm}$  fraction was analyzed and consequently, their results may appear slightly elevated.

In a comprehensive study by the Illinois EPA, 273 sediment samples from 63 Illinois lakes were analyzed (Kelly and Hite, 1981). Bulk sediment was ground to fine grain size prior to metal analysis. A comparison with the White Rock Creek Watershed shows very similar mean metal concentrations, although where reported, the Illinois lake mean concentrations have slightly higher standard deviations.

A study of a small urban lake in Chicago, Illinois (Striegl, 1988) found moderately high zinc concentrations and particularly high lead concentrations. The Illinois data were derived by analyzing the <0.762 mm sediment size fraction as opposed to the <2.0 mm analyzed in this study. The Illinois data may be elevated slightly because of modest differences in particle sizes analyzed.

Several streams studies have been recently conducted in Pennsylvania (Yorke and others, 1985), Ohio (Helsel and Koltun, 1986), Missouri (Mantei and Coonrod, 1989), and Montana (Moore and others, 1989). The studys' objectives are variable and respectively include an evaluation of regional metal distributions, urban landuse and metals, an evaluation of sanitary landfill leachate and metals, and an evaluation of metals in a contaminated river near a mining district (Table 14). When considered altogether, these metal concentrations are variable, but show that White Rock Creek Watershed concentrations are either typical or below most of the areas.

A final comparison can be made with an extensive U.S. Geological Survey database which contains values for native soils throughout the United States (Shacklette and Boerngen, 1984). Bulk soil samples were collected from 1,318 nationwide sites which were selected only if they appeared in an undisturbed natural condition. Soil samples were pulverized to  $\leq 2\text{mm}$  before analysis. Even though the data are

for metals in soils, they represent metal concentrations that are naturally available to watersheds. A comparison between the White Rock Creek Watershed and the database shows that the watershed's mean zinc concentration is elevated, lead and copper are slightly elevated, and chromium is actually lower than the database's mean chromium concentration.

In summary, White Rock Creek Watershed metal concentrations are not elevated according to TWC guidelines and similar to concentrations found locally. In comparison to EPA guidelines, zinc and cadmium are slightly elevated, and lead, copper, and chromium are below EPA values. Further comparisons with nationwide studies show that the watershed's concentrations are typical of those found in most urban watersheds.

## CHAPTER 6

### SUMMARY

The data set demonstrates that the watershed's metal concentrations and sorption factors are highly variable (Table 8). As determined by Kruskal-Wallis AOV results, the variation is not sporadically distributed, but occurs among watershed areas. These results determined that, except for copper and silt, significant among area differences exist for all metals and sorption factors. Although copper is not significantly different, copper concentrations shown graphically indicate a spatial distribution similar to the other metals (Figure 10).

Spatial distributions of metal concentrations reveal a trend of elevated concentrations at Area E and secondly at Area A. This pattern demonstrates that metal concentrations:

- (1) do not gradually change in a downstream direction,
- (2) do not increase below the confluences of major tributaries, and
- (3) are not determined by increasing areal drainage.

Spatial distributions of sorption factors reveal a similar trend of high values at Area E and secondly at

Area A (Figure 11). The striking similarity in spatial patterns exhibited by sorption factors and metals indicates that sediment metal concentrations are primarily dependent upon the availability of various sorption factors to attract metals.

Multiple correlations indicate that most metal concentrations are more closely correlated with clay (and inversely, sand) than any other variables (Table 11). Clay and sand are also more closely correlated with the other sorption factors (Table 13). When considering that clay and sand have the highest coefficients of variation (Table 8), the importance of particle size is further substantiated. As a result, clay and sand are the most efficient linear regression variables.

In addition to the relationships seen between metals and particle size, numerous and strong correlations occur discriminately between individual metals and sorption components (organic carbon, iron, and manganese). The correlation coefficients from these relationships indicate that the affinities of individual metals to the various sorption components are metal specific. And finally, when considering the numerous intercorrelations between all variables and clay, the results indicate that clay particles indirectly control metal concentrations by attracting sediment components which are in turn attracting trace metals (Jenne, 1977).

When comparing metal concentrations among studies, particle size effects need to be considered. However, even when the analyzed size is different among studies, general comparisons appear viable. This is first suggested when comparing guidelines by the Texas Water Commission and the U.S. EPA. These guidelines were independently derived but values for bulk sediment metals are quite similar (Table 14).

Additional evidence is shown when comparing the contaminated Clark Fork River, Montana with the other areas (Table 14). The Clark Fork River is highly contaminated from mining activities, whereas other areas in Table 14 are not necessarily expected to contain elevated metal concentrations. As shown in Table 14, contrastingly higher values are found at the Montana area in comparison to the other studies. The Clark Fork River results are further supportive of bulk sediment comparisons because the Clark Fork River is a high gradient stream and the sediments analyzed were coarse grained.

With increasing concern over contaminated sediment toxicity, regulators are currently examining methods to develop sediment quality criteria. Important considerations when deciding which method prevails are that laboratory analyses must be able to routinely evaluate sediment on a nationwide basis, and the analyses should reasonably

indicate toxicity. Currently, the methods being evaluated are: 1) a numerical sediment quality criteria similar to the water quality criteria approach, 2) an equilibrium partitioning (EP) approach, and 3) bioassays (Shea, 1988).

The EP approach is receiving favorable attention, but several major assumptions are required which are not completely accepted. The EP's principal assumptions are that sediment toxicity is dependent primarily upon the dissolved concentration in pore water, and that adsorption coefficients for individual sorption components can be derived which can collectively predict active pore water concentrations.

The first problem arises when considering that solution uptake is not the only bioaccumulation pathway, but sediment ingestion by particulate detritus feeders can be equally as important (Luoma, 1989). Even if particulate feeders are disregarded, sediment component absorption coefficients must be rigorous because of the enormous differences in metal concentrations in sediment and water. A slight change in water chemistry may consequently result in a profound change in pore water equilibrium. Finally, even if it is accepted that sorption coefficients can reasonably predict pore water concentrations, bioavailability often varies among areas irrespective of metal concentrations in sediment or water (Luoma, 1989). This variation can result because of innumerable among area physical differences.

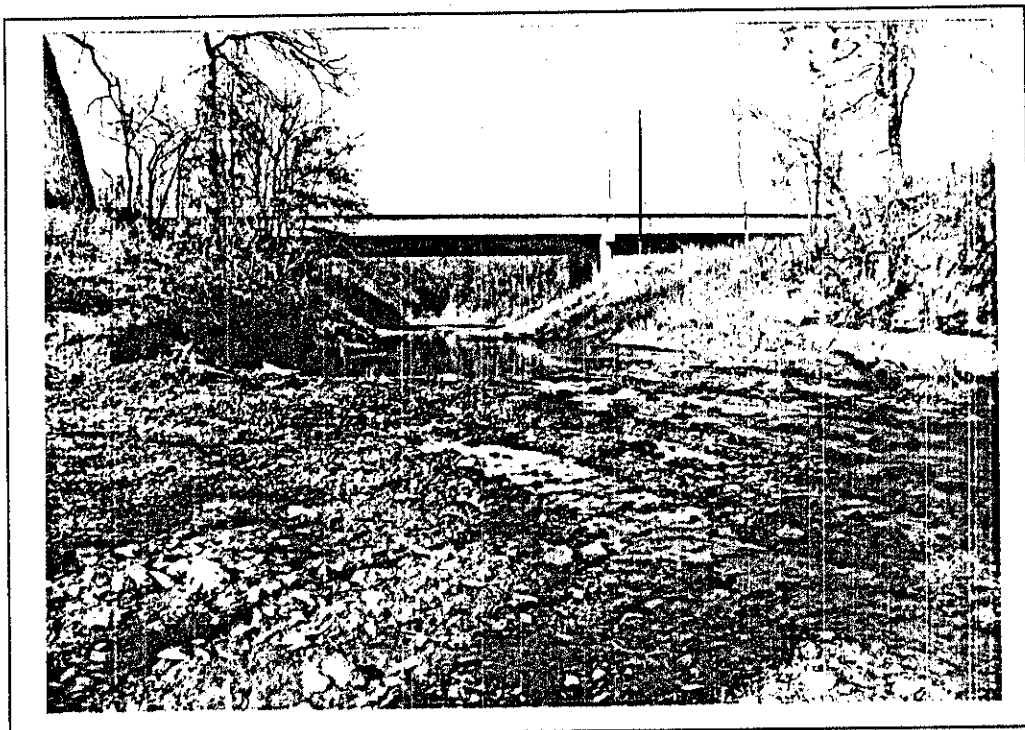


Until these complex interactive factors can be confidently predicted and verified, a simpler approach appears more practical by using bulk sediment numerical data in combination with toxicity tests. Bulk sediment data can be routinely measured and then be compared to an established numerical value similar to EPA and TWC guidelines (Table 14). If the concentration is above or below the numerical value, then no further tests are required and a regulatory decision can be made. If intermediate concentrations appear to be of concern, toxicity tests with a cosmopolitan species could examine actual effects.

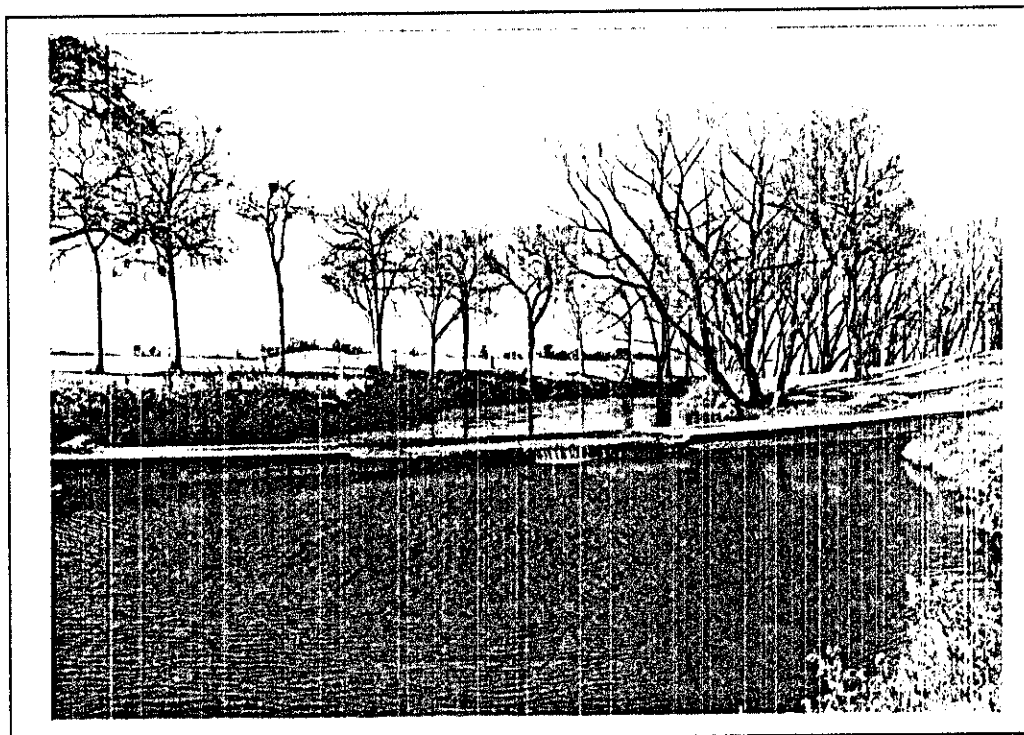
The significant relationships found in the White Rock Creek Watershed, and the numerous among area differences in sorption factors and metal concentrations make it advantageous for evaluating the above methods. Further study would also indicate bioavailability at metal concentrations representative of many urban watersheds.

