THE APPLICATION OF A SEQUENTIAL PARTIAL EXTRACTION PROCEDURE TO INVESTIGATE URANIUM, COPPER, ZINC, IRON AND MANGANESE PARTITIONING IN RECENT LAKE, STREAM AND BOG SEDIMENTS, NORTHERN SASKATCHEWAN

BY

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ABSTRACT

Sequential partial extractions show that partitioning of uranium, copper, zinc, iron and manganese into lake, stream and bog sediments are affected by the type and abundance of component fractions present in sediments and by the physico-chemical conditions of the superjacent waters. The water pH influences the concentration of uranium retained by organic matter as well as the relative proportion partitioned into the amorphous iron hydroxide fraction and the humic and fulvic acid components of the organic matter fraction. Copper partitioning is controlled by the percent carbon content of sediments which influences the concentration of metal retained in the organic matter fraction. The amount of copper retained by other component fractions is determined by their relative abundance in sediments. The Eh-pH conditions of the superjacent waters control the solubilities of iron, manganese and zinc thereby affecting the availability and sorption of these metals into the organic matter and inorganic hydroxide fractions of sediment. Metal partitioning characteristics and physico-chemical factors which influence metal partitioning should be considered when using lake, stream and bog sediments in geochemical exploration.

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CHAPTER 1

INTRODUCTION

1.1 General Statement

During the last decade, drainage system sediments have been used extensively for geochemical exploration purposes in Canada. Metal concentrations in the sediments has been utilized to assess the possibility of economic mineral deposits within each drainage system investigated (Allan, 1970; Arnold, 1970; Coker et al, 1974, 1975, 1977, 1979; Garrett and Hornbrook, 1975; Lehto, Arnold and Smith, 1976; Parslow, 1977, 1979; Sopuck, Lehto and Alley, 1980).

Sediments consist of a number of component fractions including organic matter, amorphous and crystalline hydroxides as well as carbonate and detrital minerals. Until recently a complete acid digestion has been used in geochemical exploration which only gave the total amount of each of the metals analyzed in the sediment sample. Information on the partitioning of specific metals within the various fractions of the sediment was not available by this method.

1.2 Purpose of the Present Study

The present study was initiated to examine aspects of uranium, copper, zinc, iron and manganese partitioning in drainage system sediments. Emphasis has been placed on determining metal concentrations in the organic matter, carbonate mineral, amorphous manganese hydroxide, amorphous iron hydroxide, crystalline iron hydroxide and detrital mineral fractions of lake, stream and bog sediments. Metal concentrations in the humic and fulvic acid components of sediment organic matter have also been determined in order to study the respective roles of organic acids in fixing metals.

1.3 Previous Development and Application of Partial and Sequential Partial Extraction Procedures

Partial extraction procedures had been developed by the early 1900's. By these techniques, individual component fractions of soil were extracted and examined in light of their physico-chemical properties. Soil organic matter was first extracted and examined by procedures described by Gortner, 1916; Oden, 1919; Eden, 1924; Troell, 1931; and Bremner and Lees, 1949 (Appendix I). Some of the first techniques to extract iron hydroxides were described by Robinson and Holmes, 1924; Drosdoff, 1935; Allison and Scarseth, 1942; Marshall and Jeffries, 1946; Deb, 1949; Schofield, 1950; and Aguilera and Jackson, 1953. Methods to extract manganese hydroxides and carbonate minerals were developed later and are described by Jackson, 1956; Goldberg and Arrhenius, 1958; and Chester and Hughes, 1967.

Sequential partial extraction methods have evolved out of partial extraction techniques. By these procedures, an aliquot of sample is successively treated with several partial extraction reagents to individually remove each of the component fractions of the sediment. Jackson (1956) first reported on the use of a sequential partial extraction technique to remove carbonate, organic matter and iron hydroxides from soil.

Some of the first applications of partial and sequential partial extraction techniques were concerned with partitioning of metal in iron and manganese hydroxide and carbonate component fractions of soil, marine and stream sediments (White, 1957; Goldberg and Arrhenius, 1958; LeRiche and Weir, 1963; McKeague and Day, 1966; Chester and Huges, 1967; and Gamble and Daniels, 1972). Recent studies have included the examination of metal partitioning in organic matter and silicate minerals as well as iron and manganese hydroxide and carbonate component fractions of soil, stream and fresh-water lake sediments (Baker, 1973; Gibbs, 1973; Chao and Anderson, 1974; Schaef, 1975; Chao, 1976; Grieve and Fletcher, 1976; Meineke and Klaysmat, 1976; Gatehouse et al, 1977; Gibb, 1977; Huang et al, 1977, 1978; Hoffman, 1978; Tessier, 1979; Bogle, 1980; and Sopuck Lehto and Alley, 1980; and Sopuck et al, 1980).

Recently data from sequential partial extractions have been used in exploration to aid in the interpretation of geochemical anomalies. For example, Dijkstra (1978) has used sequential partial extractions to distinguish between natural, and smelter-induced stream sediment anomalies in the northeast Pb-Zn mining district of Belgium. Hoffman and Fletcher (1978) in a similar manner has distinguished between source-related and source-unrelated anomalies in stream, soil and lake sediments in the Cordilleron region of Canada.

CHAPTER 2

DESCRIPTION OF THE STUDY AREA

2.1 General Description and Location

The study area lies approximately 170 km east-northeast of LaRonge, Saskatchewan and 90 km north-northwest of Flin Flon, Manitoba in the Maguire Lake region of east-central Saskatchewan (Figure 1).

This study is centered within a southward flowing drainage system comprising a headwater lake and two down-drainage lakes each connected by a series of streams and bogs (Figure 1). Each of the lakes, streams and bogs have been numerically labelled 1, 2, and 3 and are hereafter referred to as such.

Fixed wing, float-equipped aircraft from Pelican Narrows, Saskatchewan, located 17 km to the south-southeast provide access to the area.

2.2 Bedrock Geology

2.2.1 Regional Setting

Precambrian rocks of the Maguire Lake - Pelican Narrows region have been described as migmatite, biotite gneiss and granitoids of quartz diorite, granodiorite, quartz monzonite, granite and pegmatite composition (Satterly, 1932; Kirkland, 1956; Taylor, 1958; Kirkland, 1976; Sopuck, Lehto and Alley, 1980).

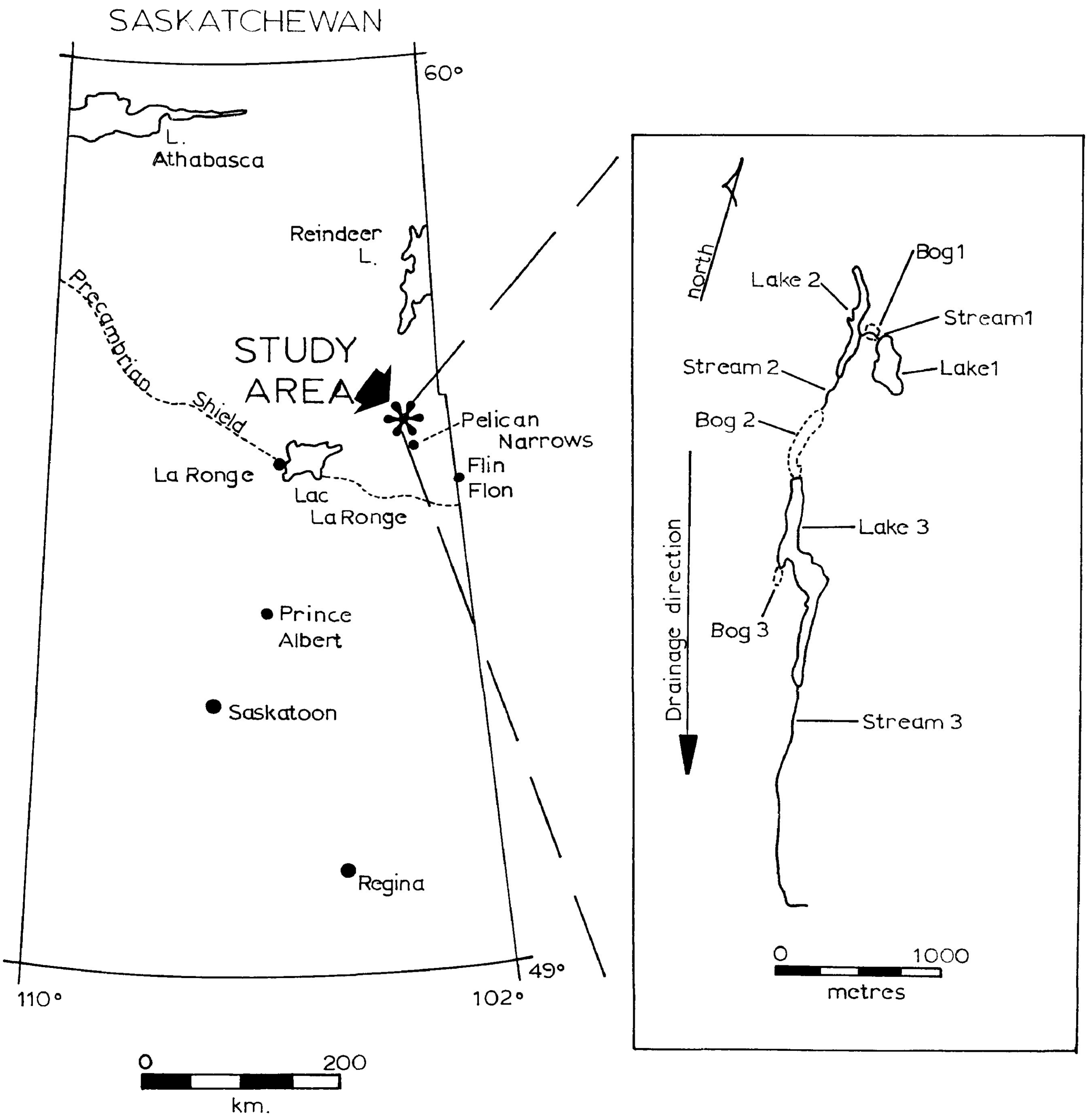

Figure 1 Location of the study area

2.2.2 Local Geology

The geology of the immediate study area has been described by Sopuck, Lehto and Alley (1980). In the area between Lakes 1 and 2, plagioclase-biotite-garnet pegmatite (P_1) and quartz-plagioclase-K feldspar-biotite-garnet pegmatite (P_2) intrudes biotite gneiss (Figure 2). The P_1 pegmatites in this area contain up to 0.24% U_3O_8 across 0.5 metres (Sopuck, Lehto and Alley, 1980). Uranium mineralization has been attributed to secondary alteration which appears as a yellow carnotite stain on surface exposures. Thin sections and autoradiographs indicate the presence of slightly radioactive monazite and zircon but no true uranium minerals have been identified Southward from Lakes 1 and 2, biotite gneiss and migmatite outcrop adjacent to the drainage basin. Mean metal values in "barren" biotite gneiss and pegmatite of the present study area and the Maguire Lake area in general are summarized in Table 1.

2.3 Regional Quaternary Geology and Glacial History

The Quaternary geology and glacial history of the Maguire Lake region has been interpreted by Schreiner, Alley and Christiansen, 1975; and Sopuck, Lehto and Alley, 1980. Glacial lacustrine silt, clay, sand and gravel and glaciofluvial sand and gravel are the dominant surficial deposits in the region. Striations and ice cast grooves record a dominant Wisconsin ice flow direction of S20°W. Deglaciation was followed by complete inundation of the area by Lake Agassiz until about 10,500 years B.P. (Elson, 1961).

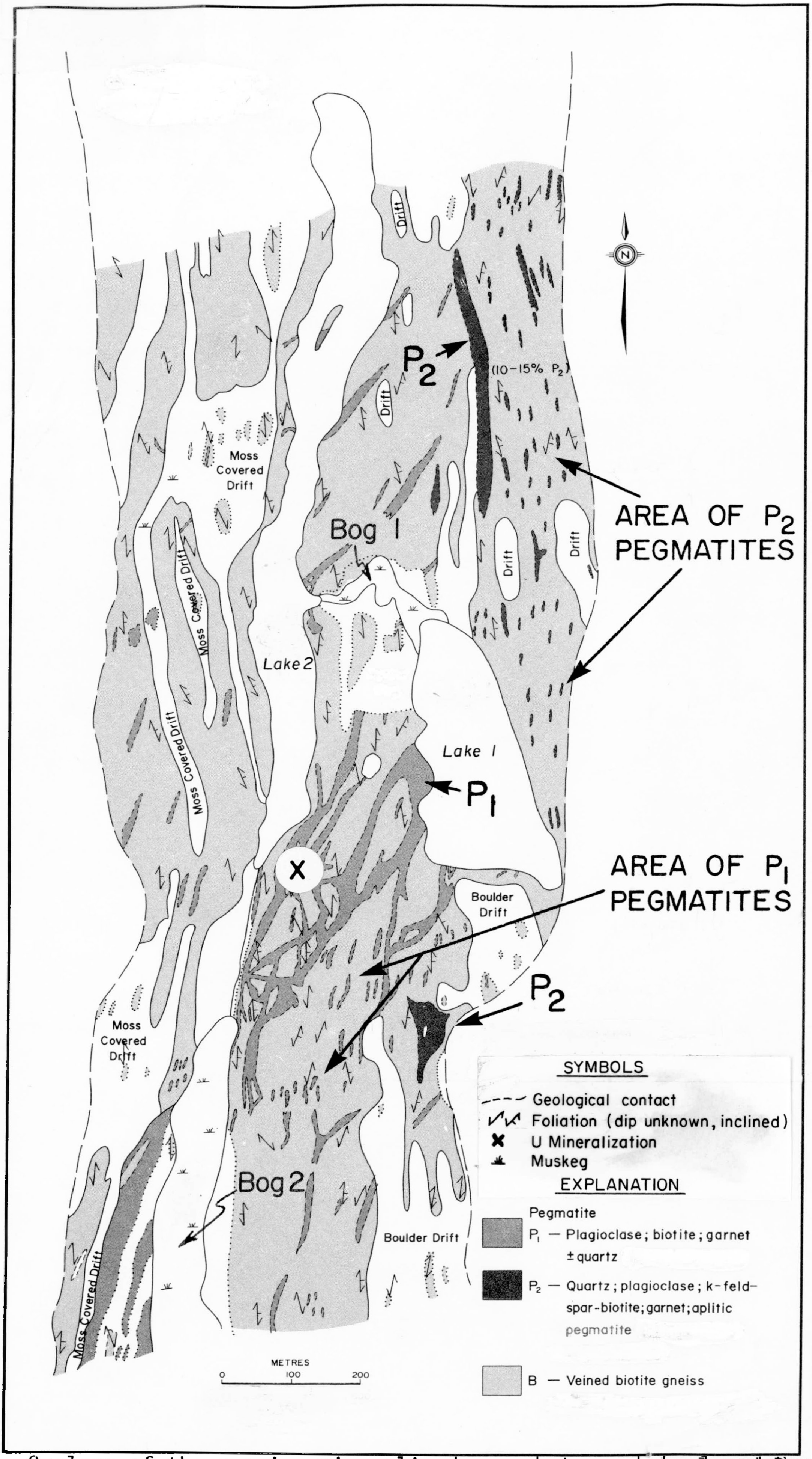

Figure 2 Geology of the uranium mineralized zone between Lake 1 and Bog 2 (after Sopuck, Lehto and Alley, 1980)

Bedrock Type	No. of Samples		Cu	Zn	Fe	Mn	U
Pelitic gneiss	68	Mean Value	25	80	3.01%	138	1
		Range	3-82	10-171	0.20-5.50%	15-439	D.L.-3
Pegmatite	121	Mean Value	7	26	0.06%	34	13
		Range	D.L.-53	4-129	D.L.-2.40%	3-183	D.L.-123

D.L. = detection limit

Table 1: Mean element concentrations (ppm) in "barren" bedrock of the present study area and the entire Maguire Lake area (modified after Sopuck, Lehto & Alley (1980).