**APPENDIX A**

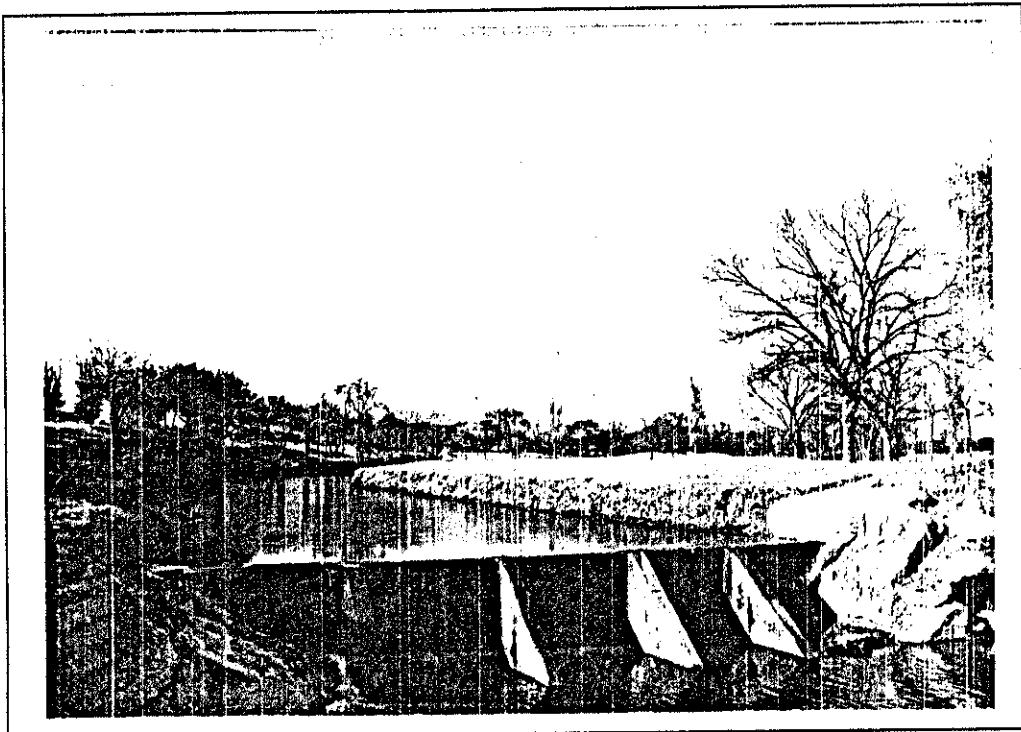
**PHOTOGRAPHS**



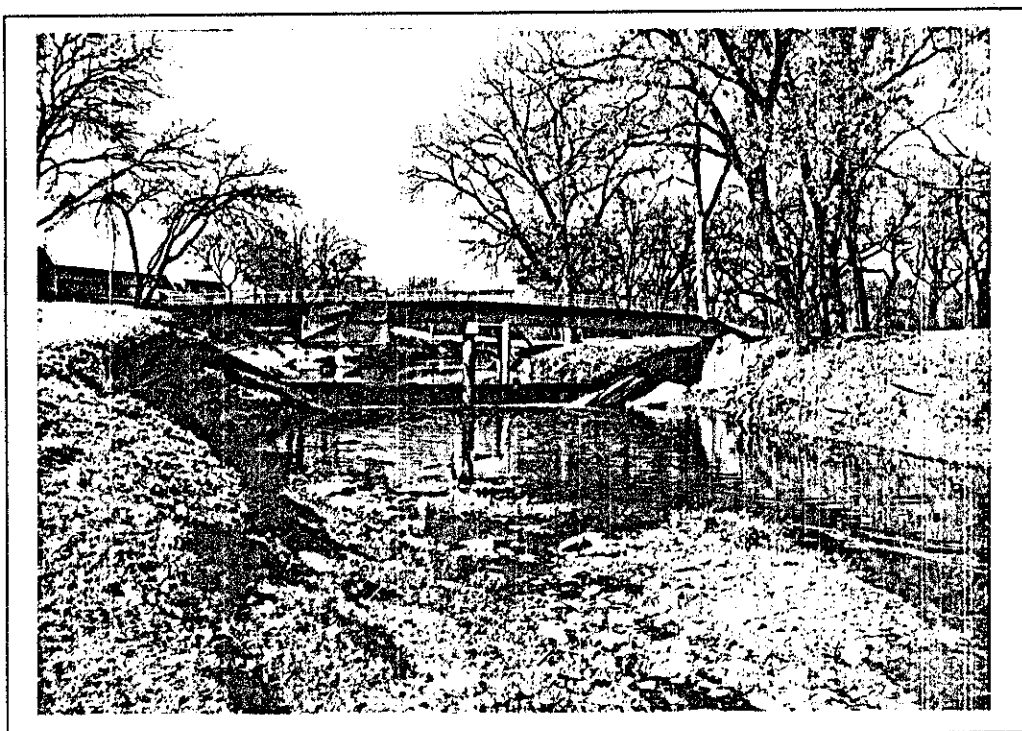
White Rock Creek near the Dallas-Collin County line  
at Frankford Road.



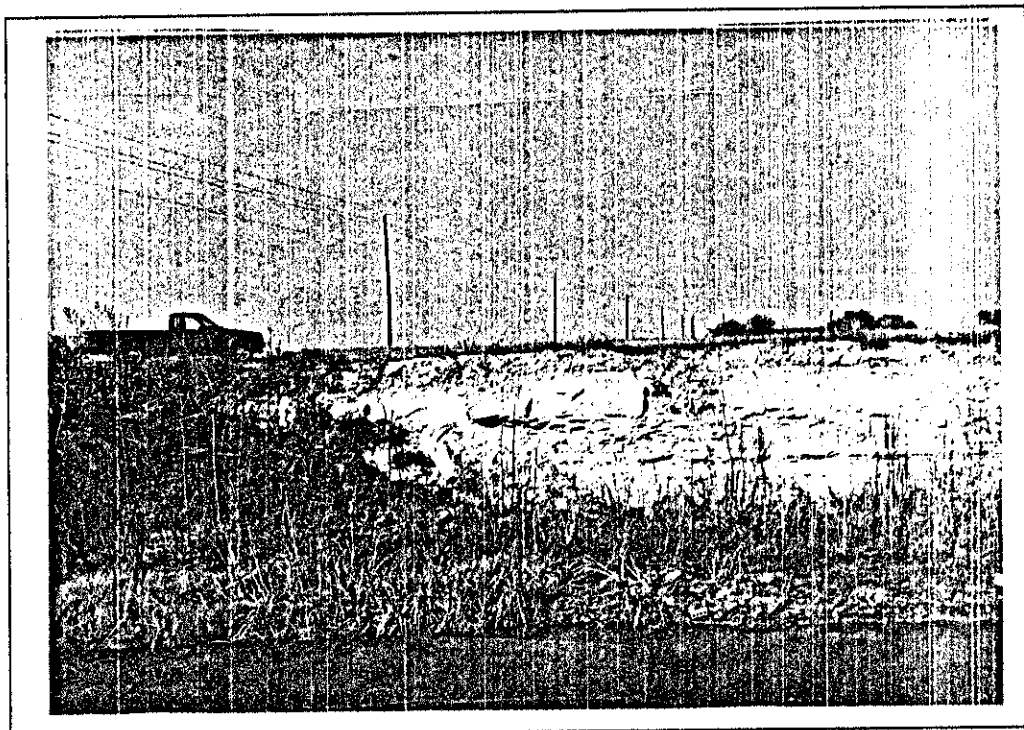
Low level dam at Area A



Low level dam at Area B



Low level dam at Area C



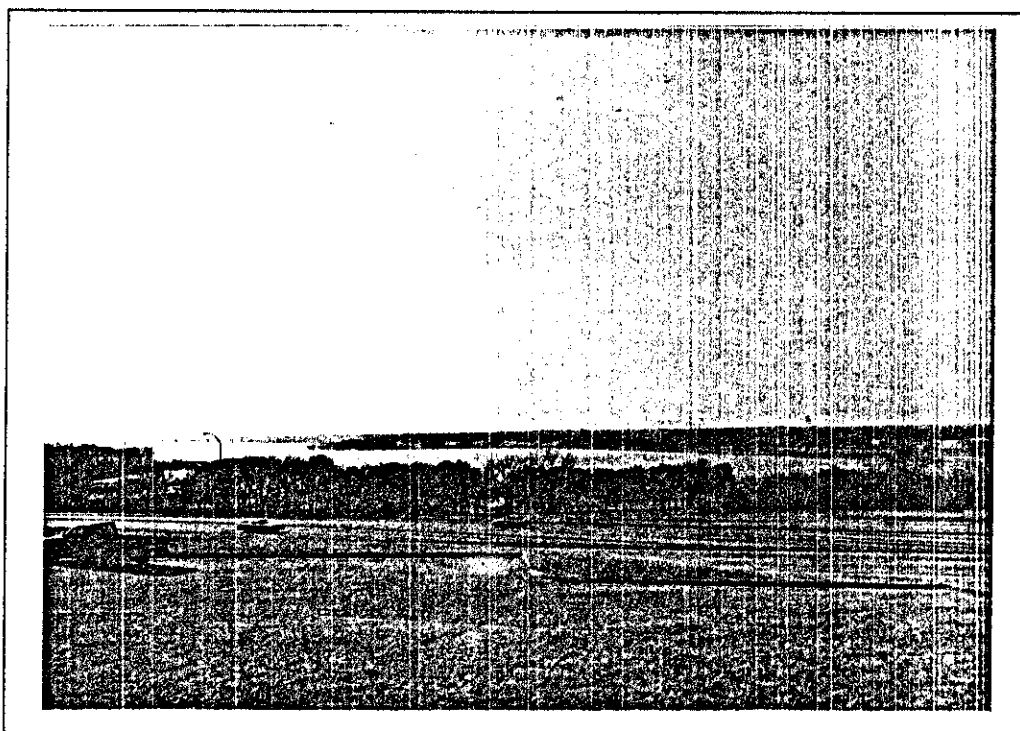
A quarry cut in the lower Austin Chalk unit.



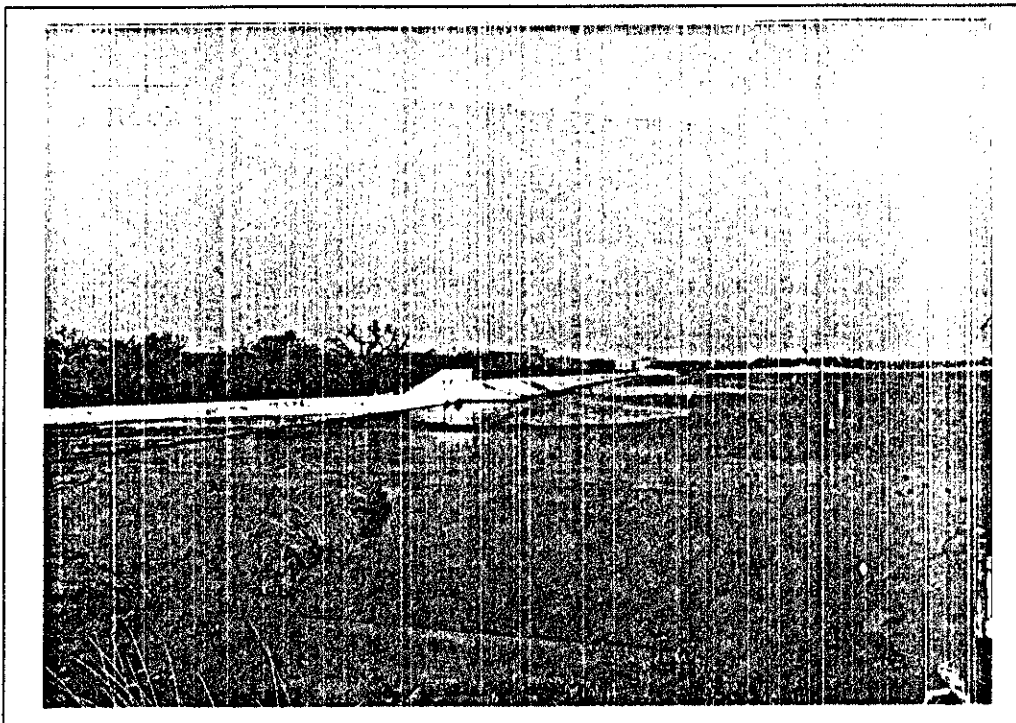
Exposed bedrock typical of many reaches in White Rock Creek.



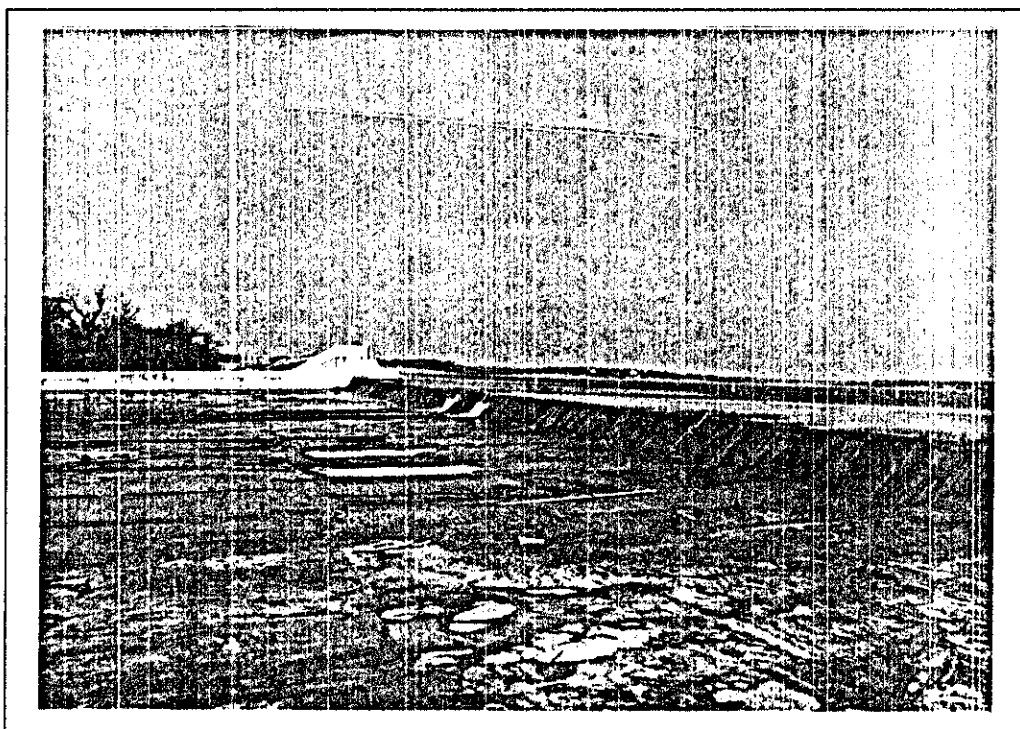
Agricultural area in the watershed's upper section.  
The thin Austin soil is shown in foreground.



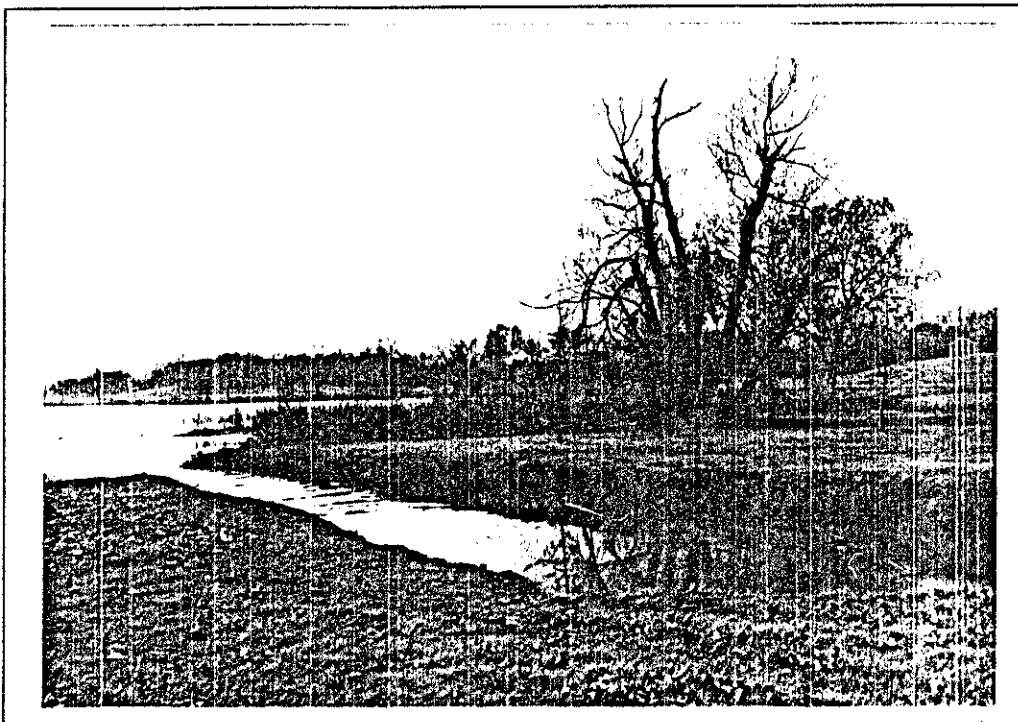
White Rock Lake as viewed from the east.



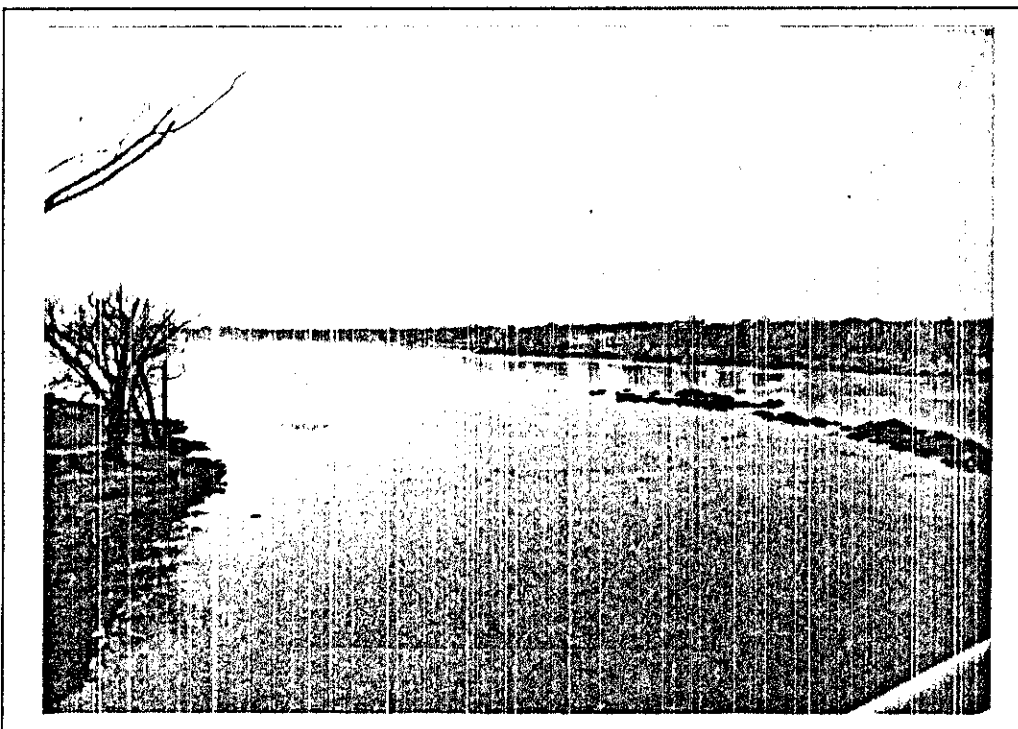
White Rock Lake (right) and dam. Sample Area E is in center of lake at right side of photograph.



White Rock Lake (background) and spillway (foreground)



Low lying delta area at White Rock Lake with wetland grasses and cattails.



White Rock Lake main delta and location of sample area D as viewed south towards lake.



## APPENDIX B

### DATA

White Rock Creek Watershed  
1989 Sample Program  
Data Set Summary

| Sample | Zn    | Pb   | Cr   | Cu     | Cd  | Fe      | Mn     | pct sol | vol sol | % OC | %clay | %silt | %sand |
|--------|-------|------|------|--------|-----|---------|--------|---------|---------|------|-------|-------|-------|
| A1     | 121.3 | 15.0 | 24.6 | 17.636 | 1.5 | 14830.4 | 409.0  | 49.17   | 5.21    | 0.75 | 66.82 | 16.46 | 16.73 |
| A1D    | 112.6 | 16.7 | 23.7 | 18.642 | 2.8 | 14649.0 | 404.0  | 48.89   | 5.55    | 0.82 | 68.41 | 16.49 | 15.09 |
| A2     | 112.9 | 16.9 | 24.1 | 15.154 | 2.1 | 14116.0 | 491.3  | 49.27   | 5.41    | 0.81 | 43.69 | 34.87 | 21.45 |
| A3     | 126.9 | 13.1 | 22.8 | 22.787 | 1.5 | 17563.3 | 318.2  | 38.88   | 6.41    | 0.66 | 53.77 | 43.86 | 2.37  |
| A4     | 122.3 | 14.1 | 23.0 | 18.669 | 1.9 | 17704.8 | 411.5  | 46.90   | 6.14    | 0.75 | 49.72 | 38.19 | 12.09 |
| A5     | 115.6 | 14.4 | 21.7 | 16.679 | 1.9 | 14523.5 | 356.4  | 54.29   | 5.68    | 1.00 | 36.80 | 39.23 | 23.96 |
| B1     | 92.1  | 12.8 | 13.2 | 14.304 | 1.7 | 11100.5 | 324.0  | 67.73   | 2.80    | 0.47 | 16.40 | 24.94 | 58.66 |
| B2     | 77.0  | 12.2 | 11.6 | 11.703 | 1.8 | 8734.3  | 329.6  | 70.12   | 2.17    | 0.45 | 13.72 | 19.82 | 66.46 |
| B3     | 104.5 | 25.2 | 19.8 | 24.237 | 5.0 | 13051.8 | 327.5  | 44.34   | 4.81    | 0.75 | 23.80 | 11.80 | 64.40 |
| B4     | 110.9 | 17.4 | 16.4 | 13.075 | 2.1 | 11812.5 | 406.6  | 64.65   | 3.81    | 0.55 | 16.00 | 23.97 | 60.03 |
| B5     | 93.3  | 14.4 | 14.0 | 13.054 | 2.1 | 10113.1 | 392.6  | 64.08   | 2.67    | 0.83 | 12.87 | 22.39 | 64.74 |
| C1     | 155.4 | 29.5 | 24.8 | 22.839 | 2.6 | 17485.3 | 528.7  | 33.64   | 6.06    | 0.75 | 50.61 | 46.95 | 2.44  |
| C2     | 101.8 | 21.3 | 18.2 | 16.431 | 2.5 | 13473.3 | 547.7  | 63.56   | 3.15    | 0.72 | 25.82 | 37.74 | 36.44 |
| C2D    | 89.3  | 29.3 | 19.0 | 16.740 | 2.6 | 10305.3 | 515.6  | 63.65   | 2.96    | 0.71 | 27.31 | 35.46 | 37.23 |
| C3     | 91.6  | 16.5 | 11.2 | 13.361 | 2.4 | 14951.3 | 361.2  | 73.91   | 1.70    | 0.29 | 7.97  | 16.70 | 75.33 |
| C4     | 87.7  | 18.8 | 12.1 | 12.575 | 2.4 | 12353.0 | 361.5  | 69.82   | 1.71    | 0.31 | 9.28  | 10.18 | 80.54 |
| C5     | 157.6 | 29.2 | 25.4 | 28.499 | 3.6 | 14348.2 | 570.6  | 31.90   | 5.67    | 0.80 | 53.23 | 42.52 | 4.25  |
| D1     | 89.9  | 29.8 | 12.3 | 11.712 | 2.3 | 8971.1  | 436.7  | 71.10   | 2.02    | 0.44 | 17.91 | 21.87 | 60.22 |
| D2     | 114.8 | 19.4 | 13.6 | 13.139 | 2.1 | 8897.3  | 411.2  | 70.94   | 1.90    | 0.42 | 14.77 | 28.42 | 56.81 |
| D3     | 87.1  | 20.2 | 12.8 | 12.992 | 2.4 | 8632.9  | 422.4  | 71.96   | 1.65    | 0.32 | 14.39 | 25.44 | 60.17 |
| D4     | 91.3  | 23.4 | 10.7 | 12.677 | 2.2 | 7187.9  | 395.9  | 74.52   | 1.28    | 0.45 | 10.88 | 18.60 | 70.52 |
| D5     | 76.2  | 23.9 | 11.8 | 31.937 | 2.4 | 7962.7  | 434.3  | 70.81   | 1.68    | 0.39 | 13.47 | 20.85 | 65.68 |
| E1     | 147.6 | 42.6 | 28.7 | 22.512 | 2.6 | 18401.9 | 907.2  | 30.71   | 6.10    | 0.96 | 68.10 | 31.59 | 0.31  |
| E2     | 147.3 | 40.7 | 28.4 | 22.413 | 2.6 | 18488.1 | 888.2  | 30.25   | 5.95    | 0.69 | 59.72 | 40.12 | 0.16  |
| E3     | 157.6 | 46.4 | 29.0 | 25.595 | 2.4 | 17862.8 | 1007.1 | 29.86   | 5.68    | 0.78 | 69.13 | 30.74 | 0.13  |
| E4     | 150.2 | 46.8 | 27.5 | 20.551 | 2.6 | 18748.2 | 934.6  | 31.09   | 5.59    | 0.79 | 68.73 | 31.17 | 0.10  |
| E5     | 156.3 | 44.6 | 30.5 | 22.265 | 2.3 | 17996.1 | 843.0  | 31.37   | 5.13    | 0.83 | 61.21 | 38.67 | 0.12  |
| E5D    | 148.5 | 44.3 | 28.5 | 23.247 | 2.3 | 19531.0 | 882.1  | 31.36   | 4.78    | 0.77 | 66.20 | 33.59 | 0.20  |

QA-QC VALUES:

Average precision from three duplicates and metal spike recoveries:

|           | Zn     | Pb    | Cr    | Cu    | Cd    | Fe    | Mn    | %sol  | vol sol | %OC   | %clay | %silt | %sand |
|-----------|--------|-------|-------|-------|-------|-------|-------|-------|---------|-------|-------|-------|-------|
| % prec.   | 99.92  | 99.84 | 99.95 | 99.96 | 99.72 | 99.89 | 99.96 | 99.99 | 99.94   | 99.94 | 99.95 | 99.94 | 99.74 |
| Avg MSrec | 112.58 | 94.63 | 90.91 | 88.24 | 89.14 | -     | 98.45 | -     | -       | -     | -     | -     | -     |

Note: Avg MSrec is average percent recovery for spiked metal samples.  
All metals reported in mg/kg.