2.4 Topography and Physiography

Topography of the study area is characterized by rugged, steep ridges of bedrock that extend parallel to and flank the drainage system. Maximum relief along the ridges is approximately 30 metres. Physiography of the drainage basin is dominated by bedrock and glacial deposits which account for approximately 40% and 30% of the land surface, respectively. Lakes cover 20% and bogs and streams the remaining 10% of the area.

2.5 Drainage

The Maguire Lake area is defined by a regional drainage divide which routes water either to the north or south. The present study area is located along the south-flowing flank of this divide. Local topography defines an elongate, trough-shaped drainage system which extends for approximately 3.75 km southward from its' headwaters. Lakes within the basin are typically elongate and the ratios of length to width for Lakes 1, 2 and 3 are 5:1, 12:1 and 9:1, respectively. The bogs along this drainage basin are low relief wetlands typical of the Precambrian Shield areas. Streams are narrow, never exceeding 1 metre in width and flow even during the late summer season.

2.6 Previous Reports of Uranium in the Area

The first report of uranium within the area was provided by Kirkland (1956) who documented the presence of radioactive pegmatites to the west of the present study. Interest in the area was renewed in 1975 following the release of data from the Geological Survey of Canada - Saskatchewan Survey Regional Reconnaissance Lake Sediment Survey. This survey indicated the presence of a 60 km^2 lake sediment anomaly centered about Maguire Lake 5.0 km to the east of the present study area. Investigations of this and other regional anomalies were completed by the Saskatchewan Research Council between 1975 and 1978 as part of a provincial program of resource evaluation (Lehto, Arnold and Smith, 1976, 1978).

CHAPTER 3

DESCRIPTION OF FIELD AND LABORATORY PROCEDURES

3.1 Sample Collection

Sampling of the lakes, streams and bogs following the standard methods utilized by the Geology Division, Saskatchewan Research Council was carried out during the first ten days of August 1978.

3.1.1 Lake Sediments

Thirty-three lake bottom sediment samples were collected from a twelve foot, motorized "Canova" craft using conventional Eckman dredge equipment (Maps 2-6). The sampling procedure consisted of lowering the loaded dredge along an attached line to the point of maximum penetration in bottom sediment. A brass weight 'messenger' was run along the line which activated the spring-loaded jaws. The dredge was retrieved onboard and the contained sediment was transferred to polyethylene bags, sealed and labelled.

3.1.2 Stream Sediments

Eighteen samples were collected from the three streams along the drainage system (Maps 2-6). Samples were retrieved by hand from the upper 0.5 metre thickness of sediment, transferred to polyethylene bags, sealed and labelled.

3.1.3 Bog Sediments

Seventeen samples of basal organic-rich sediment were collected from the local bogs (Maps 2-6). Sampling operations involve hand-augering through the surface moss and intermediate organic layers to basal peat-like accumulations. The lower basal sections of bogs are typically marked by a sharp contact with underlying glacial sediments. This contact is easily determined since the auger will not readily penetrate into the glacial sediments. Samples were transferred to polyethylene bags, sealed and labelled.

As soon as possible after collection, the lake, stream and bog sediment samples were refrigerated at 5°C. Samples were shipped to and temporarily stored at Saskatchewan Research Council facilities in Saskatoon, Saskatchewan prior to final shipment to Lakehead University, Thunder Bay, Ontario in May 1979. During all stages of transit the samples were refrigerated.

3.2 Investigations of Sediment Stratigraphy Through Lake and Bog Basins

The late Quaternary stratigraphy of the lake and bog basins was investigated with the aid of a portable overburden drill. The system used in this study was originally employed by the Finnish Geological Survey for regional and detailed drift sampling programs. The application of the system in Canada has been previously reported by Sopuck & Lehto (1978). The system consists of 1 metre length inter-connecting drill rods and flow-through drill bits of either 25 or 35mm diameter operated by a 28 kg gas-driven percussion hammer. The flow-through bit marks one of the many innovations of the system. Drilling is not hampered because of the internal wedge shape of the drill bit that allows material to continually pass through the nose and out the larger diameter side opening until the desired sampling media is intersected. When hammering ceases, sediment in the bit at that time remains wedged. Rods are retrieved with a mechanical jack and the sample extruded from the bit.

The application of the system to lake basin studies required that a drill platform be mounted between two braced canoes. Four anchors maintained the raft in a stationary position while drilling of at least seven sites per lake was carried out from the platform (Map 1). At each site, the entire thickness of organic sediment was penetrated in order to examine the nature of the underlying glacial sediment. Bog basin studies were restricted to Bog 2 where drilling operations were conducted from the ground (Map 1).

3.3 Investigation of Physico-Chemical Parameters

Physico-chemical parameter measurements including pH, oxidation reduction potential (Eh), conductivity, dissolved oxygen (DO) and temperature were taken of the superjacent water above the sediment-water interface at each lake, stream and bog sample stations. Observations were made in-situ with a Hydrolab Model 6-D water quality analyzer which was designed for field use and comprises a surface recording unit connected by cable to a submersible sonde which houses the five individual probes. The operation involved lowering the sonde to the desired depth and manually switching channels on the surface unit to display each individual parameter.

3.4 Extraction Procedures

As an initial step in processing, approximately 1.0 kg of lake, stream and bog sediment samples were oven-dried at approximately 40°C. Dried samples were disaggregated on a mechanical shaker and then homogenized by hand on an agate mortar and pestle in preparation for extraction and analytical procedures.

3.4.1 Sequental Partial Extraction

The following fractions were considered to be important in the metal partitioning with lake, stream and bog sediment samples:

1. organic matter.
2. carbonate and silicate minerals.
3. amorphous and crystalline iron and manganese hydroxides.

The choice of reagents to be used in the procedure has been based on the selectivity of each reagent in dissolving specific sediment fractions. Numerous alkaline solutions have been reported as effective reagents in the removal of sediment organic matter. The use of sodium hydroxide has been reported by Gortner, 1916; Oden, 1919; Eden, 1924; Troell, 1931; Schnitzer et al, 1958; and Kemp, 1964. Sodium hypobromite, chlorite and nitrate solutions have been used by Troell, 1971; Anderson, 1963; Lavkulich and Wiens, 1970; Schnitzer and Khan, 1972; Baker, 1973; Gibbs, 1973; Schaef, 1975; Hoffman, 1978; Bogle, 1980; Sopuck, Lehto and Alley, 1980; and Sopuck et al, 1980. Acidic solutions of hydrogen peroxide/hydrochloric acid, hydrogen peroxide/hydrofluoric acid and hydrogen peroxide/ascorbic acid have also been demonstrated as effective reagents in the dissolution of organic matter (Gortner, 1916; Eden, 1924; Lavkulich and Wiens, 1970; Presley, 1972; Meineke and Klaysmat, 1976; Huang et al, 1977; Huang and Liaw, 1978, 1979; and Tessier, 1979).

A comparative study of the relative effectiveness of sodium hypochlorite and hydrogen peroxide was carried out by Anderson (1963) and Lavkulich and Wiens (1970). They concluded thatsodium hypochlorite effectively extracts more organic matter with comparatively less destruction and removal of the carbonates, hydroxides and silicates than procedures employing hydrogen peroxide. The application of the sodium hypochlorite technique is based on its ability to oxidize and dissolve organic matter. Anderson (1963) reports that three consecutive treatments with sodium hypochlorite freshly adjusted to pH 9.5 is sufficient to remove the bulk of organic matter present in soils.

Carbonate minerals have been dissolved by sodium and ammonium acetate (Jackson, 1956; White, 1957; Zwarich and Mills, 1971; Gatehouse et al, 1977; Huang et al, 1977; Huang and Liaw, 1978; and Tessier, 1979) and with acetic, sulphuric and hydrochloric acids (Chester and Hughes, 1967, Zwarich and Mills, 1971; Presley et al, 1973; Hoffman, 1978; Sopuck, Lehto and Alley, 1980). Acetate solutions have been used primarily in conjunction with peroxide treatments whereas acidic solutions have been used insequence following hypochlorite treatment for removal of organic matter. Hoffman (1978) and Bogle (1980) report that the alkaline nature of hypochlorite may promote adsorption of metals onto clays or precipitation of metals as basic hydroxides. Subsequent treatment of the sample with acid would therefore dissolve these species as well as the carbonate minerals and associated metals.

In the event that specific examination of the metal concentration in the carbonate mineral fraction is desired, an alternative to the hypochlorite treatment, namely the acetate-peroxide treatment, may have to be sought.

Hydroxylamine hydrochloride, a weak reducing agent has been shown to be effective in dissolving amorphous manganese hydroxides in soils and sediments (Arrhenius and Korkish, 1959; Chester and Hughes, 1967; Chao, 1972; Presley, 1972; Chao and Anderson, 1974; Chao, 1976; Grieve and Fletcher, 1976; Meineke and Klaysmat, 1976; Gatehouse et al, 1977; Dijkstra et al, 1978; Hoffman, 1978; Tessier, 1979; and Sopuck, Lehto and Alley, 1980). The duration of the treatment is commonly limited to 30 minutes to ensure that only minor quantities of amorphous iron hydroxides are dissolved.

Iron hydroxides have been dissolved by sodium dithionite/sodium hydrosulphite (Deb, 1949; Aguilera and Jackson, 1953; Mitchell and McKenzie, 1954; Jackson, 1956; White, 1957; Mehra and Jackson, 1960; Coffin, 1963; Chao, 1976; Gibb, 1977; Huang et al, 1977; Dijkstra et al, 1978; Hoffman, 1978; Huang and Liaw, 1979). This method employs the sodium dithionite as a reducing agent in conjunction with sodium bicarbonate to act as a buffer solution and sodium citrate as the chelating or complexing agent for the dissolved ferric and ferrous iron. Inherent disadvantages of this method have been documented. Dithionite effectively dissolves both amorphous and crystalline iron hydroxide species thereby inhibiting separate examination of these two component fractions. Atomic adsorption spectrophotometric analysis of zinc in dithionite extracts is prohibited by extremely high positive interferences (Hoffman, 1978; Sopuck, Lehto and Alley, 1980). For these reasons, acid ammonium oxalate is the preferable reagent to dissolve amorphous iron hydroxides (Robinson and Holmes, 1924; Schofield, 1950; deEndredy, 1963; McKeague and Day, 1966; Gamble and Daniels, 1972; Huang et al, 1977; and Hoffman, 1978). Acid ammonium oxalate is also a reducing agent and thereby complexes metal ions and keeps them in solution. The extraction is commonly conducted in darkness due to the photo-sensitive nature of the reaction (Schwertmann, 1964; McKeague and Day, 1966; and Huang et al, 1977). Crystalline iron hydroxides are dissolved by hydrazine chloride which acts to reduce iron species (Gatehouse et al, 1977; Sopuck, Lehto and Alley, 1980).

Detrital minerals are commonly dissolved either by alkaline fusion or mixtures of strong acids. The acid method is preferable as the fusion method requires a large excess of fusing salt to complete the dissolution which can contribute to instability and high background readings in atomic adsorption spectrophotometry (Tessier, 1979).

The sequential partial extractions must follow an order whereby each successive chemical treatment involves a stronger reagent than the previous one. In view of the rich organic nature of the sediments being presently examined, early removal of organic matter was considered important in choosing the sequence of partial extractions. Carbonate minerals were not expected to be in abundance due to a lack of evidence of a local carbonate source. Consequently specific examination of this fraction was not necessary and sodium hypochlorite was considered to be a suitable reagent to remove organic matter.

The method adopted for the present study was as follows:

1. treatment with sodium hypochlorite to dissolve organic matter and associated metal;
2. treatment with water-hydrochloric acid solution to dissolve carbonate minerals, clay minerals and basic hydroxides and associated metal;
3. treatment with hydroxylamine hydrochloride to dissolve amorphous manganese hydroxides and associated metal;
4. treatment with acid ammonium oxalate to dissolve amorphous iron hydroxides and associated metal;
5. treatment with hydrazine chloride to dissolve crystalline iron hydroxides and associated metal;
6. treatment with hydrofluoric-nitric-perchloric acid to dissolve residual detrital minerals and associated metal.

The sequential partial extraction procedure modified from Gatehouse et al, 1977 and Hoffman, 1978 is described in detail in Appendix II(I). Extractions of sediment samples (1.000 g dry weight) were conducted in 50.0 ml nalgene centrifuge tubes. Each of extractions 1 through 5 were repeated three times in succession with 20.0 ml of reagent. Solution extracts were separated from the sample residue after each step by spinning the samples at 2400 rpm for 10 minutes in a Sorvall Model RC2-B Automatic Refrigerated Superspeed Centrifuge. Solutions were decanted into 100.0 ml Pyrex volumetric flasks and the sample residue was left in the centrifuge tubes. After the third step was carried out for each treatment, the sample residue was washed with 10 ml of deionized water to remove any reagent remaining before implementing the next extraction procedure. Solution extracts were topped up to a final volume of 100.0 ml with deionized water. The final step of sequential partial extractions was dissolution of the detrital mineral fraction which was performed by subjecting the sample residue from extraction #5 to a single treatment with concentrated hydrofluoric-nitric-perchloric acids as described in Appendix II(II). Solutions from this digestion were transferred to Pyrex volumetric flasks and brought up to a final volume of 100.0 ml with deionized water in preparation for analysis.

3.4.2 Partial Extractions

In the present study, partial extraction techniques have been utilized to separate the humic and fulvic acid components of sediment organic matter (Oden, 1919; Eden, 1924; Kemp, 1969; Schnitzer and Khan, 1972; Baker, 1973; and Schaef, 1975). Humic and fulvic acids are high molecular weight compounds that represent the dominant constituents of humic material (Manskaya and Drozdova, 1968). They consist of aromatic nuclei and nitrogen based groups in cyclic form or as peripheral chains. The presence of side-chain groups such as phenolic and alcoholic hydroxyls, OH; carboxyls, COOH; carbonyls, CO; and methoxyls, OCH, have also been documented. The capacity of humic and fulvic acids to retain metals has been attributed to the ion exchange and sorption capacity of these functional groups.

The separation of these compounds has been based on their relative solubilities in alkaline and acidic media. Reagents generally used include sodium hydroxide, sodium hyponitrate and hydrochloric acid. Humic acids are dark-coloured organic compounds that are soluble in alkaline solutions and precipitate on addition of acid to the alkaline extracts. Light straw to tea-coloured organic compounds that are soluble in both alkaline and acidic media consitute the fulvic acid component. The humin fraction of sediments includes hydroxide and detrital mineral species which are insoluble in both acid and alkaline medium. Humic-fulvic acid and humin extractions were performed in the present study with sodium hyponitrate - hydrochloric acid according to methods described by Schnitzer and Khan (1972) and Schaef (1975). Details of the procedure are provided in Appendix II(II). The extractions were performed in 250 ml centrifuge tubes initially containing 10.0000 g dry weight of sample and were repeated three times in succession on each sample. After separation, humic and fulvic residues were dried at 40°C and subjected to total acid digestion procedures in preparation for analysis (Appendix II(I)).