Zn MDL = 0.01 ppm

| sample | pct sol | wet sed | net sol | Concentration (ppm) |      |      |      | dilution | adj conc | mg/kg | %recovery |
|--------|---------|---------|---------|---------------------|------|------|------|----------|----------|-------|-----------|
|        |         |         |         | 1                   | 2    | 3    | avg  |          |          |       |           |
| A1     | 49.17   | 1.978   | 0.973   | 0.6                 | 0.58 | 0.59 | 0.59 | 1:3      | 2.36     | 121.3 |           |
| A1D    | 48.89   | 2.131   | 1.042   | 0.59                | 0.59 | 0.58 | 0.59 | 1:3      | 2.35     | 112.6 |           |
| A1MS   | 49.03   | 1.935   | 0.949   | 0.58                | 0.57 | 0.57 | 0.57 | 1:6      | 3.99     |       | 118.75    |
| A2     | 49.27   | 2.146   | 1.057   | 0.6                 | 0.6  | 0.59 | 0.60 | 1:3      | 2.39     | 112.9 |           |
| A3     | 38.88   | 2.634   | 1.024   | 0.65                | 0.65 | 0.65 | 0.65 | 1:3      | 2.60     | 126.9 |           |
| A4     | 46.90   | 2.174   | 1.020   | 0.63                | 0.62 | 0.62 | 0.62 | 1:3      | 2.49     | 122.3 |           |
| A5     | 54.29   | 1.944   | 1.055   | 0.61                | 0.61 | 0.61 | 0.61 | 1:3      | 2.44     | 115.6 |           |
| B1     | 67.73   | 1.871   | 1.267   | 0.6                 | 0.57 | 0.58 | 0.58 | 1:3      | 2.33     | 92.1  |           |
| B2     | 70.12   | 1.865   | 1.308   | 0.5                 | 0.51 | 0.5  | 0.50 | 1:3      | 2.01     | 77.0  |           |
| B3     | 44.34   | 2.375   | 1.053   | 0.56                | 0.54 | 0.55 | 0.55 | 1:3      | 2.20     | 104.5 |           |
| B4     | 64.65   | 1.664   | 1.076   | 0.6                 | 0.59 | 0.6  | 0.60 | 1:3      | 2.39     | 110.9 |           |
| B5     | 64.08   | 1.606   | 1.029   | 0.48                | 0.48 | 0.48 | 0.48 | 1:3      | 1.92     | 93.3  |           |
| C1     | 33.64   | 2.945   | 0.991   | 0.77                | 0.77 | 0.77 | 0.77 | 1:3      | 3.08     | 155.4 |           |
| C2     | 63.56   | 1.422   | 0.904   | 0.47                | 0.45 | 0.46 | 0.46 | 1:3      | 1.84     | 101.8 |           |
| C2D    | 63.65   | 1.888   | 1.202   | 0.55                | 0.53 | 0.53 | 0.54 | 1:3      | 2.15     | 89.3  |           |
| C2MS   | 63.6    | 1.702   | 1.082   | 0.45                | 0.45 | 0.44 | 0.57 | 1:6      | 3.13     |       | 110.09    |
| C3     | 73.91   | 1.319   | 0.975   | 0.45                | 0.44 | 0.45 | 0.45 | 1:3      | 1.79     | 91.6  |           |
| C4     | 69.82   | 1.6     | 1.117   | 0.49                | 0.49 | 0.49 | 0.49 | 1:3      | 1.96     | 87.7  |           |
| C5     | 31.90   | 3.05    | 0.973   | 0.77                | 0.76 | 0.77 | 0.77 | 1:3      | 3.07     | 157.6 |           |
| D1     | 71.10   | 1.71    | 1.216   | 0.54                | 0.55 | 0.55 | 0.55 | 1:3      | 2.19     | 89.9  |           |
| D2     | 70.94   | 1.367   | 0.970   | 0.55                | 0.56 | 0.56 | 0.56 | 1:3      | 2.23     | 114.8 |           |
| D3     | 71.96   | 1.543   | 1.110   | 0.48                | 0.48 | 0.49 | 0.48 | 1:3      | 1.93     | 87.1  |           |
| D4     | 74.52   | 1.283   | 0.956   | 0.43                | 0.44 | 0.44 | 0.44 | 1:3      | 1.75     | 91.3  |           |
| D5     | 70.81   | 1.618   | 1.146   | 0.43                | 0.44 | 0.44 | 0.44 | 1:3      | 1.75     | 76.2  |           |
| E1     | 30.71   | 3.323   | 1.020   | 0.76                | 0.75 | 0.75 | 0.75 | 1:3      | 3.01     | 147.6 |           |
| E2     | 30.25   | 3.381   | 1.023   | 0.76                | 0.75 | 0.75 | 0.75 | 1:3      | 3.01     | 147.3 |           |
| E3     | 29.86   | 3.188   | 0.952   | 0.75                | 0.75 | 0.75 | 0.75 | 1:3      | 3.00     | 157.6 |           |
| E4     | 31.09   | 3.211   | 0.998   | 0.75                | 0.75 | 0.75 | 0.75 | 1:3      | 3.00     | 150.2 |           |
| E5     | 31.37   | 3.114   | 0.977   | 0.76                | 0.77 | 0.76 | 0.76 | 1:3      | 3.05     | 156.3 |           |
| E5D    | 31.36   | 3.134   | 0.983   | 0.73                | 0.73 | 0.73 | 0.73 | 1:3      | 2.92     | 148.5 |           |
| E5MS   | 31.36   | 3.264   | 1.024   | 0.63                | 0.63 | 0.63 | 0.63 | 1:6      | 4.41     |       | 108.89    |
| Blank1 |         |         |         | 0                   | 0    | 0    | 0.00 |          |          |       |           |
| Blank2 |         |         |         | 0.06                | 0.06 | 0.05 | 0.06 |          |          |       |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.

A1D, C2D, and E5D are lab duplicates.

A1MS, C2MS, and E5MS are spiked samples.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100

where: spike concentration = 1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

Pb MDL = 0.05 ppm

| sample | pct sol | wet sed | net sol | Concentration (ppm) |      |      | avg  | dilution | adj conc | mg/kg | %recovery |
|--------|---------|---------|---------|---------------------|------|------|------|----------|----------|-------|-----------|
|        |         |         |         | 1                   | 2    | 3    |      |          |          |       |           |
| A1     | 49.17   | 1.969   | 0.968   | 0.29                | 0.29 | 0.29 | 0.29 | none     | 0.29     | 15.0  | 93.02     |
| A1D    | 48.89   | 1.959   | 0.958   | 0.32                | 0.32 | 0.32 | 0.32 | none     | 0.32     | 16.7  |           |
| A1MS   | 49.03   | 2.088   | 1.024   | 1.20                | 1.20 | 1.20 | 1.20 | none     | 1.20     | -     |           |
| A2     | 49.27   | 1.925   | 0.948   | 0.31                | 0.32 | 0.33 | 0.32 | none     | 0.32     | 16.9  |           |
| A3     | 38.88   | 2.619   | 1.018   | 0.27                | 0.26 | 0.27 | 0.27 | none     | 0.27     | 13.1  |           |
| A4     | 46.90   | 2.197   | 1.030   | 0.28                | 0.3  | 0.29 | 0.29 | none     | 0.29     | 14.1  | 91.55     |
| A5     | 54.29   | 1.938   | 1.052   | 0.31                | 0.3  | 0.3  | 0.30 | none     | 0.30     | 14.4  |           |
| B1     | 67.73   | 1.75    | 1.185   | 0.3                 | 0.3  | 0.31 | 0.30 | none     | 0.30     | 12.8  |           |
| B2     | 70.12   | 1.592   | 1.116   | 0.27                | 0.28 | 0.27 | 0.27 | none     | 0.27     | 12.2  |           |
| B3     | 44.34   | 0.909   | 0.403   | 0.19                | 0.21 | 0.21 | 0.20 | none     | 0.20     | 25.2  |           |
| B4     | 64.65   | 1.571   | 1.016   | 0.35                | 0.36 | 0.35 | 0.35 | none     | 0.35     | 17.4  | 99.33     |
| B5     | 64.08   | 1.872   | 1.200   | 0.34                | 0.35 | 0.35 | 0.35 | none     | 0.35     | 14.4  |           |
| C1     | 33.64   | 2.901   | 0.976   | 0.59                | 0.56 | 0.58 | 0.58 | none     | 0.58     | 29.5  |           |
| C2     | 63.56   | 1.56    | 0.992   | 0.42                | 0.43 | 0.42 | 0.42 | none     | 0.42     | 21.3  |           |
| C2D    | 63.65   | 1.502   | 0.956   | 0.58                | 0.56 | 0.54 | 0.56 | none     | 0.56     | 29.3  |           |
| C2MS   | 63.6    | 1.559   | 0.992   | 1.30                | 1.29 | 1.31 | 1.30 | none     | 1.30     | -     | 99.33     |
| C3     | 73.91   | 1.708   | 1.262   | 0.41                | 0.42 | 0.42 | 0.42 | none     | 0.42     | 16.5  |           |
| C4     | 69.82   | 1.462   | 1.021   | 0.38                | 0.39 | 0.38 | 0.38 | none     | 0.38     | 18.8  |           |
| C5     | 31.90   | 2.181   | 0.696   | 0.41                | 0.4  | 0.41 | 0.41 | none     | 0.41     | 29.2  |           |
| D1     | 71.10   | 1.504   | 1.069   | 0.64                | 0.63 | 0.64 | 0.64 | none     | 0.64     | 29.8  |           |
| D2     | 70.94   | 1.33    | 0.944   | 0.37                | 0.37 | 0.36 | 0.37 | none     | 0.37     | 19.4  | 99.33     |
| D3     | 71.96   | 1.375   | 0.989   | 0.4                 | 0.39 | 0.41 | 0.40 | none     | 0.40     | 20.2  |           |
| D4     | 74.52   | 1.522   | 1.134   | 0.53                | 0.54 | 0.52 | 0.53 | none     | 0.53     | 23.4  |           |
| D5     | 70.81   | 1.772   | 1.255   | 0.61                | 0.59 | 0.6  | 0.60 | none     | 0.60     | 23.9  |           |
| E1     | 30.71   | 3.123   | 0.959   | 0.82                | 0.81 | 0.82 | 0.82 | none     | 0.82     | 42.6  |           |
| E2     | 30.25   | 3.238   | 0.980   | 0.8                 | 0.79 | 0.8  | 0.80 | none     | 0.80     | 40.7  | 99.33     |
| E3     | 29.86   | 3.478   | 1.039   | 0.96                | 0.95 | 0.98 | 0.96 | none     | 0.96     | 46.4  |           |
| E4     | 31.09   | 3.782   | 1.176   | 1.09                | 1.11 | 1.1  | 1.10 | none     | 1.10     | 46.8  |           |
| E5     | 31.37   | 3.517   | 1.103   | 1.01                | 0.98 | 0.96 | 0.98 | none     | 0.98     | 44.6  |           |
| E5D    | 31.36   | 3.47    | 1.088   | 0.95                | 0.96 | 0.98 | 0.96 | none     | 0.96     | 44.3  |           |
| E5MS   | 31.36   | 3.512   | 1.101   | 2                   | 1.96 | 1.95 | 1.97 | none     | 1.97     | 89.4  | 99.33     |
| Blank1 |         |         | 0.000   | 0.05                | 0.07 | 0.07 | 0.06 | none     | 0.06     | -     |           |
| Blank2 |         |         | 0.000   | 0.05                | 0.04 | 0.05 | 0.05 | none     | 0.05     | -     |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.

A1D, C2D, and E5D are lab duplicates.

A1MS, C2MS, and E5MS are spiked samples.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100

where: spike concentration = 1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

Cr MDL = 0.02 ppm

| sample | pct sol | wet sed | net sol | Concentration (ppm) |      |      | avg  | dilution | adj conc | mg/kg | %recovery |
|--------|---------|---------|---------|---------------------|------|------|------|----------|----------|-------|-----------|
|        |         |         |         | 1                   | 2    | 3    |      |          |          |       |           |
| A1     | 49.17   | 1.969   | 0.968   | 0.48                | 0.47 | 0.48 | 0.48 | none     | 0.48     | 24.6  | 86.49     |
| A1D    | 48.89   | 1.959   | 0.958   | 0.45                | 0.45 | 0.46 | 0.45 | none     | 0.45     | 23.7  |           |
| A1MS   | 49.03   | 2.088   | 1.024   | 0.32                | 0.32 | 0.32 | 0.32 | 1:3      | 1.28     |       |           |
| A2     | 49.27   | 1.925   | 0.948   | 0.46                | 0.46 | 0.45 | 0.46 | none     | 0.46     | 24.1  |           |
| A3     | 38.88   | 2.619   | 1.018   | 0.46                | 0.47 | 0.46 | 0.46 | none     | 0.46     | 22.8  |           |
| A4     | 46.90   | 2.197   | 1.030   | 0.48                | 0.47 | 0.47 | 0.47 | none     | 0.47     | 23.0  |           |
| A5     | 54.29   | 1.938   | 1.052   | 0.46                | 0.45 | 0.46 | 0.46 | none     | 0.46     | 21.7  |           |
| B1     | 67.73   | 1.75    | 1.185   | 0.32                | 0.31 | 0.31 | 0.31 | none     | 0.31     | 13.2  |           |
| B2     | 70.12   | 1.592   | 1.116   | 0.26                | 0.26 | 0.26 | 0.26 | none     | 0.26     | 11.6  |           |
| B3     | 44.34   | 0.909   | 0.403   | 0.16                | 0.16 | 0.16 | 0.16 | none     | 0.16     | 19.8  |           |
| B4     | 64.65   | 1.571   | 1.016   | 0.33                | 0.33 | 0.34 | 0.33 | none     | 0.33     | 16.4  | 88.24     |
| B5     | 64.08   | 1.872   | 1.200   | 0.34                | 0.34 | 0.33 | 0.34 | none     | 0.34     | 14.0  |           |
| C1     | 33.64   | 2.901   | 0.976   | 0.48                | 0.49 | 0.48 | 0.48 | none     | 0.48     | 24.8  |           |
| C2     | 63.56   | 1.56    | 0.992   | 0.36                | 0.36 | 0.36 | 0.36 | none     | 0.36     | 18.2  |           |
| C2D    | 63.65   | 1.502   | 0.956   | 0.36                | 0.37 | 0.36 | 0.36 | none     | 0.36     | 19.0  |           |
| C2MS   | 63.6    | 1.559   | 0.992   | 0.94                | 0.30 | 0.30 | 0.30 | 1:3      | 1.20     |       |           |
| C3     | 73.91   | 1.708   | 1.262   | 0.28                | 0.28 | 0.29 | 0.28 | none     | 0.28     | 11.2  |           |
| C4     | 69.82   | 1.462   | 1.021   | 0.25                | 0.24 | 0.25 | 0.25 | none     | 0.25     | 12.1  |           |
| C5     | 31.90   | 2.181   | 0.696   | 0.35                | 0.36 | 0.35 | 0.35 | none     | 0.35     | 25.4  |           |
| D1     | 71.10   | 1.504   | 1.069   | 0.26                | 0.26 | 0.27 | 0.26 | none     | 0.26     | 12.3  | 98.01     |
| D2     | 70.94   | 1.33    | 0.944   | 0.26                | 0.25 | 0.26 | 0.26 | none     | 0.26     | 13.6  |           |
| D3     | 71.96   | 1.375   | 0.989   | 0.25                | 0.25 | 0.26 | 0.25 | none     | 0.25     | 12.8  |           |
| D4     | 74.52   | 1.522   | 1.134   | 0.24                | 0.25 | 0.24 | 0.24 | none     | 0.24     | 10.7  |           |
| D5     | 70.81   | 1.772   | 1.255   | 0.3                 | 0.3  | 0.29 | 0.30 | none     | 0.30     | 11.8  |           |
| E1     | 30.71   | 3.123   | 0.959   | 0.54                | 0.55 | 0.56 | 0.55 | none     | 0.55     | 28.7  |           |
| E2     | 30.25   | 3.238   | 0.980   | 0.56                | 0.55 | 0.56 | 0.56 | none     | 0.56     | 28.4  |           |
| E3     | 29.86   | 3.478   | 1.039   | 0.61                | 0.6  | 0.6  | 0.60 | none     | 0.60     | 29.0  |           |
| E4     | 31.09   | 3.782   | 1.176   | 0.65                | 0.64 | 0.65 | 0.65 | none     | 0.65     | 27.5  |           |
| E5     | 31.37   | 3.517   | 1.103   | 0.67                | 0.67 | 0.68 | 0.67 | none     | 0.67     | 30.5  |           |
| E5D    | 31.36   | 3.47    | 1.088   | 0.62                | 0.62 | 0.62 | 0.62 | none     | 0.62     | 28.5  | 98.01     |
| E5MS   | 31.36   | 3.512   | 1.101   | 0.41                | 0.41 | 0.41 | 0.41 | 1:3      | 1.64     |       |           |
| Blank1 |         |         | 0.000   | 0                   | 0    | 0    | 0.00 | none     | 0.00     |       |           |
| Blank2 |         |         | 0.000   | 0.01                | 0.01 | 0.01 | 0.01 | none     | 0.01     |       |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.

A1D, C2D, and E5D are lab duplicates.

A1MS, C2MS, and E5MS are spiked samples.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100

where: spike concentration = 1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

Cu MDL = 0.01 ppm

| sample | pct sol | wet sed | net sol | Concentration (ppm) |       |       | dilution | adj conc | mg/kg  | %recovery |
|--------|---------|---------|---------|---------------------|-------|-------|----------|----------|--------|-----------|
|        |         |         |         | 1                   | 2     | avg   |          |          |        |           |
| A1     | 49.17   | 2.191   | 1.077   | 0.38                | 0.380 | 0.38  | none     | 0.38     | 17.636 |           |
| A1D    | 48.89   | 2.112   | 1.033   | 0.38                | 0.390 | 0.385 | none     | 0.385    | 18.642 |           |
| A2     | 49.27   | 2.009   | 0.990   | 0.3                 | 0.300 | 0.3   | none     | 0.3      | 15.154 |           |
| A3     | 38.88   | 2.737   | 1.064   | 0.49                | 0.480 | 0.485 | none     | 0.485    | 22.787 |           |
| A4     | 46.90   | 2.056   | 0.964   | 0.36                | 0.360 | 0.36  | none     | 0.36     | 18.669 |           |
| A5     | 54.29   | 1.767   | 0.959   | 0.32                | 0.320 | 0.32  | none     | 0.32     | 16.679 |           |
| B1     | 67.73   | 1.6     | 1.084   | 0.31                | 0.310 | 0.31  | none     | 0.31     | 14.304 |           |
| B2     | 70.12   | 1.706   | 1.196   | 0.28                | 0.280 | 0.28  | none     | 0.28     | 11.703 |           |
| B3     | 44.34   | 2.117   | 0.939   | 0.46                | 0.450 | 0.455 | none     | 0.455    | 24.237 |           |
| B4     | 64.65   | 1.538   | 0.994   | 0.26                | 0.260 | 0.26  | none     | 0.26     | 13.075 |           |
| B5     | 64.08   | 1.853   | 1.187   | 0.31                | 0.310 | 0.31  | none     | 0.31     | 13.054 |           |
| C1     | 33.64   | 3.026   | 1.018   | 0.47                | 0.460 | 0.465 | none     | 0.465    | 22.839 |           |
| C2     | 63.56   | 1.556   | 0.989   | 0.33                | 0.32  | 0.325 | none     | 0.325    | 16.431 |           |
| C2D    | 63.65   | 1.525   | 0.971   | 0.33                | 0.32  | 0.325 | none     | 0.325    | 16.740 |           |
| C2MS   | 63.6    | 1.607   | 1.022   | 0.38                | 0.37  | 0.375 | none     | 0.375    | 18.345 | 88.24     |
| C3     | 73.91   | 1.367   | 1.010   | 0.27                | 0.27  | 0.27  | none     | 0.27     | 13.361 |           |
| C4     | 69.82   | 1.566   | 1.093   | 0.27                | 0.28  | 0.275 | none     | 0.275    | 12.575 |           |
| C5     | 31.90   | 3.19    | 1.018   | 0.58                | 0.58  | 0.58  | none     | 0.58     | 28.499 |           |
| D1     | 71.10   | 1.381   | 0.982   | 0.23                | 0.23  | 0.23  | none     | 0.23     | 11.712 |           |
| D2     | 70.94   | 1.341   | 0.951   | 0.25                | 0.25  | 0.25  | none     | 0.25     | 13.139 |           |
| D3     | 71.96   | 1.444   | 1.039   | 0.27                | 0.27  | 0.27  | none     | 0.27     | 12.992 |           |
| D4     | 74.52   | 1.429   | 1.065   | 0.27                | 0.27  | 0.27  | none     | 0.27     | 12.677 |           |
| D5     | 70.81   | 1.415   | 1.002   | 0.64                | 0.64  | 0.64  | none     | 0.64     | 31.937 |           |
| E1     | 30.71   | 3.544   | 1.088   | 0.49                | 0.49  | 0.49  | none     | 0.49     | 22.512 |           |
| E2     | 30.25   | 3.208   | 0.970   | 0.43                | 0.44  | 0.435 | none     | 0.435    | 22.413 |           |
| E3     | 29.86   | 3.238   | 0.967   | 0.5                 | 0.49  | 0.495 | none     | 0.495    | 25.595 |           |
| E4     | 31.09   | 3.13    | 0.973   | 0.4                 | 0.4   | 0.4   | none     | 0.4      | 20.551 |           |
| E5     | 31.37   | 3.365   | 1.055   | 0.47                | 0.47  | 0.47  | none     | 0.47     | 22.265 |           |
| E5D    | 31.36   | 3.223   | 1.011   | 0.47                | 0.47  | 0.47  | none     | 0.47     | 23.247 |           |
| Blank1 |         |         |         | 0                   | 0     | 0     | none     | 0        |        |           |
| Blank2 |         |         |         | 0                   | 0     | 0     | none     | 0        |        |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.  
 A1D, C2D, and E5D are lab duplicates.  
 C2MS is a spiked sample.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100  
 where: spike concentration = 0.1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