3.5 Analytical Techniques

The concentrations of copper, zinc, iron, and manganese were determined by standard atomic adsorption spectrophotometric methods using Perkin Elmer Model 303, and Model 2380 instruments. The use of two instruments for the present study was necessitated by the replacement of the old Model 303 instrument with a newer model. This decision to replace the spectrophotometer was out of the hands of the researcher and resulted in approximately 80% of the analyses being carried out on Model 303 and the remaining 20% on Model 2380. Features of each instrument and details of operating procedures are provided in Appendix III(I).

Uranium concentrations in sediment samples were determined by fluorimetric analysis using a Jarrell Ash Fluorimeter. Features of the instrument and operating procedures are provided in Appendix III(II).

Determination of percent carbon concentrations in sediment samples were performed on a Perkin Elmer Model 240 Elemental analyzer. Details of the operation and features of the instrument are provided in Appendix III(III).

CHAPTER 4

RESULTS OF FIELD AND ANALYTICAL PROCEDURES

4.1 Field Procedures

4.1.1 Lake and Bog Basin Stratigraphy

The late Quaternary stratigraphy of the lake and bog basins is shown in Map 1. Four traverses spaced at 100 m intervals were completed across Lake 1. Along each traverse at least one centre basin and two nearshore drill sites were chosen. Traverses were completed only on Lake 1 as Lakes 2 and 3 were too narrow. Drilling has shown that the thickest sequence of post-glacial organic sediment (6.5 m) occurs in the centre of Lake 1 and thins to an average of about 3 metres toward the shore. In Lake 2, the organic sediments range in thickness from 2 metres in the south to 5 metres in the central regions and the northern end of the lake. Organic sediments in Lake 3 varied in thickness from 1.5 to 4.5 metres.

At most lake drill locations the organic sediments are immediately underlain by glaciolacustrine clays or mixtures of clay and sand. The thickness of penetration through clay and sand ranges from 2 to 10 metres.

Four traverses spaced at 90 m intervals were completed along the length of Bog 2 (Map 1). The organic sediment in this bog ranges up to 6 metres in thickness and is observed to be underlain by glaciolacustrine clay and clay-sand deposits up to 8 metres in thickness.

4.1.2 Physico-Chemical Parameters and Percent Carbon Concentrations in the Lake, Stream and Bog Environment

Physico-chemical parameter values including pH, oxidation reduction potential (Eh), conductivity, dissolved oxygen, and temperature of the superjacent water and carbon concentrations for each sediment sample location are listed in Appendix VI, Table 19.

The pH values of the superjacent waters in the study area are generally in the acidic to alkaline range (pH 4.7 - 7.6; Appendix VI, Table 19). Mean values of 6.5 and 6.2 are recorded for local lakes and streams respectively. Bog waters are more acidic as illustrated by a mean pH value of 5.5 (Table 2). The oxidation reduction potentials are recorded in millivolts and show that oxidizing conditions prevail (Eh $^{+}$485 - $^{+}$700 millivolts, Appendix VI, Table 19). The mean Eh values for lakes and streams is 603 and 607 millivolts, respectively. The Eh conditions in bogs are less oxidizing than in lakes and streams according to a lower mean value of 545 millivolts (Table 2). Conductivity is a measure of ionic concentration in water and is recorded in micromhos/cm. In the present study, conductivity is highest in bog waters according to a mean value of 112 micromhos/cm. The mean conductivity for lakes and streams are 108 micromhos/cm and 104 micromhos/cm, respectively (Table 2). Dissolved oxygen is a measure of the oxygenation level in waters and is reported in ppm. The mean dissolved oxygen values in lakes and streams are 8.4 ppm and 6.5 ppm, respectively. Bog waters which have a mean value of 3.2 ppm dissolved oxygen are less oxygenated than lakes and streams (Table 2). Temperature values are reported in °C and for local stream

Environment Type		Physico-Chemical Parameters (measured in water adjacent to sediment water interface)					% C Analyzed in Sediment
		pH	Oxidation reduction potential (+millivolts)	Conductivity (micromhos/cm)	Dissolved Oxygen (ppm)	Temp. °C	
Lake 1	$\bar{x}$	7.0	561	103	9.7	16.4	30.3
n = 11	c.v.	3	8	7	2	4	23
Lake 2	$\bar{x}$	6.0	664	86	8.6	14.4	18.1
n = 7	c.v.	3	4	9	2	6	11
Lake 3	$\bar{x}$	6.3	605	121	7.3	12.9	23.2
n = 15	c.v.	3	19	26	3	28	28
All lakes	$\bar{x}$	6.5	603	108	8.4	14.4	24.5
n = 33	c.v.	7	15	24	13	21	31
Stream 1	$\bar{x}$	6.4	620	110	3.5	10.5	2.7
n = 1	c.v.						
Stream 2	$\bar{x}$	6.5	603	90	5.6	12.3	21.9
n = 6	c.v.	10	9	0	13	7	38
Stream 3	$\bar{x}$	6.1	608	111	7.3	15.7	10.4
n = 11	c.v.	2	3	13	18	22	68
All Streams	$\bar{x}$	6.2	607	104	6.5	14.8	14.0
n = 18	c.v.	7	6	14	23	18	68
Bog 1	$\bar{x}$	6.4	580	105	15	12.1	45.9
n = 4	c.v.	4	8	5	51	28	3
Bog 2	$\bar{x}$	5.1	529	116	2.3	11.9	41.4
n = 9	c.v.	4	5	14	39	8	10
Bog 3	$\bar{x}$						25.2
n = 4	c.v.						7
All Bogs	$\bar{x}$	5.5	545	112	3.2	11.9	37.5
n = 13	c.v.	12	7	13	63	17	9

$\bar{x}$ - mean

c.v. - coefficient of variation about mean value expressed as standard deviation/mean value x 100%

Table 2 Summary of physico-chemical parameters in superjacent waters and percent carbon concentrations in sediments of the study area and lake waters mean values are 14.8°C and 14.4°C, respectively. Bog waters are typically colder than lake and stream waters according to a mean value of 11.9°C (Table 2). The total carbon concentrations of sediment samples reflect their organic matter content (Coker, 1974). The highest total carbon concentrations occur in bogs ($\bar{x}$ = 37.5%). Local lake sediments contain a mean value of 24.5%C and stream sediments a mean value of 14.8%C (Table 2).

4.2 Analytical Procedures

4.2.1 Comparison of Data Generated by Model 303 and Model 2380 Atomic Adsorption Spectrophotometers

The Mann Whitney statistical test has been used to determine whether or not analytical data derived from Model 303 and Model 2380 Atomic Adsorption Spectrophotometers are comparable (Mann and Whitney, 1947; Kreyszig, 1970). The test which is described in Appendix IV has been applied to data from a series of replicate samples. The replicate suite comprises three samples, one each from local lake, stream and bog environments which have been separately subjected to extractions and analyzed ten times for copper, zinc, iron and manganese over the course of the study (Appendix V, Tables 14 to 18). For each replicate sample, two analyses were performed on the Model 2380 and eight on the Model 303. Results of the Mann Whitney test indicate that at the 95% confidence interval there is no significant statistical difference in metal values generated from either instrument (Table 3). The analytical data for the entire study are therefore considered to be comparable.

		CALCULATED W VALUES IN EXTRACTIONS							
		TOTAL DIGESTION	SEQUENTIAL PARTIAL EXTRACTIONS						
SEDIMENT TYPE	ELEMENT ANALYZED	TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI H_2O HYDROCHLORIC ACID (CARBONATES) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES (AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES (AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES (XTALLINE)#5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Lake	Cu	5*	4	14.5	15	6.5	6.5	9	12
	Zn	15.5	13	13.5	9	5.5	12.5	15	11.5
	Fe	13	16.5	12.5	5	14	6.5	17	15
	Mn	11	8.5	13	13	9.5	10	4.5	7
Stream	Cu	4	10.5	10	4	12	6.5	4.5	7.5
	Zn	13	11	10	3.5	4	12.5	14.5	9.5
	Fe	4	11	13.5	15.5	5.5	6	14	7
	Mn	13	11	13	4	4	13	18	14.5
Bog	Cu	15	10	10	8.5	12.5	9	13.5	7.5
	Zn	12	16	13	5	4	14.5	15	8
	Fe	14	11	10.5	14	6	15.5	8.5	16.5
	Mn	12	12	9	17	11.5	10	8	8.5

*W is a sum of the rank score from the Model 2380 Atomic Adsorption spectrophotometer data. The reader is referred to Appendix IV(I) for details concerning the computation of W. Values between 3 and 19 indicate no statistical difference between element values determined on Model 303 and Model 2380 Atomic Adsorption spectrophotometers. The confidence interval is 95%.

Table 3 Sum of rank score values for copper, zinc, iron and manganese from the Model 2380 Atomic Adsorption Spectrophotometer

4.2.2 Accuracy and Precision of Atomic Adsorption and Fluorimetric Analysis

The analytical accuracy and precision of atomic adsorption spectrophotometric and fluorimetric analyses have been determined by a series of standard samples and a suite of replicate analyses, respectively. Details of procedures used in the present study are provided in Appendix III(IV). Accuracy is considered to be a measure of the ability of a procedure to reflect the true concentration of an element and in the present study has been judged by comparing metal values determined from routine analysis to the established value of standard samples. A quantitative measure of accuracy has been provided by calculating the coefficient of variation about the established value (Table 4). At the upper limit of metal concentration established as 100 ppm copper, 240 ppm zinc, 5.9% iron, 600 ppm manganese and 36 ppm uranium, accuracy is $\leq$ 10% for atomic adsorption spectrophotometric and fluorimetric analyses. At concentration values approximately one tenth the upper limit, accuracy is $\leq$ 20%.

Analytical precision refers to the limits within which analyses are reproducible. A quantitative measure of precision has been provided by calculating the coefficient of variation about a mean metal value determined from replicate analysis of a selected sample. In the present study, three replicate samples have been selected, one each from lake, stream and bog environments. Each sample has been separately subjected to sequential partial extractions ten times over the course of the study and analysed for uranium, copper, zinc, iron and manganese (Appendix V, Tables 14-18). Precision of analysis for

Element Analyzed	Established Value of Standard Sample	Observed Values in Standard Sample			
		N	$\bar{x}$	σ	c.v.(%)
Cu (ppm)	100	42	98	5.8	6
	10	36	9	1.5	15
Zn (ppm)	240	29	239	11.9	5
	20	29	22	3.9	20
Fe (%)	5.00	44	5.21	.44	9
	.50	38	.60	.09	15
Mn (ppm)	600	39	597	11.7	2
	50	35	54	6.5	13
U (ppm)	36	43	35	3.3	9

N = no. of observations

$\bar{x}$ = arithmetic mean

σ = standard deviation

cv = coefficient of variation about absolute value expressed as σ/absolute value x 100% (this value is interpreted as a quantitative measure of analytical accuracy)

Table 4 Accuracy of analysis for copper, zinc, iron, manganese and uranium each replicate lake, stream and bog sample is summarized in Tables 5, 6 and 7, respectively. Precision of the entire combined sequential partial extraction procedure has been determined by computing the coefficient of variation for the summed value of all six extractions. The value was determined to be less than 11% in each of lake, stream and bog sediments (Tables 5,6,7). For each of the sequential extractions, the coefficient of variation was observed to vary according to the proportion of total metal extracted and the results can be summarized as follows:

(a) $\leq$ 12% for extractions containing more than or equal to 30% of total metal in the sample.

(b) from 12 to 20% for extractions containing 10 to 30% of the total metal in the sample.

(c) from 20 to 30% for extractions containing 5 to 10% of the total metal in the sample.

(d) greater than 30% for extractions containing less than 5% of the total metal in the sample.

4.2.3 Reliability of the Sequential Partial Extraction Technique

The reliability of the sequential partial extraction technique has been determined by testing the comparability between metal values computed as a sum of all extractions and metal values reported from a total digestion of the same sample. The substitute t test described in Appendix IV has been used for this purpose (Miller and Kahn, 1962; Dixon and Massey, 1969). The test is based on the premise that the sum of partial extractions should equal the total metal concentration

SEDIMENT TYPE (DUPLICATE ANALYSIS)	ELEMENT ANALYZED	OBSERVATIONS	EXTRACTION TYPE							
			TOTAL		SEQUENTIAL PARTIAL					
			TOTAL DIGESTION IN HYDROFLUORIC-NITRIC PERCHLORIC ACIDS	SUM OF PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORATE (ORGANIC MATTER)#1	D.I.H2O-HYDROCHLORIC ACID (CARBONATES)#2	HYDROXYLAMINE HYDRO-CHLORIDE (MN HYDROXIDES)#3 (AMORPHOUS)	ACID AMMONIUM OXALATE (FE HYDROXIDES (AMORPHOUS)) #4	HYDRAZINE CHLORIDE (FE HYDROXIDE (XTALLINE)) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Lake Sediments n=10	U (ppm)	x̄	a. 100 b. 173	a. 100 b. 167	a. 83 b.138	a. 2 b. 4	a.< 1 b. 1	a. 14 b. 23	a.< 1 b.< DL	a. < 1 b. 1
			8.90	7.31	8.58	2.33	0.40	2.77		0.45
			5	4	6	58	40	12		45
	Cu (ppm)		a. 100 b. 88	a. 100 b. 88	a. 57 b. 50	a. 2 b. 2	a. 3 b. 3	a. 30 b. 26	a. 6 b. 5	a. 2 b. 2
			3.63	4.76	3.00	0.63	1.11	3.27	1.14	0.81
		cv	4	5	6	32	37	13	23	40
	Zn (ppm)	x̄	a. 100 b. 249	a. 100 b. 254	a. 20 b. 49	a.<1 b.<1	a. 13 b. 31	a. 54 b.138	a. 6 b. 14	a. 7 b. 18
			14.89	9.86	6.84	0.80	8.53	13.06	5.46	3.92
		cv	6	4	14	> 100	27	9	39	22
	Fe (%)	x̄	a. 100 b. 1.66	a. 100 b. 1.65	a. 5 b. .08	a.<1 b.<Dl	a. 2 b. .04	a. 50 b. .82	a. 13 b. .22	a. 30 b. 49
			0.06	0.06	.02		.02	.07	.03	.07
		cv	4	3	25		50	9	14	14
	Mn (ppm)	x̄	a. 100 b. 269	a. 100 b. 271	a. 39 b.107	a.< 1 b. 1	a. 22 b. 59	a. 20 b. 54	a. 5 b. 11	a. 14 b. 39
			6.29	5.64	11.28	0.80	7.42	4.24	2.00	2.89
		cv	2	2	11	80	13	8	18	7

n = no. of replicate analysis
x̄ = arithmetic mean a. expressed as a percentage of total sum of partial extraction.
b. in concentration units of ppm unless otherwise stated.
σ = standard deviation
cv = coefficient of variation about mean expressed as σ/x̄ x 100 (This value is interpreted as a quantitative measure of analytical precision).

Table 5 Precision of Analysis for uranium, copper, zinc, iron and manganese in total and sequential partial extractions of lake sediments

SEDIMENT TYPE (REPLICATE ANALYSIS)	ELEMENT ANALYZED	OBSERVATIONS	EXTRACTION TYPE TOTAL TOTAL DIGESTION (IN HYDROFLUORIC-NITRIC-PERCHLORIC ACIDS	SUM OF PARTIAL EXTRACTIONS 1+2+3+4+5+6	SEQUENTIAL PARTIAL SODIUM HYPOCHLORATE (ORGANIC MATTER)#1	D.I.H2O-HYDROCHLORIC ACID (CARBONATES)#2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES) (AMORPHOUS))#3	ACID AMMONIUM OXALATE (FE HYDROXIDES (AMORPHOUS)) #4	HYDRAZINE CHLORIDE (FE HYDROXIDE (XTALLINE)) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Stream Sediments n=10	U (ppm)	x̄	a. 100 b. 43	a. 100 b. 41	a. 83 b. 34	a. 2 b. 1	a. <1 b. <DL	a. 14 b. 6	a. <1 b. <DL	a. 2 b. 1
			3.69	3.38	3.17	0.49		0.92		0.40
		cv	9	8	9	49		15		40
	Cu (ppm)	x̄	a. 100 b. 48	a. 100 b. 46	a. 28 b. 13	a. 2 b. 1	a. 4 b. 2	a. 33 b. 15	a. 11 b. 5	a. 22 b. 10
			2.97	2.71	1.22	1	0.78	0.94	1.11	2.60
		cv	6	6	9	100	39	6	22	26
	Zn (ppm)	x̄	a. 100 b. 95	a. 100 b. 94	a. 10 b. 9	a. 1 b. 1	a. 1 b. 1	a. 36 b. 34	a. 6 b. 6	a. 47 b. 44
			3.32	4.13	2.87	0.67	0.81	4.67	1.50	3.04
			3	4	32	67	81	14	25	7
	Fe		a. 100 b. 4.59	a. 100 b. 4.60	a. 2 b. 0.09	a. 3 b. 0.15	a. 2 b. 0.07	a. 36 b. 1.68	a. 4 b. 0.19	a. 54 b. 2.48
			0.12	0.14	0.02	0.03	0.02	0.12	0.03	0.19
			3	3	22	20	29	7	16	8
	Mn (ppm)	x̄	a. 100 b. 568	a. 100 b. 564	a. 15 b. 83	a. <1 b. <1	a. 13 b. 73	a. 19 b. 108	a. 5 b. 27	a. 48 b. 273
			10.86	10.71	11.61	0.66	6.88	6.50	4.45	13.73
		cv	2	2	14	>100	9	6	16	5

n = no. of replicate analysis
x̄ = arithmetic mean a. expressed as a percentage of sum total of partial extractions
b. in concentration units of ppm unless otherwise stated
σ = standard deviation
cv = coefficient of variation about mean expressed as σ/x̄ x 100 (This value is interpreted as a quantitative measure of analytical precision).

Table 6 Precision of analysis for uranium, copper, zinc, iron and manganese in total and sequential partial extractions of stream sediments

			EXTRACTION TYPE							
			TOTAL	SEQUENTIAL PARTIAL						
SEDIMENT TYPE (REPLICATE ANALYSES)	ELEMENT ANALYZED	CALCULATIONS	TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACIDS	SUM OF PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORATE (ORGANIC MATTER) #1	D.I. H2O-HYDROCHLORIC ACID (CARBONATES) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDE XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Bog Sediments n=10	U (ppm)	$\bar{x}$	a. 100 b. 172	a. 100 b. 170	a. 99 b.169	a. 1 b. 1	a.< 1 b.<DL	a.< 1 b. 1	a.< 1 b. 1	a.< 1 b.< 1
			10.56	9.09	8.75	1		1	0.40	0.63
			6	5	5	100		100	40	> 100
	Cu (ppm)		a. 100 b. 52	a. 100 b. 54	a. 78 b. 42	a.< 1 b.< 1	a. 4 b. 2	a. 7 b. 4	a. 6 b. 3	a. 7 b. 4
			3.74	3.31	2.83	0.48	0.66	0.70	0.81	0.49
		cv	7	6	7	> 100	33	18	27	12
	Zn (ppm)	$\bar{x}$	a. 100 b. 30	a. 100 b. 32	a. 53 b. 17	a.< 1 b.< 1	a. 9 b. 3	a. 13 b. 4	a. 13 b. 4	a. 9 b. 3
			3.27	3.53	3.98	0.3	1.25	1.56	1.51	1.22
			11	11	23	> 100	42	39	38	41
	Fe ( )		a. 100 b. 0.50	a. 100 b. 0.52	a. 48 b.0.25	a. 6 b.0.03	a. 8 b.0.04	a. 19 b.0.10	a. 12 b.0.06	a. 8 b. 0.04
			0.05	0.04	0.03	0.01	0.01	0.02	0.02	0.01
			10	8	12	33	25	20	33	25
	Mn (ppm)	$\bar{x}$	a. 100 b. 57	a. 100 b. 55	a. 67 b. 37	a. 2 b. 1	a. 4 b. 2	a. 5 b. 3	a. 7 b. 4	a. 15 b. 8
			3.29	3.84	4.06	1.22	1.14	1.17	1.35	1.45
		cv	6	7	11	> 100	57	39	34	18

n = no. of replicate analysis
$\bar{x}$ = arithmetic mean
σ = standard deviation
cv = coefficient of variation about mean expressed as $\sigma/\bar{x}$ x 100 (This value is interpreted as a quantitative measure of analytical precision).

Table 7 Precision of Analysis for uranium, copper, zinc, iron and manganese in total and sequential partial extractions of bog sediments of the sample. T ratios have been determined for uranium, copper, zinc, iron and manganese in each of lake, stream and bog sediments from the replicate suite of samples previously described. Results are listed in Table 8, and indicate that at the 95% confidence interval there is no statistical difference between mean values determined from the sum of sequential partial extractions and mean values determined from total digestion. The sequential partial extraction technique is therefore considered to be a reliable method.

ELEMENT ANALYZED	OBSERVATIONS n = 10	Lake Sediments		Stream Sediments		Bog Sediments	
		TOTAL DIGESTION	SUM OF PARTIAL EXTRACTIONS	TOTAL DIGESTION	SUM OF PARTIAL EXTRACTIONS	TOTAL DIGESTION	SUM OF PARTIAL EXTRACTIONS
U (ppm)	$\bar{x}$	(1) 173	(2) 167	(1) 43	(2) 41	(1) 172	(2) 170
	w	(1) 30	(2) 28	(1) 12	(2) 11	(1) 32	(2) 27
	τd	.207*		.167		.068	
Cu (ppm)	$\bar{x}$	(1) 88	(2) 88	(1) 48	(2) 46	(1) 52	(2) 54
	w	(1) 14	(2) 16	(1) 9	(2) 9	(1) 11	(2) 11
	τd	.000		.222		-.182	
Zn (ppm)	$\bar{x}$	(1) 249	(2) 254	(1) 95	(2) 94	(1) 30	(2) 32
	w	(1) 42	(2) 29	(1) 11	(2) 11	(1) 11	(2) 13
	τd	-.141		.091		-.166	
Fe (%)	$\bar{x}$	(1) 1.66	(2) 1.65	(1) 4.59	(2) 4.60	(1) 0.50	(2) 0.52
	w	(1) .18	(2) .18	(1) .33	(2) .37	(1) .15	(2) .10
	τd	.056		-.029		-.160	
Mn (ppm)	$\bar{x}$	(1) 269	(2) 271	(1) 568	(2) 564	(1) 57	(2) 55
	w	(1) 18	(2) 21	(1) 40	(2) 38	(1) 11	(2) 10
	τd	-.102		.103		.190	

$\bar{x}$ = arithmetic mean
w = range of observed concentrations
τd = t-test ratio for the significance between mean $\bar{X}(1)$ and $\bar{X}(2)$. The value is expressed as $(\bar{X}_1-\bar{X}_2)/1/2(W_1+W_2)$
*τd values between -.304 and +.304 indicate no significant difference between mean $\bar{x}(1)$ and $\bar{x}(2)$ of the 95% confidence interval

Table 8 T-test ratios for uranium, copper, zinc, iron and manganese in total digestion versus sum of sequential partial extractions

CHAPTER 5

RESULTS OF SEQUENTIAL PARTIAL EXTRACTION PROCEDURES

5.1 Introduction

Sequential partial extractions of lake, stream and bog sediment samples have been used to investigate uranium, copper, zinc, iron and manganese partitioning into: 1) organic matter; 2) carbonate minerals; 3) amorphous manganese hydroxides; 4) amorphous iron hydroxides; 5) crystalline iron hydroxides; and 6) detrital minerals (Appendix VI, Tables 20 to 24). Histograms which illustrate proportions of total metal in each of the six sequential partial extractions have been plotted on Maps 2 to 6.

5.2 Uranium

5.2.1 Lake Sediments

The organic matter and amorphous iron hydroxide fractions generally account for all of the uranium present in lake sediments of the present study (Map 2). The mean ($\bar{x}$) and ranges (w) of uranium in organic matter have been expressed as proportions of total uranium in sediment and are as follows: in Lake 1 $\bar{x}$ = 95%, w = 84 to 100%; in Lake 2 $\bar{x}$ = 84%, w = 72 to 91%; and in Lake 3 $\bar{x}$ = 84%, w = 54 to 98%. Proportions of total uranium in the amorphous iron hydroxide fraction of sediments are as follows: in Lake 1 $\bar{x}$ = 5%, w = 0 to 16%; in Lake 2 $\bar{x}$ = 16%, w = 9 to 28%; and in Lake 3 $\bar{x}$ = 11%, w = 1 to 39% (Table 9,

		Proportion of Total Uranium in Each Fraction (expressed as a % of Total Metal)					
Sediment Type		Organic Matter	Carbonate Minerals	Amorphous Manganese Hydroxide	Amorphous Iron Hydroxide	Crystalline Iron Hydroxide	Detrital Minerals
Lake 1 n=11	$\bar{x}$	95	-	-	5	-	-
	c.v.	5	-	-	80	-	-
Lake 2 n = 7	$\bar{x}$	84	-	-	16	-	-
	c.v.	6	-	-	38	-	-
Lake 3 n = 15	$\bar{x}$	84	2	-	13	-	1
	c.v.	14	100	-	77	-	100
All Lakes n = 33	$\bar{x}$	88	1	-	11	-	-
	c.v.	11	100	-	82	-	-
Stream 1 n = 1	$\bar{x}$	80	-	3	10	-	7
	c.v.	-	-	-	-	-	-
Stream 2 n = 6	$\bar{x}$	88	-	-	11	-	-
	c.v.	10	-	-	77	-	-
Stream 3 n = 11	$\bar{x}$	85	-	1	4	2	8
	c.v.	13	-	> 100	> 100	> 100	> 100
All Streams n = 18	$\bar{x}$	85	-	1	7	1	6
	c.v.	13	-	> 100	> 100	> 100	> 100
Bog 1 n = 4	$\bar{x}$	96	-	2	2	-	-
	c.v.	1	-	25	74	-	-
Bog 2 n = 9	$\bar{x}$	99	-	-	1	-	1
	c.v.	1	-	-	> 100	-	> 100
Bog 3 n = 4	$\bar{x}$	92	-	1	3	-	4
	c.v.	5	-	> 100	36	-	61
All Bogs n = 17	$\bar{x}$	97	-	1	1	-	1
	c.v.	4	-	> 100	> 100	-	> 100

$\bar{x}$ - mean

c.v. - coefficient of variation expressed as standard deviation/$\bar{x}$ x 100

Table 9 The proportion of total uranium in component fractions of lake, stream and bog sediments

Appendix VI, Table 20). The mean proportions of uranium in the carbonate mineral, amorphous manganese hydroxide, crystalline iron hydroxide and detrital mineral fractions generally account for less than 5% of the total uranium in lake sediments.

5.2.2 Stream Sediments

The organic matter, amorphous iron hydroxide and detrital mineral fractions generally account for all of the uranium present in area stream sediments (Map 2). Proportions of total uranium in sediments are as follows: in organic matter in Stream 1 $\bar{x}$ = 80%, in Stream 2 $\bar{x}$ = 88% and w = 78 to 100%, in Stream 3 $\bar{x}$ = 85% and w = 66 to 98%; in amorphous iron hydroxides in Stream 1 $\bar{x}$ = 10%, in Stream 2 $\bar{x}$ = 11% and w = 0 to 22%, in Stream 3 $\bar{x}$ = 4% and w = 0 to 19%; and in detrital minerals in Stream 1 $\bar{x}$ = 7%, in Stream 2 $\bar{x}$ = 0% and w = 0 to 2% and in Stream 3 $\bar{x}$ = 8% and w = 0 to 33% (Table 9, Appendix VI, Table 20). The mean proportions of uranium in the carbonate mineral, amorphous manganese hydroxide and crystalline iron hydroxide fractions generally account for less than 5% of the total uranium in stream sediments (Table 9).

5.2.3 Bog Sediments

Proportions of total uranium in bog sediments are as follows: in organic matter in Bog 1 $\bar{x}$ = 96% and w = 94 to 98%, in Bog 2 $\bar{x}$ = 99% and w = 98 to 100%, and in Bog 3 $\bar{x}$ = 92% and w = 88 to 98% (Map 2, Table 9, Appendix VI, Table 20). Mean proportions in each of the carbonate mineral, amorphous manganese and iron hydroxide, crystalline iron hydroxide and detrital mineral fractions generally account for less than 5% of the total uranium content of bog sediments (Table 9).

5.3 Copper

5.3.1 Lake Sediments

The organic matter, amorphous iron hydroxide, crystalline iron hydroxide, amorphous manganese hydroxide and detrital mineral fractions account for the bulk of copper present in lake sediments of the present study (Map 3). Means and ranges of copper expressed as proportions of total metal in sediment are as follows: in organic matter in Lake 1 $\bar{x}$ = 52% and w = 11 to 62%, in Lake 2 $\bar{x}$ = 39% and w = 31 to 68%, in Lake 3 $\bar{x}$ = 48% and w = 27 to 60%; in amorphous iron hydroxide in Lake 1 $\bar{x}$ = 19% and w = 15 to 23%, in Lake 2 $\bar{x}$ = 42% and w = 31 to 68%, in Lake 3 $\bar{x}$ = 29% and w = 10 to 59%; in crystalline iron hydroxides in Lake 1 $\bar{x}$ = 9% and w = 7 to 13%, in Lake 2 $\bar{x}$ = 8% and w = 6 to 11%, in Lake 3 $\bar{x}$ = 9% and w = 1 to 20%; in amorphous manganese hydroxides in Lake 1 $\bar{x}$ = 5% and w = 3 to 8%, in Lake 2 $\bar{x}$ = 7% and w = 4 to 9%, in Lake 3 $\bar{x}$ = 7% and w = 4 to 10%; and in detrital minerals in Lake 1 $\bar{x}$ = 11% and w = 0 to 15%, in Lake 2 $\bar{x}$ = 2% and w = 1 to 3%, and in Lake 3 $\bar{x}$ = 5% and w = 4 to 8% (Table 10, Appendix VI, Table 21). Mean proportions in the carbonate mineral fraction generally account for less than 5% of the total copper content of lake sediments (Table 10).