Cd MDL = 0.01 ppm

| sample | pct sol | wet sed | net sol | Concentration (ppm) |      |      | avg  | dilution | adj conc | mg/kg | %recovery |
|--------|---------|---------|---------|---------------------|------|------|------|----------|----------|-------|-----------|
|        |         |         |         | 1                   | 2    | 3    |      |          |          |       |           |
| A1     | 49.17   | 1.969   | 0.968   | 0.03                | 0.03 | 0.03 | 0.03 | none     | 0.03     | 1.5   |           |
| A1D    | 48.89   | 1.959   | 0.958   | 0.06                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.8   |           |
| A1MS   | 49.03   | 2.088   | 1.024   | 0.92                | 0.92 | 0.92 | 0.92 | none     | 0.92     | -     | 89.32     |
| A2     | 49.27   | 1.925   | 0.948   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 2.1   |           |
| A3     | 38.88   | 2.619   | 1.018   | 0.03                | 0.03 | 0.03 | 0.03 | none     | 0.03     | 1.5   |           |
| A4     | 46.90   | 2.197   | 1.030   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 1.9   |           |
| A5     | 54.29   | 1.938   | 1.052   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 1.9   |           |
| B1     | 67.73   | 1.75    | 1.185   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 1.7   |           |
| B2     | 70.12   | 1.592   | 1.116   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 1.8   |           |
| B3     | 44.34   | 0.909   | 0.403   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 5.0   |           |
| B4     | 64.65   | 1.571   | 1.016   | 0.04                | 0.05 | 0.04 | 0.04 | none     | 0.04     | 2.1   |           |
| B5     | 64.08   | 1.872   | 1.200   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.1   |           |
| C1     | 33.64   | 2.901   | 0.976   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.6   |           |
| C2     | 63.56   | 1.56    | 0.992   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.5   |           |
| C2D    | 63.65   | 1.502   | 0.956   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.6   |           |
| C2MS   | 63.6    | 1.559   | 0.992   | 0.93                | 0.93 | 0.93 | 0.93 | none     | 0.93     | -     | 88.57     |
| C3     | 73.91   | 1.708   | 1.262   | 0.06                | 0.06 | 0.06 | 0.06 | none     | 0.06     | 2.4   |           |
| C4     | 69.82   | 1.462   | 1.021   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.4   |           |
| C5     | 31.90   | 2.181   | 0.696   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 3.6   |           |
| D1     | 71.10   | 1.504   | 1.069   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.3   |           |
| D2     | 70.94   | 1.33    | 0.944   | 0.04                | 0.04 | 0.04 | 0.04 | none     | 0.04     | 2.1   |           |
| D3     | 71.96   | 1.375   | 0.989   | 0.05                | 0.04 | 0.05 | 0.05 | none     | 0.05     | 2.4   |           |
| D4     | 74.52   | 1.522   | 1.134   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.2   |           |
| D5     | 70.81   | 1.772   | 1.255   | 0.06                | 0.06 | 0.06 | 0.06 | none     | 0.06     | 2.4   |           |
| E1     | 30.71   | 3.123   | 0.959   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.6   |           |
| E2     | 30.25   | 3.238   | 0.980   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.6   |           |
| E3     | 29.86   | 3.478   | 1.039   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.4   |           |
| E4     | 31.09   | 3.782   | 1.176   | 0.06                | 0.06 | 0.06 | 0.06 | none     | 0.06     | 2.6   |           |
| E5     | 31.37   | 3.517   | 1.103   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.3   |           |
| E5D    | 31.36   | 3.47    | 1.088   | 0.05                | 0.05 | 0.05 | 0.05 | none     | 0.05     | 2.3   |           |
| E5MS   | 31.36   | 3.512   | 1.101   | 0.89                | 0.93 | 0.93 | 0.93 | none     | 0.93     | -     | 89.52     |
| Blank1 |         |         | 0.000   | 0                   | 0    | 0    | 0.00 | none     | 0.00     | -     |           |
| Blank2 |         |         | 0.000   | 0                   | 0    | 0    | 0.00 | none     | 0.00     | -     |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.

A1D, C2D, and E5D are lab duplicates.

A1MS, C2MS, and E5MS are spiked samples.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100

where: spike concentration = 1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

Fe MDL = 0.05

| sample | pct sol | wet sed | net sol | Concentration (ppm) |       |       | dilution | adj conc | mg/kg   |
|--------|---------|---------|---------|---------------------|-------|-------|----------|----------|---------|
|        |         |         |         | 1                   | 2     | avg   |          |          |         |
| A1     | 49.17   | 2.191   | 1.077   | 3.96                | 3.930 | 3.945 | 1:80     | 319.545  | 14830.4 |
| A1D    | 48.89   | 2.112   | 1.033   | 3.74                | 3.730 | 3.735 | 1:80     | 302.535  | 14649.0 |
| A2     | 49.27   | 2.009   | 0.990   | 3.47                | 3.430 | 3.45  | 1:80     | 279.45   | 14116.0 |
| A3     | 38.88   | 2.737   | 1.064   | 4.64                | 4.590 | 4.615 | 1:80     | 373.815  | 17563.3 |
| A4     | 46.90   | 2.056   | 0.964   | 4.23                | 4.200 | 4.215 | 1:80     | 341.415  | 17704.8 |
| A5     | 54.29   | 1.767   | 0.959   | 3.45                | 3.430 | 3.44  | 1:80     | 278.64   | 14523.5 |
| B1     | 67.73   | 1.6     | 1.084   | 2.97                | 2.970 | 2.97  | 1:80     | 240.57   | 11100.5 |
| B2     | 70.12   | 1.706   | 1.196   | 2.59                | 2.570 | 2.58  | 1:80     | 208.98   | 8734.3  |
| B3     | 44.34   | 2.117   | 0.939   | 3.04                | 3.010 | 3.025 | 1:80     | 245.025  | 13051.8 |
| B4     | 64.65   | 1.538   | 0.994   | 2.92                | 2.880 | 2.9   | 1:80     | 234.9    | 11812.5 |
| B5     | 64.08   | 1.853   | 1.187   | 2.98                | 2.950 | 2.965 | 1:80     | 240.165  | 10113.1 |
| C1     | 33.64   | 3.026   | 1.018   | 4.4                 | 4.390 | 4.395 | 1:80     | 355.995  | 17485.3 |
| C2     | 63.56   | 1.556   | 0.989   | 3.3                 | 3.28  | 3.29  | 1:80     | 266.49   | 13473.3 |
| C2D    | 63.65   | 1.525   | 0.971   | 2.48                | 2.46  | 2.47  | 1:80     | 200.07   | 10305.3 |
| C3     | 73.91   | 1.367   | 1.010   | 3.75                | 3.71  | 3.73  | 1:80     | 302.13   | 14951.3 |
| C4     | 69.82   | 1.566   | 1.093   | 3.35                | 3.32  | 3.335 | 1:80     | 270.135  | 12353.0 |
| C5     | 31.90   | 3.19    | 1.018   | 3.62                | 3.59  | 3.605 | 1:80     | 292.005  | 14348.2 |
| D1     | 71.10   | 1.381   | 0.982   | 2.18                | 2.17  | 2.175 | 1:80     | 176.175  | 8971.1  |
| D2     | 70.94   | 1.341   | 0.951   | 2.1                 | 2.08  | 2.09  | 1:80     | 169.29   | 8897.3  |
| D3     | 71.96   | 1.444   | 1.039   | 2.21                | 2.22  | 2.215 | 1:80     | 179.415  | 8632.9  |
| D4     | 74.52   | 1.429   | 1.065   | 1.91                | 1.87  | 1.89  | 1:80     | 153.09   | 7187.9  |
| D5     | 70.81   | 1.415   | 1.002   | 1.98                | 1.96  | 1.97  | 1:80     | 159.57   | 7962.7  |
| E1     | 30.71   | 3.544   | 1.088   | 4.94                | 4.95  | 4.945 | 1:80     | 400.545  | 18401.9 |
| E2     | 30.25   | 3.208   | 0.970   | 4.44                | 4.42  | 4.43  | 1:80     | 358.83   | 18488.1 |
| E3     | 29.86   | 3.238   | 0.967   | 4.29                | 4.24  | 4.265 | 1:80     | 345.465  | 17862.8 |
| E4     | 31.09   | 3.13    | 0.973   | 4.53                | 4.48  | 4.505 | 1:80     | 364.905  | 18748.2 |
| E5     | 31.37   | 3.365   | 1.055   | 4.71                | 4.67  | 4.69  | 1:80     | 379.89   | 17996.1 |
| E5D    | 31.36   | 3.223   | 1.011   | 4.88                | 4.87  | 4.875 | 1:80     | 394.875  | 19531.0 |
| Blank1 |         |         |         | 0.88                | 0.89  | 0.885 | none     |          |         |
| Blank2 |         |         |         | 0.65                | 0.65  | 0.65  | none     |          |         |

Note: %recovery not calculated for Fe because of high concentrations.

#### Explanation:

The blank samples consisted of aqua regia only and no sediment.  
A1D, C2D, and E5D are lab duplicates.

To calculate mg/kg: 
$$\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$$

where vol. digestate = 0.05 L



Mn MDL = 0.05

| sample | pct sol | wet sed | net sol | Concentration (ppm) |      |      | avg  | dilution | adj conc | mg/kg  | %recovery |
|--------|---------|---------|---------|---------------------|------|------|------|----------|----------|--------|-----------|
|        |         |         |         | 1                   | 2    | 3    |      |          |          |        |           |
| A1     | 49.17   | 1.969   | 0.968   | 1.32                | 1.32 | 1.32 | 1.32 | 1:5      | 7.92     | 409.0  | 111.66    |
| A1D    | 48.89   | 1.959   | 0.958   | 1.29                | 1.29 | 1.29 | 1.29 | 1:5      | 7.74     | 404.0  |           |
| A1MS   | 49.03   | 2.088   | 1.024   | 1.66                | 1.66 | 1.66 | 1.66 | 1:5      | 9.96     | 486.4  |           |
| A2     | 49.27   | 1.925   | 0.948   | 1.55                | 1.55 | 1.56 | 1.55 | 1:5      | 9.32     | 491.3  |           |
| A3     | 38.88   | 2.619   | 1.018   | 1.08                | 1.08 | 1.08 | 1.08 | 1:5      | 6.48     | 318.2  |           |
| A4     | 46.90   | 2.197   | 1.030   | 1.41                | 1.42 | 1.41 | 1.41 | 1:5      | 8.48     | 411.5  | 82.97     |
| A5     | 54.29   | 1.938   | 1.052   | 1.25                | 1.25 | 1.25 | 1.25 | 1:5      | 7.5      | 356.4  |           |
| B1     | 67.73   | 1.75    | 1.185   | 1.28                | 1.28 | 1.28 | 1.28 | 1:5      | 7.68     | 324.0  |           |
| B2     | 70.12   | 1.592   | 1.116   | 1.23                | 1.22 | 1.23 | 1.23 | 1:5      | 7.36     | 329.6  |           |
| B3     | 44.34   | 0.909   | 0.403   | 0.44                | 0.44 | 0.44 | 0.44 | 1:5      | 2.64     | 327.5  |           |
| B4     | 64.65   | 1.571   | 1.016   | 1.38                | 1.37 | 1.38 | 1.38 | 1:5      | 8.26     | 406.6  | 100.71    |
| B5     | 64.08   | 1.872   | 1.200   | 1.57                | 1.57 | 1.57 | 1.57 | 1:5      | 9.42     | 392.6  |           |
| C1     | 33.64   | 2.901   | 0.976   | 1.72                | 1.72 | 1.72 | 1.72 | 1:5      | 10.32    | 528.7  |           |
| C2     | 63.56   | 1.56    | 0.992   | 1.81                | 1.81 | 1.81 | 1.81 | 1:5      | 10.86    | 547.7  |           |
| C2D    | 63.65   | 1.502   | 0.956   | 1.64                | 1.65 | 1.64 | 1.64 | 1:5      | 9.86     | 515.6  |           |
| C2MS   | 63.6    | 1.559   | 0.992   | 1.64                | 1.64 | 1.64 | 1.64 | 1:5      | 9.84     | 496.2  | 100.71    |
| C3     | 73.91   | 1.708   | 1.262   | 1.52                | 1.52 | 1.52 | 1.52 | 1:5      | 9.12     | 361.2  |           |
| C4     | 69.82   | 1.462   | 1.021   | 1.23                | 1.23 | 1.23 | 1.23 | 1:5      | 7.38     | 361.5  |           |
| C5     | 31.90   | 2.181   | 0.696   | 1.32                | 1.33 | 1.32 | 1.32 | 1:5      | 7.94     | 570.6  |           |
| D1     | 71.10   | 1.504   | 1.069   | 1.56                | 1.56 | 1.55 | 1.56 | 1:5      | 9.34     | 436.7  |           |
| D2     | 70.94   | 1.33    | 0.944   | 1.29                | 1.3  | 1.29 | 1.29 | 1:5      | 7.76     | 411.2  | 100.71    |
| D3     | 71.96   | 1.375   | 0.989   | 1.39                | 1.39 | 1.4  | 1.39 | 1:5      | 8.36     | 422.4  |           |
| D4     | 74.52   | 1.522   | 1.134   | 1.49                | 1.5  | 1.5  | 1.50 | 1:5      | 8.98     | 395.9  |           |
| D5     | 70.81   | 1.772   | 1.255   | 1.82                | 1.82 | 1.81 | 1.82 | 1:5      | 10.9     | 434.3  |           |
| E1     | 30.71   | 3.123   | 0.959   | 2.89                | 2.9  | 2.91 | 2.90 | 1:5      | 17.4     | 907.2  |           |
| E2     | 30.25   | 3.238   | 0.980   | 2.89                | 2.91 | 2.9  | 2.90 | 1:5      | 17.4     | 888.2  | 100.71    |
| E3     | 29.86   | 3.478   | 1.039   | 3.49                | 3.48 | 3.49 | 3.49 | 1:5      | 20.92    | 1007.1 |           |
| E4     | 31.09   | 3.782   | 1.176   | 3.66                | 3.66 | 3.67 | 3.66 | 1:5      | 21.98    | 934.6  |           |
| E5     | 31.37   | 3.517   | 1.103   | 3.1                 | 3.1  | 3.1  | 3.10 | 1:5      | 18.6     | 843.0  |           |
| E5D    | 31.36   | 3.47    | 1.088   | 3.2                 | 3.2  | 3.2  | 3.20 | 1:5      | 19.2     | 882.1  |           |
| E5MS   | 31.36   | 3.512   | 1.101   | 3.3                 | 3.3  | 3.3  | 3.29 | 1:5      | 19.74    | 896.2  | 100.71    |
| Blank1 |         |         | 0.000   | 0.01                | 0.01 | 0.01 | 0.01 |          |          |        |           |
| Blank2 |         |         | 0.000   | 0.01                | 0.01 | 0.01 | 0.01 |          |          |        |           |