5.3.2 Stream Sediments

The detrital mineral, amorphous iron hydroxide, organic matter, crystalline iron hydroxide and amorphous manganese hydroxide fractions account for the bulk of copper in local stream sediments (Map 3). Proportions of total copper in sediment are as follows: in detrital

		Proportion of Total Copper in Each Fraction (expressed as a % of Total Metal)					
Sediment Type		Organic Matter	Carbonate Minerals	Amorphous Manganese Hydroxide	Amorphous Iron Hydroxide	Crystalline Iron Hydroxide	Detrital Minerals
Lake 1 n=11	x̄	52	3	5	19	9	11
	c.v.	27	66	20	16	22	> 100
Lake 2 n = 7	x̄	39	-	7	42	8	2
	c.v.	23	-	29	26	13	50
Lake 3 n = 15	x̄	48	2	7	29	9	5
	c.v.	23	100	29	52	55	60
All Lakes n = 33	x̄	47	2	6	27	10	6
	c.v.	27	98	29	50	31	> 100
Stream 1 n = 1	x̄	9	4	7	22	9	49
	c.v.	-	-	-	-	-	-
Stream 2 n = 6	x̄	48	4	8	16	15	11
	c.v.	15	50	38	31	13	36
Stream 3 n = 11	x̄	24	2	8	26	8	28
	c.v.	66	100	50	38	63	54
All Streams n = 18	x̄	32	3	8	21	8	23
	c.v.	56	68	43	47	68	68
Bog 1 n = 4	x̄	63	8	8	9	13	1
	c.v.	9	25	25	22	7	100
Bog 2 n = 9	x̄	61	1	10	13	10	3
	c.v.	14	100	33	54	33	66
Bog 3 n = 4	x̄	55	2	4	16	2	23
	c.v.	29	50	25	25	50	48
All Bogs n = 17	x̄	62	3	7	12	9	7
	c.v.	18	98	61	56	53	> 100

$\bar{x}$ - mean

c.v. - coefficient of variation

Table 10 The proportion of total copper in component fractions of lake, stream and bog sediments minerals in the single Stream 1 sample $\bar{x}$ = 49%, in Stream 2 $\bar{x}$ = 11% and w = 6 to 15%, in Stream 3 $\bar{x}$ = 28% and w = 4 to 52%; in amorphous iron hydroxides in Stream 1 $\bar{x}$ = 22%, in Stream 2 $\bar{x}$ = 16% and w = 12 to 28%, in Stream 3 $\bar{x}$ = 26% and w = 19 to 55%; in organic matter in Stream 1 $\bar{x}$ = 9%, in Stream 2 $\bar{x}$ = 48% and w = 34 to 56%, in Stream 3 $\bar{x}$ = 24% and w = 4 to 42%; in crystalline iron hydroxides in Stream 1 $\bar{x}$ = 9%, in Stream 2 $\bar{x}$ = 15% and w = 12 to 18%, in Stream 3 $\bar{x}$ = 8% and w = 5 to 19%; and in amorphous manganese hydroxides in Stream 1 $\bar{x}$ = 7%, in Stream 2 $\bar{x}$ = 8% and w = 4 to 11%, in Stream 3 $\bar{x}$ = 8% and w = 1 to 15% (Table 10, Appendix VI, Table 21). Mean proportions in the carbonate mineral fraction generally account for less than 5% of the total copper in stream sediments (Table 10).

5.3.3 Bog Sediments

Means and ranges of copper expressed as proportions of total metal in bog sediments are as follows: in organic matter in Bog 1 $\bar{x}$ = 63% and w = 54 to 68%, in Bog 2 $\bar{x}$ = 61% and w = 49 to 75%, in Bog 3 $\bar{x}$ = 55% and w = 37 to 80%; in amorphous iron hydroxide in Bog 1 $\bar{x}$ = 9% and w = 6 to 11%, in Bog 2 $\bar{x}$ = 13% and w = 4 to 29%, in Bog 3 $\bar{x}$ = 16% and w = 10 to 49%; in crystalline iron hydroxides in Bog 1 $\bar{x}$ = 13% and w = 12 to 13%, in Bog 2 $\bar{x}$ = 10% and w = 6 to 15%, in Bog 3 $\bar{x}$ = 2% and w = 1 to 4%; in amorphous manganese hydroxides in Bog 1 $\bar{x}$ = 8% and w = 5 to 11%, in Bog 2 $\bar{x}$ = 10% and w = 5 to 17%, in Bog 3 $\bar{x}$ = 4% and w = 2 to 6%; in detrital minerals in Bog 1 $\bar{x}$ = 1% and w = 0 to 2%, in Bog 2 $\bar{x}$ = 3% and w = 1 to 8%, in Bog 3 $\bar{x}$ = 23% and w = 5 to 37%; and in carbonate minerals in Bog 1 $\bar{x}$ = 8% and w = 5 to 11%; in Bog 2 $\bar{x}$ = 1% and w = 0 to 3%, in Bog 3 $\bar{x}$ = 2% and w = 0 to 2% (Map 3, Table 10, Appendix VI, Table 22).

5.4 Zinc

5.4.1 Lake Sediments

The amorphous iron hydroxide, organic matter, detrital mineral, amorphous manganese hydroxide and crystalline iron hydroxide fractions account for the bulk of zinc in local lake sediments (Map 4). Means and ranges of zinc expressed as proportions of total metal in sediment are as follows: in amorphous iron hydroxides in Lake 1 $\bar{x}$ = 39% and w = 21 to 48%, in Lake 2 $\bar{x}$ = 45% and w = 30 to 68%, in Lake 3 $\bar{x}$ = 42% and w = 9 to 61%; in organic matter in Lake 1 $\bar{x}$ = 36% and w = 15 to 55%, in Lake 2 $\bar{x}$ = 17% and w = 8 to 26%, in Lake 3 $\bar{x}$ = 30% and w = 5 to 67%; in detrital minerals in Lake 1 $\bar{x}$ = 12% and w = 0 to 33%, in Lake 2 $\bar{x}$ = 16% and w = 11 to 29%, in Lake 3 $\bar{x}$ = 17% and w = 5 to 33%; in amorphous manganese hydroxides in Lake 1 $\bar{x}$ = 2% and w = 0 to 10%, in Lake 2 $\bar{x}$ = 13% and w = 8 to 19%, in Lake 3 $\bar{x}$ = 9 and w = 5 to 15%; and in crystalline iron hydroxides in Lake 1 $\bar{x}$ = 12% and 2 = 0 to 24%, in Lake 2 $\bar{x}$ = 7% and w = 2 to 22%, in Lake 3 $\bar{x}$ = 6% and w = 0 to 20% (Table 11, Appendix VI, Table 22). Mean proportions in the carbonate mineral fraction generally account for less than 5% of the total zinc in lake sediments (Table 11).

5.4.2 Stream Sediments

The detrital mineral, amorphous iron hydroxide, organic matter, crystalline iron hydroxide and amorphous manganese hydroxide fractions account for the bulk of zinc in local stream sediments (Map 4). Means

Sediment Type		Proportion of Total Zinc in Each Fraction (expressed as a % of Total Metal) Organic Matter	Carbonate Minerals	Amorphous Manganese Hydroxide	Amorphous Iron Hydroxide	Crystalline Iron Hydroxide	Detrital Minerals
Lake 1 n=11	$\bar{x}$	36	-	2	39	12	12
	c.v.	33	-	> 100	21	58	75
Lake 2 n = 7	$\bar{x}$	17	< 1	13	45	7	16
	c.v.	35	100	31	27	86	38
Lake 3 n = 15	$\bar{x}$	30	< 1	9	42	6	17
	c.v.	53	100	44	33	66	47
All Lakes n = 33	$\bar{x}$	28	< 1	7	41	8	15
	c.v.	54	> 100	75	30	81	56
Stream 1 n = 1	$\bar{x}$	10	-	4	18	10	58
	c.v.						
Stream 2 n = 6	$\bar{x}$	24	-	7	23	5	38
	c.v.	58	-	71	43	20	50
Stream 3 n = 11	$\bar{x}$	5	2	6	13	5	63
	c.v.	45	100	50	> 100	80	25
All Streams n = 18	$\bar{x}$	15	1	7	15	6	54
	c.v.	71	> 100	58	92	57	38
Bog 1 n = 4	$\bar{x}$	90	-	-	1	7	2
	c.v.	10	-	-	> 100	71	> 100
Bog 2 n = 9	$\bar{x}$	57	2	4	10	10	17
	c.v.	33	> 100	100	60	70	> 100
Bog 3 n = 4	$\bar{x}$	17	3	4	11	6	58
	c.v.	65	66	75	36	17	34
All Bogs n = 17	$\bar{x}$	55	2	3	8	8	24
	c.v.	53	> 100	> 100	77	80	> 100

$\bar{x}$ - mean

c.v. - coefficient of variation

Table 11 The proportion of total zinc in the component fractions of lake, stream and bog sediments and ranges expressed as proportions of total metal in sediment are as follows: in detrital minerals in the single sample along Stream 1 $\bar{x}$ = 58%, in Stream 2 $\bar{x}$ = 38% and w = 12 to 73%, in Stream 3 $\bar{x}$ = 63% and w = 24 to 82%; in amorphous iron hydroxides in Stream 1 $\bar{x}$ = 18%, in Stream 2 $\bar{x}$ = 23% and w = 7 to 35%, in Stream 3 $\bar{x}$ = 13% and w = 5 to 59%; in organic matter in Stream 1 $\bar{x}$ = 10%, in Stream 2 $\bar{x}$ = 24% and w = 4 to 49%, in Stream 3 $\bar{x}$ = 5% and w = 6 to 20%; in crystalline iron hydroxides in Stream 1 $\bar{x}$ = 10%, in Stream 2 $\bar{x}$ = 5% and w = 4 to 10%, in Stream 3 $\bar{x}$ = 5% and w = 2 to 16%; and in amorphous manganese hydroxides in Stream 1 $\bar{x}$ = 4%, in Stream 2 $\bar{x}$ = 7% and w = 0 to 14%, in Stream 3 $\bar{x}$ = 6% and w = 4 to 13% (Table 12, Appendix VI, Table 22). Mean proportions in the carbonate mineral fraction generally account for less than 5% of the total zinc in stream sediments (Table 11).

5.4.3 Bog Sediments

The organic matter, detrital mineral, crystalline iron hydroxide and amorphous iron hydroxide fractions account for the bulk of zinc in local bog sediments (Map 4). Means and ranges expressed as proportions of total zinc in sediment are as follows: in organic matter in Bog 1 $\bar{x}$ = 90% and w = 76 to 100%, in Bog 2 $\bar{x}$ = 57% and w = 22 to 77%, in Bog 3 $\bar{x}$ = 17% and w = 11 to 35%; in detrital minerals in Bog 1 $\bar{x}$ = 2% and w = 0 to 8%, in Bog 2 $\bar{x}$ = 17% and w = 0 to 54%, in Bog 3 $\bar{x}$ = 58% and w = 23 to 70%; in crystalline iron hydroxides in Bog 1 $\bar{x}$ = 7% and w = 0 to 12%, in Bog 2 $\bar{x}$ = 10% and w = 0 to 25%, in Bog 3 $\bar{x}$ = 6% and w = 4 to 18%; and in amorphous iron hydroxides in Bog 1 $\bar{x}$ = 1% and w = 0 to 9%, in Bog 2 $\bar{x}$ = 10% and w = 0 to 15%, in Bog 3 $\bar{x}$ = 11% and w = 7 to 18% (Table 11, Appendix VI, Table 22). Mean proportions in the carbonate mineral and amorphous manganese hydroxide fractions generally account for less than 5% of the total zinc in bog sediments (Table 11).

5.5 Iron

5.5.1 Lake Sediments

The amorphous iron hydroxide, detrital mineral, crystalline iron hydroxide and organic matter fractions retain the bulk of iron present in lake sediments of the present study (Map 5). Means and ranges expresed as proportions of total iron in sediment are as follows: in amorphous iron hydroxides in Lake 1 $\bar{x}$ = 30% and w = 17 to 44%, in Lake 2 $\bar{x}$ = 45% and w = 27 to 67%, in Lake 3 $\bar{x}$ = 48% and w = 20 to 78%, in detrital minerals in Lake 1 $\bar{x}$ = 38% and w = 21 to 60%; in Lake 2 $\bar{x}$ = 22% and w = 9 to 44%, in Lake 3 $\bar{x}$ = 31% and w = 11 to 62%; in crystalline iron hydroxides in Lake 1 $\bar{x}$ = 12% and w = 4 to 20%, in Lake 2 $\bar{x}$ = 17% and w = 0 to 41%, in Lake 3 $\bar{x}$ = 7% and w = 2 to 19%; and in organic matter in Lake 1 $\bar{x}$ = 10% and w = 2 to 28%, in Lake 2 $\bar{x}$ = 10% and w = 3 to 19%, in Lake 3 $\bar{x}$ = 8% and w = 1 to 20% (Table 12, Appendix VI, Table 23). The mean proportions in the carbonate mineral and amorphous manganese hydroxide fractions generally account for less than 5% of the total iron in lake sediments (Table 12).

5.5.2 Stream Sediments

The detrital mineral, amorphous iron hydroxide and organic matter fractions generally account for the bulk of iron in local stream sediments. Means and ranges expressed as proportions of total iron in

Sediment Type		Proportion of Total Iron in Each Fraction (expressed as a % of Total Metal)					
		Organic Matter	Carbonate Minerals	Amorphous Manganese Hydroxide	Amorphous Iron Hydroxide	Crystalline Iron Hydroxide	Detrital Minerals
Lake 1 n=11	$\bar{x}$	10	6	2	30	12	38
	c.v.	70	83	100	27	42	29
Lake 2 n = 7	$\bar{x}$	10	2	5	45	17	22
	c.v.	50	100	40	31	88	50
Lake 3 n = 15	$\bar{x}$	8	1	4	48	7	31
	c.v.	100	> 100	75	35	71	48
All Lakes n = 33	$\bar{x}$	9	3	4	42	11	31
	c.v.	82	> 100	66	38	83	49
Stream 1 n = 1	$\bar{x}$	8	2	2	59	4	25
	c.v.	-	-	-	-	-	-
Stream 2 n = 6	$\bar{x}$	6	1	3	19	4	53
	c.v.	66	100	66	> 100	25	42
Stream 3 n = 11	$\bar{x}$	3	1	2	17	3	74
	c.v.	66	100	50	94	33	22
All Streams n = 18	$\bar{x}$	4	< 1	2	25	3	64
	c.v.	78	> 100	66	78	48	35
Bog 1 n = 4	$\bar{x}$	40	12	9	7	21	3
	c.v.	25	92	66	> 100	43	66
Bog 2 n = 9	$\bar{x}$	39	-	7	22	6	26
	c.v.	51	-	71	32	33	69
Bog 3 n = 4	$\bar{x}$	17	1	3	16	4	59
	c.v.	> 100	100	33	69	25	58
All Bogs n = 17	$\bar{x}$	39	3	6	20	10	29
	c.v.	45	> 100	84	44	84	98

$\bar{x}$ - mean

c.v. - coefficient of variation

Table 12 The proportion of totaliron in the component fractions of lake, stream and bog sediments sediment are as follows: in detrital minerals in the single sample from Stream 1 $\bar{x}$ = 25%, in Stream 2 $\bar{x}$ = 53% and w = 30 to 91%, in Stream 3 $\bar{x}$ = 74% and w = 27 to 87%; in amorphous iron hydroxide fractions in Stream 1 $\bar{x}$ = 59%, in Stream 2 $\bar{x}$ = 19% and w = 7 to 54%, in Stream 3 $\bar{x}$ = 17% and w = 4 to 64%; and in organic matter in Stream 1 $\bar{x}$ = 8%, in Stream 2 $\bar{x}$ = 6% and w = 0 to 10%, in Stream 3 $\bar{x}$ = 3% and w = 2 to 10% (Table 12, Appendix VI, Table 23). The mean proportions in the carbonate mineral, amorphous manganese hydroxide and crystalline iron hydroxide fractions generally account for less than 5% of the total iron in stream sediments (Table 12).

5.5.3 Bog Sediments

Means and ranges of iron expressed as proportions of total iron in bog sediments are as follows: in organic matter in Bog 1 $\bar{x}$ = 40% and w = 26 to 51%, in Bog 2 $\bar{x}$ = 39% and w = 13 to 77%, in Bog 3 $\bar{x}$ = 17% and w = 2 to 54%; in detrital minerals in Bog 1 $\bar{x}$ = 3% and w = 1 to 6%, in Bog 2 $\bar{x}$ = 26% and w = 6 to 59%, in Bog 3 $\bar{x}$ = 59% and w = 1 to 83%; in amorphous iron hydroxides in Bog 1 $\bar{x}$ = 7% and w = 3 to 20%, in Bog 2 $\bar{x}$ = 22% and w = 16 to 31%, in Bog 3 $\bar{x}$ = 16% and w = 10 to 35%; in crystalline iron hydroxides in Bog 1 $\bar{x}$ = 21% and 2 = 17 to 33%; in Bog 2 $\bar{x}$ = 6% and w = 4 to 11%, in Bog 3 $\bar{x}$ = 4% and w = 3 to 6%; in carbonate minerals in Bog 1 $\bar{x}$ = 12% and w = 3 to 29%, in Bog 2 $\bar{x}$ = 0% and w = 0 to <1%; in Bog 3 $\bar{x}$ = 1% and w = 0 to 2%; and in amorphous manganese hydroxides in Bog 1 $\bar{x}$ = 9% and w = 1 to 13%, in Bog 2 $\bar{x}$ = 7% and w = 1 to 21%, in Bog 3 $\bar{x}$ = 3% and w = 2 to 4% (Map 5, Table 12, Appendix VI, Table 23).

5.6 Manganese

5.6.1 Lake Sediments

The organic matter, amorphous iron hydroxide, amorphous manganese hydroxide and detrital mineral fractions account for the bulk of manganese in lake sediments of the present study (Map 6). Means and ranges expressed as proportions of total metal in sediment are as follows: in organic matter in Lake 1 $\bar{x}$ = 52% and w = 21 to 67%, in Lake 2 $\bar{x}$ = 30% and w = 6 to 36%, in Lake 3 $\bar{x}$ = 42% and w = 18 to 76%; in amorphous iron hydroxides in Lake 1 $\bar{x}$ = 16% and w = 14 to 22%, in Lake 2 $\bar{x}$ = 21% and w = 15 to 33%, in Lake 3 $\bar{x}$ = 18% and w = 7 to 34%; in amorphous manganese hydroxide in Lake 1 $\bar{x}$ = 9% and w = 5 to 11%, in Lake 2 $\bar{x}$ = 22% and w = 13 to 50%, in Lake 3 $\bar{x}$ = 17% and w = 6 to 28%; and in detrital minerals in Lake 1 $\bar{x}$ = 16% and w = 7 to 50%, in Lake 2 $\bar{x}$ = 22% and w = 7 to 31%, in Lake 3 $\bar{x}$ = 20% and w = 4 to 51%. The mean proportions in the carbonate mineral and crystalline iron hydroxide fractions generally account for less than 5% of the total manganese in lake sediments (Table 13, Appendix VI, Table 24).

5.6.2 Stream Sediments

The amorphous iron hydroxide, organic matter, detrital mineral and amorphous manganese hydroxide fractions account for the bulk of manganese in local stream sediments (Map 6). Means and ranges expressed as proportions of total metal in sediment are as follows: in amorphous iron hydroxides in the single Stream 1 sample $\bar{x}$ = 51%, in Stream 2 $\bar{x}$ = 9% and w = 6 to 18%, in Stream 3 $\bar{x}$ = 6% and w = 1 to 33%, in organic matter in Stream 1 $\bar{x}$ = 9%, in Stream 2 $\bar{x}$ = 37% and w = 8 to 65%, in Stream 3 $\bar{x}$ = 7% and w = 3 to 19%; in detrital minerals in Stream 1

Sediment Type		Proportion of Total Manganese in Each Fraction (expressed as a % of Total Metal) Organic Matter	Carbonate Minerals	Amorphous Manganese Hydroxide	Amorphous Iron Hydroxide	Crystalline Iron Hydroxide	Detrital Minerals
Lake 1 n=11	$\bar{x}$	52	1	9	16	6	16
	c.v.	23	100	22	19	33	75
Lake 2 n = 7	$\bar{x}$	30	-	22	21	5	22
	c.v.	33	-	55	24	20	36
Lake 3 n = 15	$\bar{x}$	42	-	17	18	4	20
	c.v.	43	-	41	39	27	75
All Lakes n = 33	$\bar{x}$	43	< 1	15	18	6	19
	c.v.	38	97	60	34	86	68
Stream 1 n = 1	$\bar{x}$	9	3	30	51	4	4
	c.v.	-	-	-	-	-	-
Stream 2 n = 6	$\bar{x}$	37	< 1	9	9	3	33
	c.v.	52	71	31	60	33	84
Stream 3 n = 11	$\bar{x}$	7	< 1	5	6	2	79
	c.v.	71	92	> 100	> 100	55	27
All Streams n = 18	$\bar{x}$	17	1	8	11	2	61
	c.v.	> 100	> 100	> 100	> 100	66	52
Bog 1 n = 4	$\bar{x}$	84	4	4	5	3	-
	c.v.	4	60	25	40	33	-
Bog 2 n = 9	$\bar{x}$	66	2	2	3	6	21
	c.v.	22	80	50	100	70	71
Bog 3 n = 4	$\bar{x}$	23	1	3	5	2	67
	c.v.	> 100	> 100	77	62	50	49
All Bogs n = 17	$\bar{x}$	60	2	3	3	4	22
	c.v.	45	100	49	100	86	> 100

$\bar{x}$ - mean

c.v. - coefficient of variation

Table 13 The proportion of total manganese in the component fractions of lake, stream and bog sediments

$\bar{x}$ = 4%, in Stream 2 $\bar{x}$ = 33% and w = 10 to 76%, in Stream 3 $\bar{x}$ = 79% and w = 18 to 94%; and in amorphous manganese hydroxides in Stream 1 $\bar{x}$ = 30%, in Stream 2 $\bar{x}$ = 9% and w = 5 to 13%, in Stream 3 $\bar{x}$ = 5% and w = 1 to 30%. The mean proportions in the carbonate mineral and crystalline iron hydroxide fractions generally account for less than 5% of the total manganese in stream sediments (Table 13, Appendix VI, Table 24).

5.6.3 Bog Sediments

The organic matter and detrital mineral fractions retain the bulk of manganese in local bog sediments (Map 6). Means and ranges expressed as proportions of total manganese in sediment are as follows: in organic matter in Bog 1 $\bar{x}$ = 84% and w = 79 to 86%, in Bog 2 $\bar{x}$ = 66% and w = 47 to 86%, in Bog 3 $\bar{x}$ = 23% and w = 3 to 66%; and in detrital mineral fractions in Bog 1 $\bar{x}$ = 0% and w = 0 to 1%, in Bog 2 $\bar{x}$ = 21% and w = 4 to 45%, in Bog 3 $\bar{x}$ = 67% and w = 11 to 94% (Table 13, Appendix VI, Table 24). The mean proportions in the carbonate mineral, amorphous iron and manganese hydroxide and crystalline iron hydroxide fractions generally account for less than 5% of the total manganese in bog sediments (Table 13).

CHAPTER 6

RESULTS OF PARTIAL EXTRACTION PROCEDURES

6.1 Introduction

Uranium, copper, zinc, and manganese concentrations in the humic-fulvic acid and humin components of sediment organic matter are compiled in Appendix VI, Tables 25 to 28. Iron concentrations in humic-fulvic acid components have not been analyzed for since iron is observed to show a relatively low preference for partitioning into most sediment organic matter. In lake sediments the mean proportion of total iron in organic matter is 9% compared to 88% for uranium, 47% for copper, 28% for zinc, and 43% for manganese. In stream sediments mean proportions in organic matter are 5% for iron, 85% for uranium, 32% for copper, 15% for zinc, and 17% for manganese, whereas in bog sediments mean proportions in organic matter are 39% for iron, 97% for uranium, 62% for copper, 55% for zinc and 60% for manganese (Tables 9 to 13).

6.2 Uranium

Manskaya and Drozdova (1968) have shown that uranium is preferentially retained by organic humic and fulvic acids and that the proportion of metal in these phases is influenced by pH. Their conclusions were based on a series of laboratory experiments whereby uranyl sulphate was added to humic and fulvic acid extracts of peat which were then adjusted to various pH conditions. Results of the current study confirm that partitioning of uranium into fulvic and humic acids in a natural environment is controlled by the pH of the superjacent waters (Appendix VI, Table 25, Figure 3). The proportion of the total uranium in sediment increases from 61% to 67% in humic acid over the pH range in superjacent waters of 4.7 to 5.8 and decreases to a minimum of 23% as the pH increases to 6.5. In contrast, the proportion of total uranium in fulvic acid increases from a minimum of 22% at pH 5.8 to a maximum 49% at pH 6.5. Over the pH range 4.7 to 6.5 humic and fulvic acids combine to account for 53 to 97% of the total uranium in sediments. A maximum of 47% is fixed into the humin fraction which includes all other fractions of sediment other than organic matter. Sequential partial extractions have identified amorphous iron hydroxides and detrital minerals as the most important elements of the humin fraction to retain uranium. Maximum retention of uranium by these fractions occurs over the pH range 6.0 to 6.3.

6.3 Copper, Zinc and Manganese

In contrast to uranium, concentrations of copper, zinc and manganese in the humic and fulvic acid components of sediment organic matter are not influenced by pH (Appendix VI, Tables 26 to 28). Within the pH range 4.7 to 6.5, the largest proportion of copper in organic matter is retained by humic acid whereas the largest proportions of zinc and manganese are typically partitioned into fulvic acid. Similar relationships between copper, zinc and manganese and humic-fulvic acids have been reported for lake sediments of the Elliot Lake region of northern Ontario (Schaef 1975). Significant proportions of total

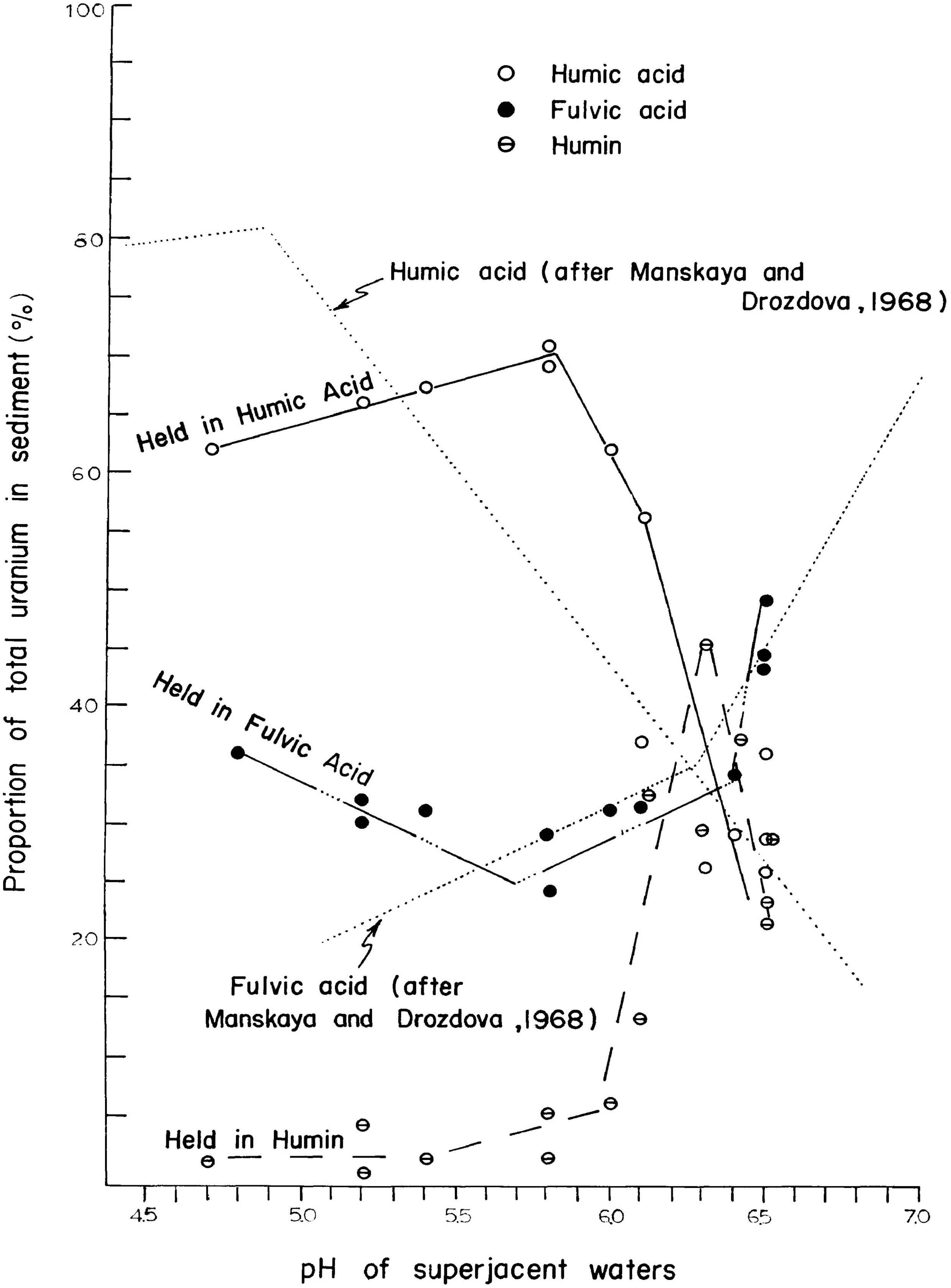

Figure 3 Proportion of total uranium in humic acid, fulvic acid and the humin fraction of sediments versus the pH of superjacent waters

copper (up to 72%) total zinc (up to 93%) and total manganese (up to 81%) are retained by the humin fraction of sediments (Tables 26 to 28). Sequential partial extractions have identified amorphous iron hydroxides and detrital minerals as the most important elements of the humin fraction to retain metals.

6.3.1 The Relationship Between Percent Carbon and the Proportions of Copper, Zinc and Manganese in the Organic Matter Fraction of Area Lake, Stream and Bog Sediments

An examination of the sequential partial extraction data shows that there are substantial variations in the proportion of total copper, zinc and manganese in the organic matter fraction of lake, stream and bog sediments of the present study. The proportions of these metals in the organic matter fraction of sediments are directly related to the amount of total carbon present in the sediments and increase as the total carbon content increases (Figure 4).