## Explanation:

The blank samples consisted of aqua regia only and no sediment.

A1D, C2D, and E5D are lab duplicates.

A1MS, C2MS, and E5MS are spiked samples.

%recovery = MS avg (ppm) / (unspiked spl + spike concentration) \* 100  
 where: spike concentration = 1ppm

To calculate mg/kg:  $\frac{\text{avg conc. (mg/L)} * \text{vol. digestate (L)}}{\text{dry wt. of sample (g)}} * 1000 \text{ g/kg}$

where vol. digestate = 0.05 L

WHITE ROCK CREEK WATERSHED  
 % Volatile Solids Calculations  
 4 August 1989

| Sample | Cruc. wt | wt+sed | wet sed | 105wt+cr | dry sol | % sol | 400 <sup>0</sup> wt+cr | 400 <sup>0</sup> wt | wt v sol | % v sol |
|--------|----------|--------|---------|----------|---------|-------|------------------------|---------------------|----------|---------|
| A1     | 7.136    | 11.116 | 3.980   | 9.093    | 1.957   | 49.17 | 8.991                  | 1.855               | 0.102    | 5.21    |
| A1D    | 8.062    | 12.261 | 4.199   | 10.115   | 2.053   | 48.89 | 10.001                 | 1.939               | 0.114    | 5.55    |
| A2     | 7.341    | 11.656 | 4.315   | 9.467    | 2.126   | 49.27 | 9.352                  | 2.011               | 0.115    | 5.41    |
| A3     | 7.829    | 12.443 | 4.614   | 9.623    | 1.794   | 38.88 | 9.508                  | 1.679               | 0.115    | 6.41    |
| A4     | 7.589    | 11.826 | 4.237   | 9.576    | 1.987   | 46.90 | 9.454                  | 1.865               | 0.122    | 6.14    |
| A5     | 7.246    | 11.980 | 4.734   | 9.816    | 2.570   | 54.29 | 9.670                  | 2.424               | 0.146    | 5.68    |
| B1     | 7.263    | 12.648 | 5.385   | 10.910   | 3.647   | 67.73 | 10.808                 | 3.545               | 0.102    | 2.80    |
| B2     | 7.538    | 13.332 | 5.794   | 11.601   | 4.063   | 70.12 | 11.513                 | 3.975               | 0.088    | 2.17    |
| B3     | 6.756    | 8.867  | 2.111   | 7.692    | 0.936   | 44.34 | 7.647                  | 0.891               | 0.045    | 4.81    |
| B4     | 9.559    | 14.232 | 4.673   | 12.580   | 3.021   | 64.65 | 12.465                 | 2.906               | 0.115    | 3.81    |
| B5     | 19.713   | 26.968 | 7.255   | 24.362   | 4.649   | 64.08 | 24.238                 | 4.525               | 0.124    | 2.67    |
| C1     | 22.675   | 25.814 | 3.139   | 23.731   | 1.056   | 33.64 | 23.667                 | 0.992               | 0.064    | 6.06    |
| C2     | 22.877   | 31.315 | 8.438   | 28.240   | 5.363   | 63.56 | 28.071                 | 5.194               | 0.169    | 3.15    |
| C2D    | 6.960    | 12.259 | 5.299   | 10.333   | 3.373   | 63.65 | 10.233                 | 3.273               | 0.100    | 2.96    |
| C3     | 7.136    | 11.824 | 4.688   | 10.601   | 3.465   | 73.91 | 10.542                 | 3.406               | 0.059    | 1.70    |
| C4     | 8.062    | 13.599 | 5.537   | 11.928   | 3.866   | 69.82 | 11.862                 | 3.800               | 0.066    | 1.71    |
| C5     | 7.340    | 9.826  | 2.486   | 8.133    | 0.793   | 31.90 | 8.088                  | 0.748               | 0.045    | 5.67    |
| D1     | 7.828    | 11.316 | 3.488   | 10.308   | 2.480   | 71.10 | 10.258                 | 2.430               | 0.050    | 2.02    |
| D2     | 7.589    | 13.302 | 5.713   | 11.642   | 4.053   | 70.94 | 11.565                 | 3.976               | 0.077    | 1.90    |
| D3     | 7.246    | 13.566 | 6.320   | 11.794   | 4.548   | 71.96 | 11.719                 | 4.473               | 0.075    | 1.65    |
| D4     | 7.264    | 12.284 | 5.020   | 11.005   | 3.741   | 74.52 | 10.957                 | 3.693               | 0.048    | 1.28    |
| D5     | 7.538    | 11.985 | 4.447   | 10.687   | 3.149   | 70.81 | 10.634                 | 3.096               | 0.053    | 1.68    |
| E1     | 6.757    | 9.691  | 2.934   | 7.658    | 0.901   | 30.71 | 7.603                  | 0.846               | 0.055    | 6.10    |
| E2     | 9.561    | 14.113 | 4.552   | 10.938   | 1.377   | 30.25 | 10.856                 | 1.295               | 0.082    | 5.95    |
| E3     | 19.716   | 25.375 | 5.659   | 21.406   | 1.690   | 29.86 | 21.310                 | 1.594               | 0.096    | 5.68    |
| E4     | 22.676   | 29.353 | 6.677   | 24.752   | 2.076   | 31.09 | 24.636                 | 1.960               | 0.116    | 5.59    |
| E5     | 22.879   | 28.353 | 5.474   | 24.596   | 1.717   | 31.37 | 24.508                 | 1.629               | 0.088    | 5.13    |
| E5D    | 6.961    | 11.093 | 4.132   | 8.257    | 1.296   | 31.36 | 8.195                  | 1.234               | 0.062    | 4.78    |

Percent Volatile Solids obtained by weight loss by ignition at 400<sup>0</sup> C for 6h (Jaffe and Walters, 1977). Samples denoted with "D" are lab duplicates.

White Rock Creek Watershed  
1989 Sample Program

Organic Carbon by Walkley-Black Method

|       | begin | end  | diff | wet sed | %solid | net dry | % OC |
|-------|-------|------|------|---------|--------|---------|------|
| A1    | 34.5  | 40.3 | 5.8  | 4.12    | 49.17  | 2.03    | 0.75 |
| A1D   | 19.9  | 25.2 | 5.3  | 4.269   | 48.89  | 2.09    | 0.82 |
| A2    | 42.3  | 47.8 | 5.5  | 4.129   | 49.27  | 2.03    | 0.81 |
| A3    | 8.1   | 14.4 | 6.3  | 5.239   | 38.88  | 2.04    | 0.66 |
| A4    | 14.4  | 20   | 5.6  | 4.565   | 46.90  | 2.14    | 0.75 |
| A5    | 32.8  | 37   | 4.2  | 3.888   | 54.29  | 2.11    | 1.00 |
| B1    | 25.3  | 32.8 | 7.5  | 2.831   | 67.73  | 1.92    | 0.47 |
| B2    | 26.8  | 34.5 | 7.7  | 2.679   | 70.12  | 1.88    | 0.45 |
| B3    | 12.8  | 19.9 | 7.1  | 3.189   | 44.34  | 1.41    | 0.75 |
| B4    | 40.3  | 47.2 | 6.9  | 3.167   | 64.65  | 2.05    | 0.55 |
| B5    | 37    | 42.3 | 5.3  | 3.208   | 64.08  | 2.06    | 0.83 |
| C1    | 20    | 25.8 | 5.8  | 6.062   | 33.64  | 2.04    | 0.75 |
| C2    | 31.8  | 37.5 | 5.7  | 3.433   | 63.56  | 2.18    | 0.72 |
| C2D   | 25.8  | 31.8 | 6    | 3.234   | 63.65  | 2.06    | 0.71 |
| C3    | 38.5  | 46.9 | 8.4  | 2.711   | 73.91  | 2.00    | 0.29 |
| C4    | 19.5  | 27.6 | 8.1  | 3.175   | 69.82  | 2.22    | 0.31 |
| C5    | 27.6  | 33.4 | 5.8  | 6.017   | 31.90  | 1.92    | 0.80 |
| D1    | 41.1  | 48.6 | 7.5  | 2.926   | 71.10  | 2.08    | 0.44 |
| D2    | 33.4  | 41.1 | 7.7  | 2.812   | 70.94  | 1.99    | 0.42 |
| D3    | 15.4  | 23.6 | 8.2  | 2.819   | 71.96  | 2.03    | 0.32 |
| D4    | 12    | 19.5 | 7.5  | 2.721   | 74.52  | 2.03    | 0.45 |
| D5    | 7.8   | 15.4 | 7.6  | 3.202   | 70.81  | 2.27    | 0.39 |
| E1    | 23.6  | 28.4 | 4.8  | 6.435   | 30.71  | 1.98    | 0.96 |
| E2    | 28.4  | 34.7 | 6.3  | 6.48    | 30.25  | 1.96    | 0.69 |
| E3    | 25.2  | 31.1 | 5.9  | 6.399   | 29.86  | 1.91    | 0.78 |
| E4    | 34.7  | 40.5 | 5.8  | 6.245   | 31.09  | 1.94    | 0.79 |
| E5    | 40.5  | 45.9 | 5.4  | 6.396   | 31.37  | 2.01    | 0.83 |
| E5D   | 19.4  | 25.2 | 5.8  | 6.305   | 31.36  | 1.98    | 0.77 |
| Blank | 16.7  | 26.7 | 10.0 |         |        |         |      |

$$\%C = \frac{[(N1 * V1) - (N2 * V2)] * 0.364}{\text{wt. of oven dry soil}}$$

where: N1 = normality of potassium dichromate (= 1.0)  
 N2 = normality of ferrous sulfate (see below)  
 V1 = vol of potassium dichromate (= 10 ml)  
 V2 = volume of ferrous sulfate added during titration  
 wt = ca. 2 gm