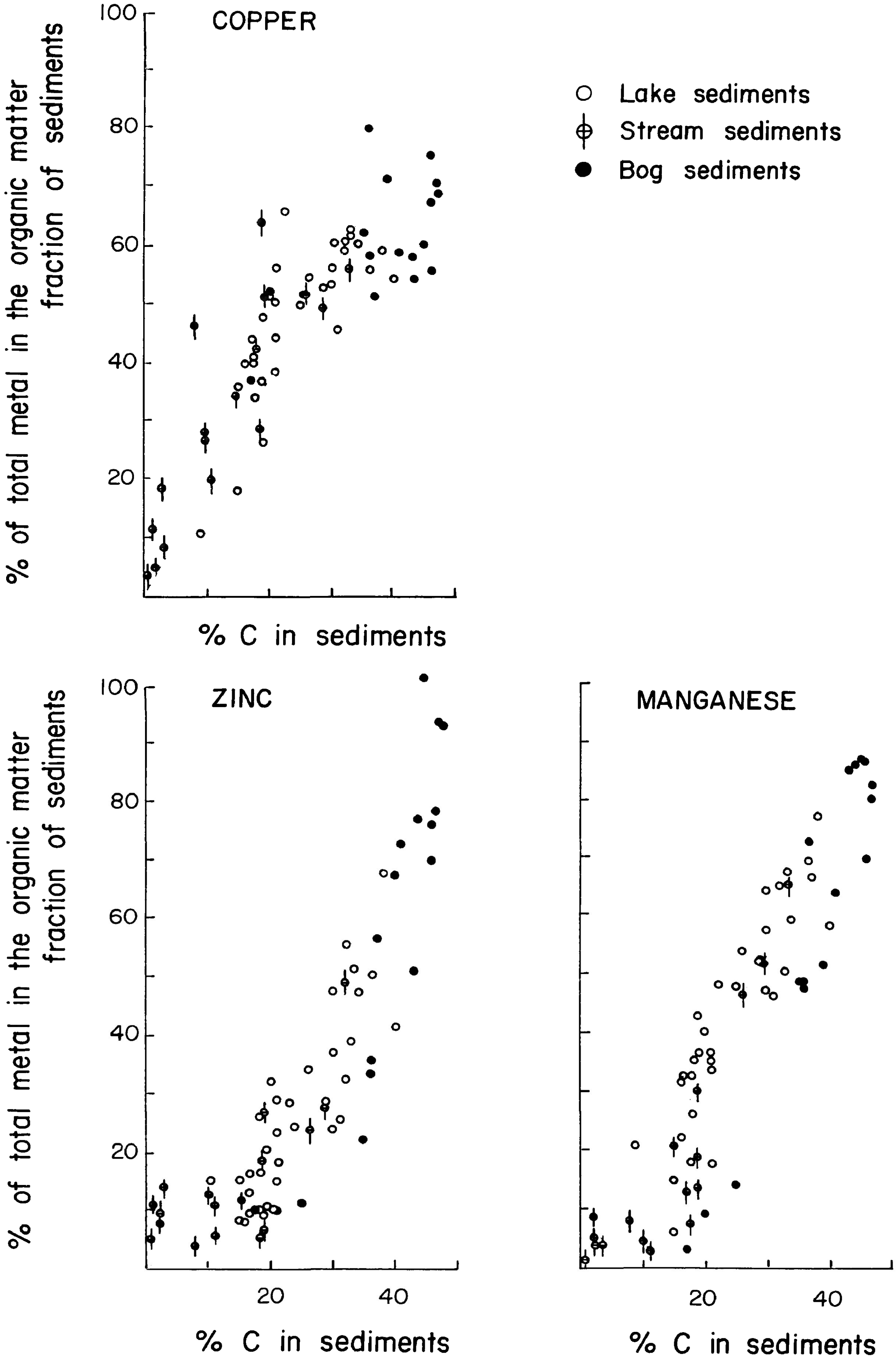

Figure 4 Proportion of total copper, zinc and manganese in the organic matter fraction of sediments versus the percent carbon in sediments

CHAPTER 7

DISCUSSION

7.1 Physico-Chemical Parameters and Percent Carbon in the Lake, Stream and Bog Environment

7.1.1 Lakes

At the time of the survey Lake 1 was characterized by having the most alkaline, conductive, oxygenated and the warmest waters of the three lakes in the study (Table 2). The sediments of the lake have the highest concentration of organic matter ($\bar{x}$ = 30.3% carbon, Table 2). The organic content of lake sediments has been shown to be related to decay processes acting on plant and animal debris within host lakes and surrounding catchment basins (Hutchinson, 1957; Swain, 1958; Ruttner, 1963; Kuznetsov, 1970; Jonasson, 1976; and Coker et al, 1979). The rich organic nature of Lake 1 sediments would indicate a relatively high supply of plant and animal debris. Both the rich organic content and shallow water depth ($\bar{x}$ = 3.6 metres) of Lake 1 may contribute to the atypical parameter values observed. Organic matter has been shown to be a potential reducing agent (Rashid and Leonard, 1973; Theis and Singer, 1973). The relatively low Eh values in the superjacent waters of Lake 1 are probably related to the rich organic nature of the underlying sediment. In Lakes 2 and 3, sediment is less organic rich and as a result Eh conditions are more oxidizing.

Superjacent waters in Lake 1 display near alkaline pH conditions ($\bar{x}$ = 7.0, Table 2) even though more acidic pH might be expected due to the organic-rich nature of the underlying sediment. Berner (1970) has shown that organic matter in sediments can promote the reduction of sulphate to hydrogen sulphide which in turn reacts with iron to produce iron sulphide. As a result of the formation of iron sulphide H^+ ions are liberated which could promote more acidic pH conditions in superjacent waters. The reaction between hydrogen sulphide and iron is represented as follows: $Fe^{++} + H_2S + S^\circ \rightarrow FeS_2 + 2H^+$ (after Berner, 1970). Relatively acidic pH's in Lakes 2 and 3 are interpreted to be the result of the formation of iron sulphide and the release of H^+ ions. In Lake 1 which is relatively shallow, superjacent waters are well oxygenated ($\bar{x}$ = 9.7 ppm, Table 2). Oxygen is therefore available to react with liberated H^+ ions to produce OH^- species which instead promotes the more alkaline pH's observed. Organic matter occurs in both dissolved and colloidal forms in waters (Coker et al, 1979). Since conductivity is related to the concentration of dissolved and colloidal material and salts in solution, environments enriched in organic matter like Lake 1 would be expected to demonstrate high conductivity ($\bar{x}$ = 103 micromhos/cm, Table 2).

The carbon content of area lake sediments vary considerably (coefficient of variation = 31, Table 2). This feature is interpreted to reflect compositional changes in sediment. A lake sediment classification system based on composition has been proposed by Jonasson (1976). Three main classifications have been described. Organic gels (gyttja) consist of fine-grained, decomposed organic mull and commonly occur in central basins of lakes. Organic sediments consist of relatively undecomposed fibrous coarse-grained mull and host partially-rotted twig, root and leaf fragments within their matrix. Organic sediments are more typical of near-shore environments particularly adjacent to wetlands. Inorganic sediments are composed of detrital mineral debris and inorganic hydroxides and abound in near-shore inlet and outlet locations. On the basis of percent carbon and sample location data, area lake sediments have been interpreted according to the Jonasson classification. Throughout most of Lake 1, the sediments are fine-grained gyttja. Percent carbon values range from 29.3 to 39.7 and are the highest encountered in the study area. Sample 1, Lake 1, along the southern shoreline is an inorganic sediment with a low percent carbon value of 9.9 (Maps 2 to 6, Appendix VI, Table 19). Lake 2 is characterized by gyttja sediments of lower percent carbon than in Lake 1 according to values that range from 15.0 to 21.0 (Appendix VI, Table 19). In Lake 3, percent carbon in sediments varies from 14.9 to 38.2. Sediments throughout most of the lake are fine-grained gyttja. Samples 39 and 40 collected adjacent to the outlet from Bog 2 are organic sediments according to their coarse-grained, fibrous character and presence of leaf and twig debris in their matrix.

7.1.2 Streams

A mean carbon value of 14.0% in area stream sediments is low in comparison to the 24.0% value in area lake sediments (Table 2). This feature is attributed to lower contents of organic matter contained in the sediments. In area stream sediments, mineral grains of detrital K-feldspar, quartz and mica are commonly observed. Mineral debris is most prevalent in Stream 3 sediments where carbon ranges from 0.5 to 19.1% and is least abundant in Stream 2 sediments where carbon ranges from 8.4 to 32.9%.

Of area streams, Stream 3 waters have the highest mean temperature and highest dissolved oxygen concentrations according to values of 15.7°C and 7.3 ppm respectively (Table 2). In contrast, mean temperature and dissolved oxygen values are 10.5°C and 3.5 ppm in Stream 1 and 12.3°C and 5.6 ppm in Stream 2. Lower values for temperature and dissolved oxygen in Streams 1 and 2 are attributed to vegetative cover which acts to partially block out warming sunlight whereas penetration is unrestricted in Stream 3 which flows through relatively open terrain. A lower mean conductivity value of 104 micromhos/cm in area streams versus 108 and 112 micromhos/cm in area lakes and bogs respectively, is probably a factor of the lower organic matter content in stream waters (Table 2).

7.1.3 Bogs

A mean of 45.9 percent carbon in Bog 1 and 41.4 percent carbon in Bog 2 reflect the organic-rich nature of contained sediments and are the highest values recorded in the study area (Table 2). A mean of 25.2 percent carbon in Bog 3 indicates a lower organic matter content of sediments in this bog. Examination of the auger samples has shown that bogs are generally comprised of an undecomposed surface and intermediate layer of moss underlain by a basal layer of completely rotted peat. The organic-rich nature of bog sediments is attributed to the supply of plant and animal debris, minimum water circulation and the efficiency of decay processes in these environments which are aided by prevailing acidic-reducing conditions. Mean conductivity determined to be 112 micromhos/cm for area bog waters, is probably related to colloidal and dissolved organic matter in solution. A low mean temperature of 11.9°C, dissolved oxygen content of 3.2 ppm and a Eh value of 545 millivolts in bogs may be linked to the effects of surface moss which partially obstructs the penetration of warming sunlight and oxygen into subsurface waters. In contrast to the acidic pH and low dissolved oxygen content in Bog 2, near alkaline pH conditions and relatively high dissolved oxygen concentrations prevail at stations 12 and 13 in Bog 1 near the outlet from Lake 1 (Appendix VI, Table 19, Maps 2 to 6). These conditions may be influenced by the alkaline, highly-oxygenated waters of Lake 1 which enter the bog from the north end of the lake.

7.2 Metal Partitioning in Lake, Stream and Bog Sediments

7.2.1 Uranium

Previous studies have only been able to suggest an association between total uranium and organic matter in drainage sediments (Lehto et al, 1976). In the present study sequential partial extractions have permitted the specific examination of organic matter and have confirmed that the bulk of uranium in sediments is retained by this fraction (Map 2, Appendix VI, Table 20). Furthermore, the proportion of uranium in organic matter components has been shown to be sensitive to the pH of the superjacent waters (Figure 3). In environments with pH < 5.8 the largest proportion of total uranium is in the humic acid component of organic matter, whereas with pH > 6.4 it is in the fulvic acid component. Between pH 5.8 and 6.4 the proportion of total uranium in organic matter decreases and significant amounts are retained in other fractions.

In the present study the pH of superjacent waters range from 6.2 to 6.8 in Bog 1 and from 4.7 to 5.4 in Bog 2. Eighty-eight to 100% of the total uranium in the sediments of these bogs is in organic matter and on the basis of pH, the bulk of metal must be in fulvic acid in Bog 1 and in humic acid in Bog 2 (Figure 3). The pH ranges from 6.5 to 7.2 in Lake 1 and 84 to 100% of the total uranium is held in organic matter. The bulk of the metal in Lake 1 sediments is likely partitioned into the fulvic acid component according to pH (Figure 3). The pH of waters in Lakes 2 and 3 vary over a wider range (pH 5.6 - 6.5) and partitioning characteristics of uranium are more complex. Relatively acidic pH prevails in near-shore sites adjacent to and down-drainage of bogs due to the mixing of acidic bog waters with lake waters (pH 5.6 to 6.0 in Samples 17 to 19 Lake 2, pH 5.8 to 6.1 in Samples 39 to 42 Lake 3). Eighty-six to 98% of the total uranium in sediments at these locations is partitioned into organic matter and as a consequence of the acidic pH the bulk of the metal must be in humic acid (Figure 3). The pH of superjacent waters is less acidic in central-lake basin locations remote from bogs (pH 6.0 to 6.4 in Samples 21 to 23 Lake 2, pH 6.1 to 6.5 in Samples 43 to 57 Lake 3).

Since organic matter retains relatively small amounts of uranium at these pH conditions, significant proportions of metal are fixed by other sediment fractions. Of particular importance in centre-lake basin sediments are amorphous iron hydroxides which retain up to 39% of the total uranium (Map 2, Sample 43, Lake 3). Jenne (1968) has shown that under normal temperatures (25°C) and pressures (1 atm), the stability of iron and manganese hydroxides is controlled by Eh-pH conditions (Figure 5). Iron hydroxides are stable where pH is greater than 3.0 and Eh is less reducing than -900 millivolts whereas manganese hydroxides are stable where pH is greater than 6.6 and Eh is less reducing than -1200 millivolts. The Eh (+270 to +700 millivolts) and pH (4.7 to 7.6) conditions of superjacent waters in the present study dictate that iron hydroxides are stable throughout local lake, stream and bog environments whereas manganese hydroxides are only stable in isolated locations of lakes and streams and are generally unstable in bogs (Appendix VI, Table 19). The proportion of uranium retained in hydroxides is therefore influenced by the pH of the superjacent waters since pH affects the stability of this fraction in sediments. Although iron hydroxides are stable throughout the study area according to Eh-pH conditions, the relative abundance of this fraction varies considerably. Iron concentrations in amorphous iron hydroxides provide an estimate of abundance and indicate that this fraction is least abundant in bogs ($\bar{x}$ = 0.19% Fe) and most abundant in lakes ($\bar{x}$ = 0.73% Fe). Therefore the largest proportions of total uranium that are retained by the amorphous iron hydroxide fraction occur in lake sediments (Map 2, Appendix VI, Table 20). Amorphous manganese