N2 = N1 (V1 / V2)  
 = usually between 0.9 and 1.1

Calculated N2 = 1.0

White Rock Creek Watershed  
1989 SAMPLING PROGRAM  
PARTICLE SIZE

| Sample | wet sed | %solid | net sed | clay boat | dry+boat | clay wt | sand boat | dry+boat | sand wt | %clay | %silt | %sand |
|--------|---------|--------|---------|-----------|----------|---------|-----------|----------|---------|-------|-------|-------|
| A1     | 9.253   | 49.17  | 4.550   | 0.987     | 1.129    | 0.226   | 1.399     | 2.16     | 0.761   | 66.82 | 16.46 | 16.73 |
| A1D    | 9.567   | 48.89  | 4.678   | 1.39      | 1.62     | 0.23    | 1.404     | 2.11     | 0.706   | 68.41 | 16.49 | 15.09 |
| A2     | 10.221  | 49.27  | 5.036   | 1.414     | 1.619    | 0.205   | 0.984     | 2.064    | 1.08    | 43.69 | 34.87 | 21.45 |
| A3     | 12.819  | 38.88  | 4.984   | 1.438     | 1.655    | 0.217   | 0.985     | 1.103    | 0.118   | 53.77 | 43.86 | 2.37  |
| A4     | 11.15   | 46.90  | 5.229   | 1.426     | 1.641    | 0.215   | 1.403     | 2.035    | 0.632   | 49.72 | 38.19 | 12.09 |
| A5     | 9.209   | 54.29  | 4.999   | 1.398     | 1.594    | 0.196   | 0.982     | 2.18     | 1.198   | 36.80 | 39.23 | 23.96 |
| B1     | 9.364   | 67.73  | 6.342   | 1.402     | 1.578    | 0.176   | 0.989     | 4.709    | 3.72    | 16.40 | 24.94 | 58.66 |
| B2     | 8.317   | 70.12  | 5.832   | 0.984     | 1.154    | 0.17    | 1.411     | 5.287    | 3.876   | 13.72 | 19.82 | 66.46 |
| B3     | 9.477   | 44.34  | 4.202   | 1.402     | 1.577    | 0.175   | 1.426     | 4.132    | 2.706   | 23.80 | 11.80 | 64.40 |
| B4     | 9.282   | 64.65  | 6.001   | 1.41      | 1.584    | 0.174   | 1.417     | 5.019    | 3.602   | 16.00 | 23.97 | 60.03 |
| B5     | 10.187  | 64.08  | 6.528   | 1.428     | 1.599    | 0.171   | 1.385     | 5.611    | 4.226   | 12.87 | 22.39 | 64.74 |
| C1     | 12.922  | 33.64  | 4.347   | 1.406     | 1.611    | 0.205   | 1.405     | 1.511    | 0.106   | 50.61 | 46.95 | 2.44  |
| C2     | 9.75    | 63.56  | 6.197   | 1.426     | 1.616    | 0.19    | 1.427     | 3.685    | 2.258   | 25.82 | 37.74 | 36.44 |
| C2D    | 10.123  | 63.65  | 6.444   | 1.354     | 1.548    | 0.194   | 1.372     | 3.771    | 2.399   | 27.31 | 35.46 | 37.23 |
| C3     | 8.145   | 73.91  | 6.020   | 1.407     | 1.569    | 0.162   | 1.406     | 5.941    | 4.535   | 7.97  | 16.70 | 75.33 |
| C4     | 8.646   | 69.82  | 6.037   | 1.412     | 1.576    | 0.164   | 1.403     | 6.265    | 4.862   | 9.28  | 10.18 | 80.54 |
| C5     | 13.427  | 31.90  | 4.283   | 1.519     | 1.726    | 0.207   | 1.455     | 1.637    | 0.182   | 53.23 | 42.52 | 4.25  |
| D1     | 7.854   | 71.10  | 5.584   | 1.372     | 1.547    | 0.175   | 0.99      | 4.353    | 3.363   | 17.91 | 21.87 | 60.22 |
| D2     | 8.397   | 70.94  | 5.957   | 0.982     | 1.154    | 0.172   | 1.414     | 4.798    | 3.384   | 14.77 | 28.42 | 56.81 |
| D3     | 8.111   | 71.96  | 5.837   | 0.985     | 1.156    | 0.171   | 1.426     | 4.938    | 3.512   | 14.39 | 25.44 | 60.17 |
| D4     | 8.386   | 74.52  | 6.249   | 1.403     | 1.57     | 0.167   | 1.428     | 5.835    | 4.407   | 10.88 | 18.60 | 70.52 |
| D5     | 8.386   | 70.81  | 5.938   | 0.987     | 1.157    | 0.17    | 1.437     | 5.337    | 3.9     | 13.47 | 20.85 | 65.68 |
| E1     | 9.563   | 30.71  | 2.937   | 1.37      | 1.57     | 0.2     | 1.39      | 1.399    | 0.009   | 68.10 | 31.59 | 0.31  |
| E2     | 10.628  | 30.25  | 3.215   | 1.419     | 1.617    | 0.198   | 1.399     | 1.404    | 0.005   | 59.72 | 40.12 | 0.16  |
| E3     | 10.269  | 29.86  | 3.067   | 1.429     | 1.632    | 0.203   | 1.358     | 1.362    | 0.004   | 69.13 | 30.74 | 0.13  |
| E4     | 9.921   | 31.09  | 3.085   | 1.387     | 1.59     | 0.203   | 1.435     | 1.438    | 0.003   | 68.73 | 31.17 | 0.10  |
| E5     | 10.417  | 31.37  | 3.267   | 1.402     | 1.602    | 0.2     | 1.427     | 1.431    | 0.004   | 61.21 | 38.67 | 0.12  |
| E5D    | 10.98   | 31.36  | 3.444   | 1.407     | 1.614    | 0.207   | 0.984     | 0.991    | 0.007   | 66.20 | 33.59 | 0.20  |

## APPENDIX C

### STATISTICAL SUMMARIES

## AREA A

| N Obs | Variable | N | Minimum  | Maximum  | Mean     | Std Dev | CV    |
|-------|----------|---|----------|----------|----------|---------|-------|
| 5     | ZN       | 5 | 112.90   | 126.90   | 119.80   | 5.57    | 4.65  |
|       | PB       | 5 | 13.10    | 16.90    | 14.70    | 1.41    | 9.58  |
|       | CR       | 5 | 21.70    | 24.60    | 23.24    | 1.14    | 4.91  |
|       | CU       | 5 | 15.15    | 22.79    | 18.18    | 2.88    | 15.84 |
|       | CD       | 5 | 1.50     | 2.10     | 1.78     | 0.27    | 15.07 |
|       | FE       | 5 | 14116.00 | 17704.80 | 15747.60 | 1741.35 | 11.06 |
|       | MN       | 5 | 318.20   | 491.30   | 397.28   | 65.39   | 16.46 |
|       | VS       | 5 | 5.21     | 6.41     | 5.77     | 0.50    | 8.66  |
|       | CLAY     | 5 | 36.80    | 66.82    | 50.16    | 11.30   | 22.54 |
|       | SILT     | 5 | 16.46    | 43.86    | 34.52    | 10.60   | 30.70 |
|       | SAND     | 5 | 2.37     | 23.96    | 15.32    | 8.55    | 55.80 |
|       | OC       | 5 | 0.66     | 1.00     | 0.79     | 0.13    | 16.00 |

## AREA B

| N Obs | Variable | N | Minimum | Maximum  | Mean     | Std Dev | CV    |
|-------|----------|---|---------|----------|----------|---------|-------|
| 5     | ZN       | 5 | 77.00   | 110.90   | 95.56    | 13.01   | 13.61 |
|       | PB       | 5 | 12.20   | 25.20    | 16.40    | 5.32    | 32.41 |
|       | CR       | 5 | 11.60   | 19.80    | 15.00    | 3.19    | 21.29 |
|       | CU       | 5 | 11.70   | 24.24    | 15.27    | 5.09    | 33.35 |
|       | CD       | 5 | 1.70    | 5.00     | 2.54     | 1.39    | 54.60 |
|       | FE       | 5 | 8734.30 | 13051.80 | 10962.44 | 1642.64 | 14.98 |
|       | MN       | 5 | 324.00  | 406.60   | 356.06   | 40.10   | 11.26 |
|       | VS       | 5 | 2.17    | 4.81     | 3.25     | 1.06    | 32.44 |
|       | CLAY     | 5 | 12.87   | 23.80    | 16.56    | 4.31    | 26.05 |
|       | SILT     | 5 | 11.80   | 24.94    | 20.58    | 5.28    | 25.64 |
|       | SAND     | 5 | 58.66   | 66.46    | 62.86    | 3.34    | 5.31  |
|       | OC       | 5 | 0.45    | 0.83     | 0.61     | 0.17    | 28.01 |

## AREA C

| N Obs | Variable | N | Minimum  | Maximum  | Mean     | Std Dev | CV    |
|-------|----------|---|----------|----------|----------|---------|-------|
| 5     | ZN       | 5 | 87.70    | 157.60   | 118.82   | 34.79   | 29.28 |
|       | PB       | 5 | 16.50    | 29.50    | 23.06    | 5.99    | 25.97 |
|       | CR       | 5 | 11.20    | 25.40    | 18.34    | 6.74    | 36.74 |
|       | CU       | 5 | 12.57    | 28.50    | 18.74    | 6.79    | 36.22 |
|       | CD       | 5 | 2.40     | 3.60     | 2.70     | 0.51    | 18.88 |
|       | FE       | 5 | 12353.00 | 17485.30 | 14522.22 | 1923.54 | 13.24 |
|       | MN       | 5 | 361.20   | 570.60   | 473.94   | 103.85  | 21.91 |
|       | VS       | 5 | 1.70     | 6.06     | 3.66     | 2.10    | 57.51 |
|       | CLAY     | 5 | 7.97     | 53.23    | 29.38    | 21.76   | 74.07 |
|       | SILT     | 5 | 10.18    | 46.95    | 30.82    | 16.36   | 53.08 |
|       | SAND     | 5 | 2.44     | 80.54    | 39.80    | 37.39   | 93.95 |
|       | OC       | 5 | 0.29     | 0.80     | 0.57     | 0.25    | 43.88 |

## AREA D

| N Obs | Variable | N | Minimum | Maximum | Mean    | Std Dev | CV    |
|-------|----------|---|---------|---------|---------|---------|-------|
| 5     | ZN       | 5 | 76.20   | 114.80  | 91.86   | 14.13   | 15.38 |
|       | PB       | 5 | 19.40   | 29.80   | 23.34   | 4.11    | 17.59 |
|       | CR       | 5 | 10.70   | 13.60   | 12.24   | 1.09    | 8.89  |
|       | CU       | 5 | 11.71   | 31.94   | 16.49   | 8.65    | 52.45 |
|       | CD       | 5 | 2.10    | 2.40    | 2.28    | 0.13    | 5.72  |
|       | FE       | 5 | 7187.90 | 8971.10 | 8330.38 | 752.31  | 9.03  |
|       | MN       | 5 | 395.90  | 436.70  | 420.10  | 16.94   | 4.03  |
|       | VS       | 5 | 1.28    | 2.02    | 1.71    | 0.28    | 16.62 |
|       | CLAY     | 5 | 10.88   | 17.91   | 14.28   | 2.53    | 17.73 |
|       | SILT     | 5 | 18.60   | 28.42   | 23.04   | 3.89    | 16.89 |
|       | SAND     | 5 | 56.81   | 70.52   | 62.68   | 5.41    | 8.64  |
|       | OC       | 5 | 0.32    | 0.45    | 0.40    | 0.05    | 12.93 |

## AREA E

| N Obs | Variable | N | Minimum  | Maximum  | Mean     | Std Dev | CV    |
|-------|----------|---|----------|----------|----------|---------|-------|
| 5     | ZN       | 5 | 147.30   | 157.60   | 151.80   | 4.86    | 3.20  |
|       | PB       | 5 | 40.70    | 46.80    | 44.22    | 2.58    | 5.83  |
|       | CR       | 5 | 27.50    | 30.50    | 28.82    | 1.09    | 3.80  |
|       | CU       | 5 | 20.55    | 25.59    | 22.67    | 1.82    | 8.04  |
|       | CD       | 5 | 2.30     | 2.60     | 2.50     | 0.14    | 5.66  |
|       | FE       | 5 | 17862.80 | 18748.20 | 18299.42 | 364.06  | 1.99  |
|       | MN       | 5 | 843.00   | 1007.10  | 916.02   | 60.88   | 6.64  |
|       | VS       | 5 | 5.13     | 6.10     | 5.69     | 0.37    | 6.57  |
|       | CLAY     | 5 | 59.72    | 69.13    | 65.38    | 4.53    | 6.93  |
|       | SILT     | 5 | 30.74    | 40.12    | 34.46    | 4.55    | 13.19 |
|       | SAND     | 5 | 0.10     | 0.31     | 0.16     | 0.08    | 51.49 |
|       | OC       | 5 | 0.69     | 0.96     | 0.81     | 0.10    | 12.13 |

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