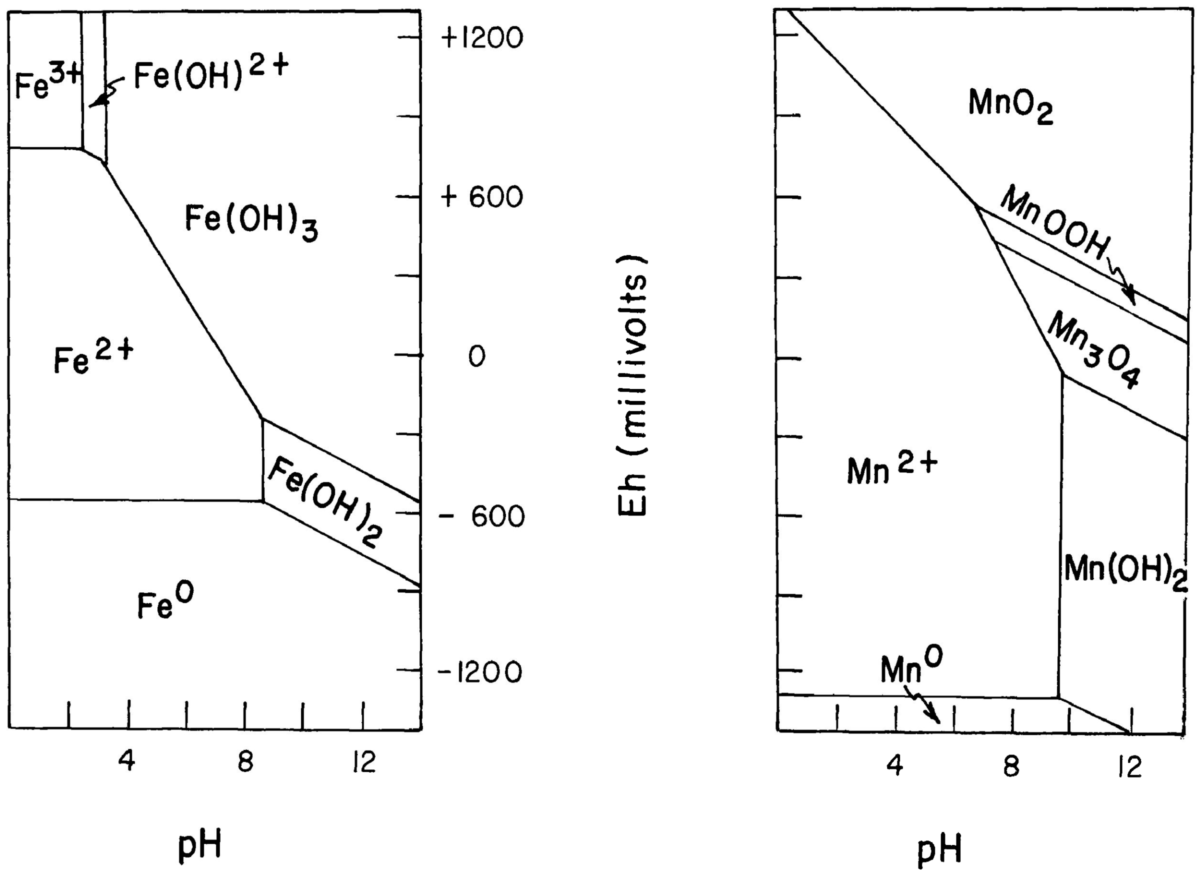

Figure 5 Eh versus pH stability field diagrams for iron and manganese ($a_{Fe,Mn}$ = 10^{-4} M; PC_{O2} = 0; P tot = 1 atm; T = 25°C) (from Jenne, 1968).

hydroxides are generally absent in bogs ($\bar{x}$ = 6 ppm Mn) which explains the absence of uranium in this fraction. Despite the relative abundance of amorphous manganese hydroxides in lake and stream sediments ($\bar{x}$ = 65 ppm Mn in amorphous manganese hydroxides) this fraction retains insignificant proportions of total uranium (<1%, Map 2, Appendix VI, Table 20).

The pH of superjacent waters in Streams 1 and 2 range from 6.4 to 7.6 and more than 79% of the total uranium in sediments occurs in the organic matter fraction (Map 2, Appendix VI, Table 20). The bulk of metal in these sediments is in the fulvic acid component of organic matter according to prevailing pH (Figure 3). The partitioning characteristics of uranium in Stream 3 sediments vary considerably. In samples 58 to 61 and 66 to 68, the pH of superjacent waters ranges from 5.9 to 6.1 and organic matter retains up to 98% of the total uranium (Map 2, Appendix VI, Table 20). Based on the prevailing pH the bulk of metal must be in the humic acid component (Figure 3). In samples 62 to 65 water pH ranges from 6.2 to 6.3 and up to 33% of the total uranium in sediments occurs in detrital minerals which include grains of feldspars, micas and quartz (Map 2, Appendix VI, Table 20). In comparison to other stream sediments, low proportions of uranium occur in organic matter in these samples (up to 77%).

In the present study, organic matter retains significant amounts of uranium in lake, stream and bog sediments whereas amorphous iron hydroxides and detrital minerals are only important in lake and stream sediments respectively. It may be important in geochemical exploration to examine metal concentrations in a fraction which is common to all sedimentary environments in order to minimize the effects that variations in overall sediment composition may have on metal concentrations. Therefore uranium concentrations in organic matter and the effects of pH have been investigated in light of their potential significance to exploration. On a graphic plot of uranium concentrations in organic matter versus pH, data from the present study concentrates in isolated groups which correspond to specific pH and sedimentary environments (Figure 6). Uranium concentrations in organic matter generally are relatively low between pH 5.8 and 6.4 and are relatively high where pH is less than 5.8 and greater than 6.4. This trend generally corresponds to the trend established from plotting proportions of uranium in organic matter versus pH (Figure 3). Where pH is less than 5.8 and greater than 6.4, large proportions of total uranium in sediments are in organic matter and therefore relatively high concentrations in organic matter might also be expected. Most samples in this pH range contain between 75 and 150 ppm uranium in organic matter and include many of the sediments from Lake 1 and Bog 2 (Figure 6). Those not included in the main groups of data are samples 7 and 9 from Lake 1 and 31, 32, 35 to 38 from Bog 2. These are located adjacent to the uranium mineralized zone and contain between 162 and 1845 ppm uranium in organic matter (Figure 7). Between pH 5.8 and 6.4 relatively low proportions of total uranium are in organic matter (Figure 3). Most samples within this pH range contain between 5 ppm and 75 ppm uranium in organic matter and include many of the samples from Lakes 2 and 3, Bog 1 and Streams 1, 2 and 3 (Figure 6).

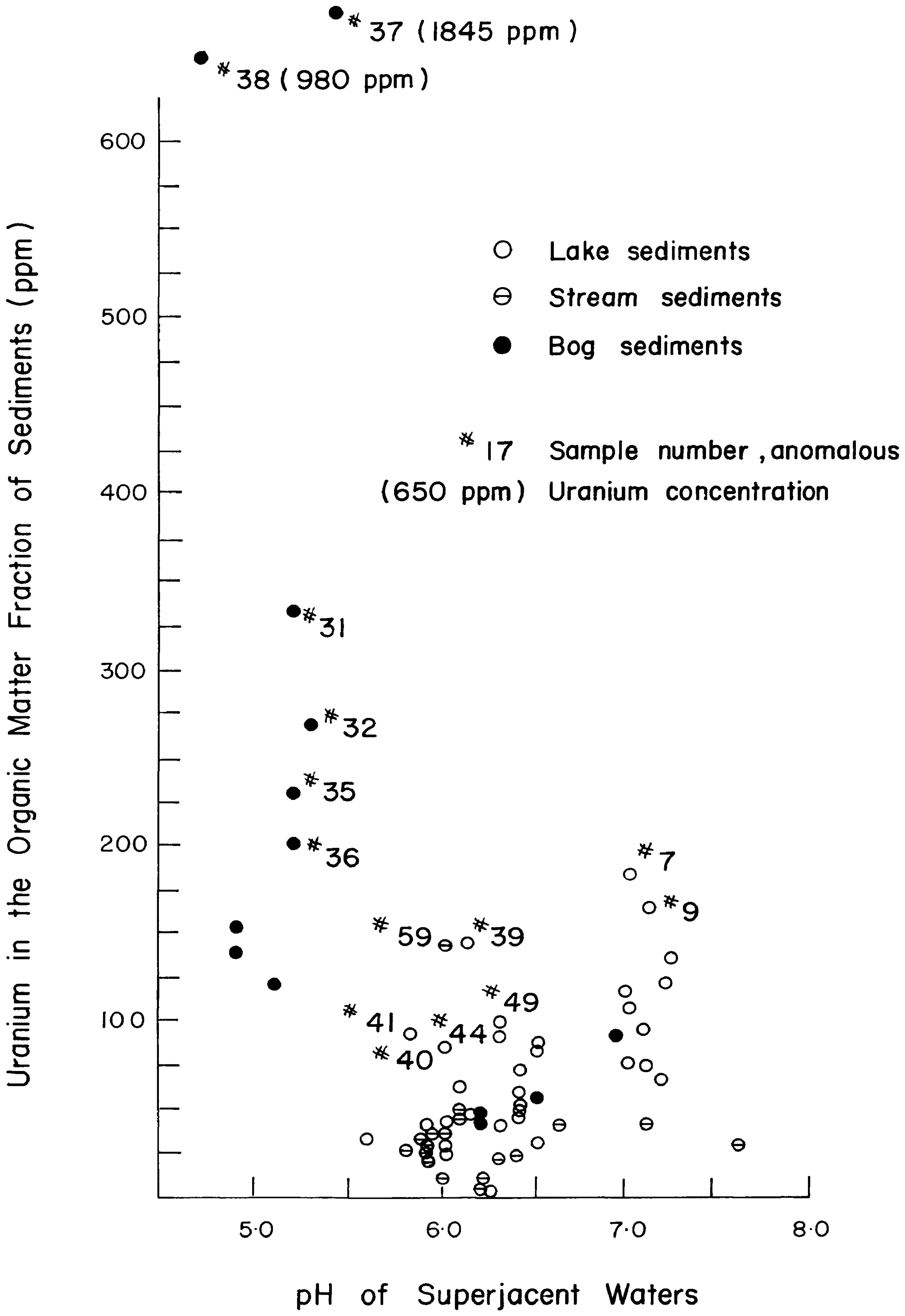

Figure 6 Concentration of uranium in the organic matter of sediments versus the pH of superjacent waters

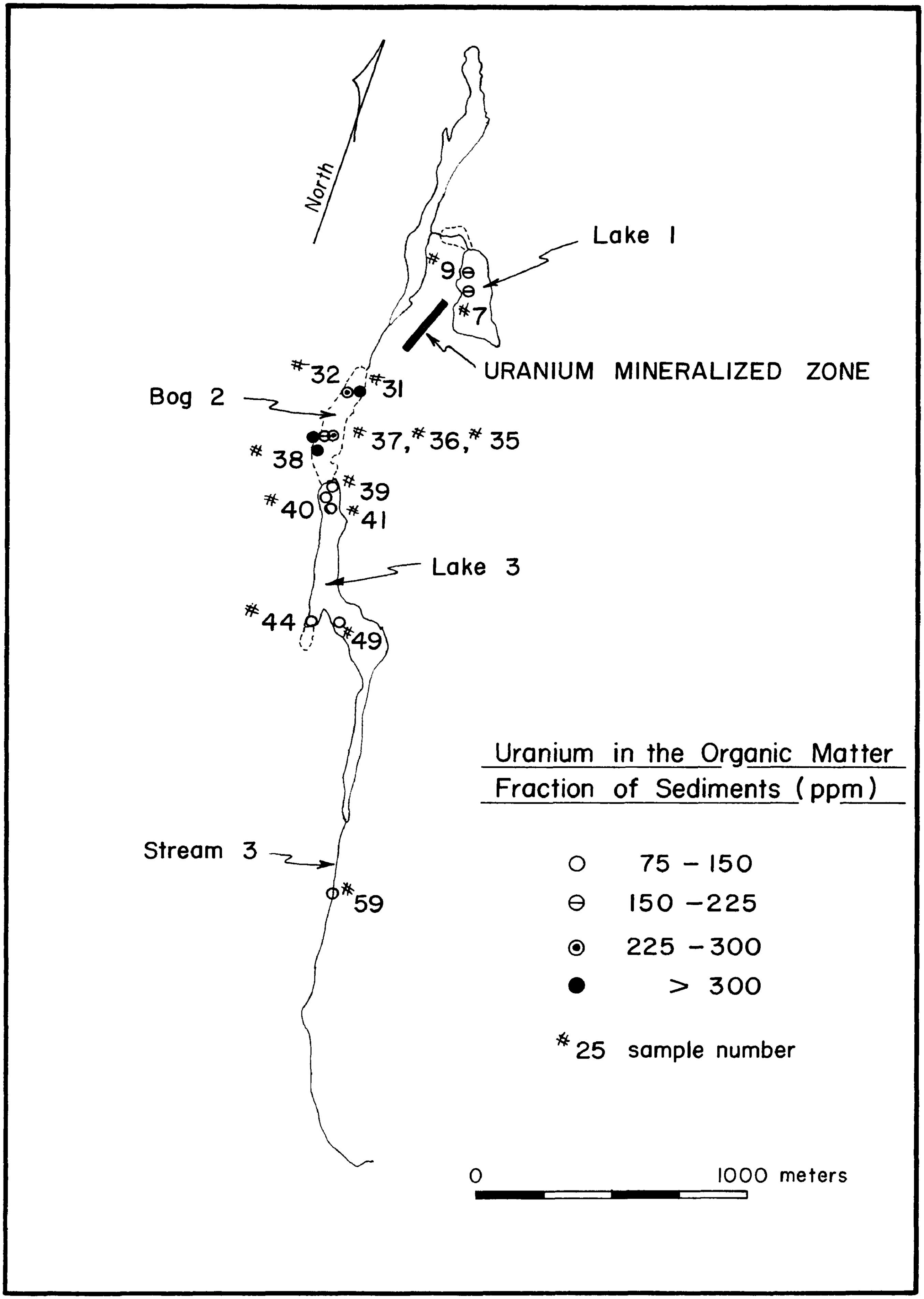

Figure 7 Locations of sediment samples containing potentially anomalous concentrations of uranium in the organic matter fraction with reference to the uranium mineralized zone located between Lake 1 and Bog 2

Samples not included in the main group of data are 39 to 41 and 44 from Lake 3 which contain between 90 and 143 ppm uranium in organic matter (Figure 6). These samples are from locations adjacent to and down-drainage of Bog 2 which may account for their high metal content in organic matter (Figure 7). Sample 49 from Lake 3 (99 ppm uranium in organic matter) and 59 from Stream 3 (145 ppm in organic matter) also plot above the main group of data. These samples are considerable distance from the known source of mineralization and may reflect a presently undiscovered source of uranium (Figure 7).

7.2.2 Copper

In the present study, copper shows a strong preference for partitioning into the organic matter fraction of sediments and the proportion of copper in this phase is directly dependent on the percent carbon in sediments (Figure 4). In local bogs up to 47.4 percent carbon occurs in centre-basin sediments and a maximum of 80% of total copper in sediment is in the organic matter fraction (Map 3, Appendix VI, Table 19 and 21). Percent carbon values in lake sediments range up to 39.7% and proportions of total copper in organic matter range up to 65%. Organic matter retains up to 64% of the total copper in some stream sediments that contain up to 32.9 percent carbon. In sediments with lower percent carbon, other fractions retain significant amounts of copper. Amorphous iron hydroxides are stable throughout the study area according to prevailing Eh-pH conditions and retain up to 29% of the total copper in near-shore bog sediments, up to 68% of the total copper in near-shore lake sediments and up to 55% of the total copper in some stream sediments. Detrital minerals constitute a significant proportion of most stream sedimens and up to 52% of the total copper remains in this fraction. Although detrital minerals are not as common in lake and bog sediments this fraction retains up to 24% and 37% respectively of the total copper in near-shore locations. Despite the presence of amorphous manganese hydroxides in lake and stream sediments, this fraction generally retains less than 17% of the total copper in these sediments.

The relationship between percent carbon and the actual copper concentration in organic matter of sediments has been investigated in view of its potential significance to geochemical exploration. For the present study, copper concentrations in organic matter are proportional to the percent carbon content in most sediments. On the basis of this relationship, most samples with a carbon content of 20% contain approximately 20 ppm copper in organic matter and so on. Exceptions are however observed and include samples 34 and 38 from Bog 2, 46 to 48 from Bog 3, 49 from Lake 3 and 59 from Stream 3 (Figure 8). Concentrations in the organic matter fraction of these sediments range between 45 and 100 ppm copper. These samples are confined to the geographic region about Lake 3 where biotite gneiss is the dominant bedrock type (Figure 9). Although copper mineralization is unknown in these rocks, relatively high concentrations of copper are common (up to 82 ppm copper, Table 1) which could account for elevated values in adjacent lake, stream and bog sediments.

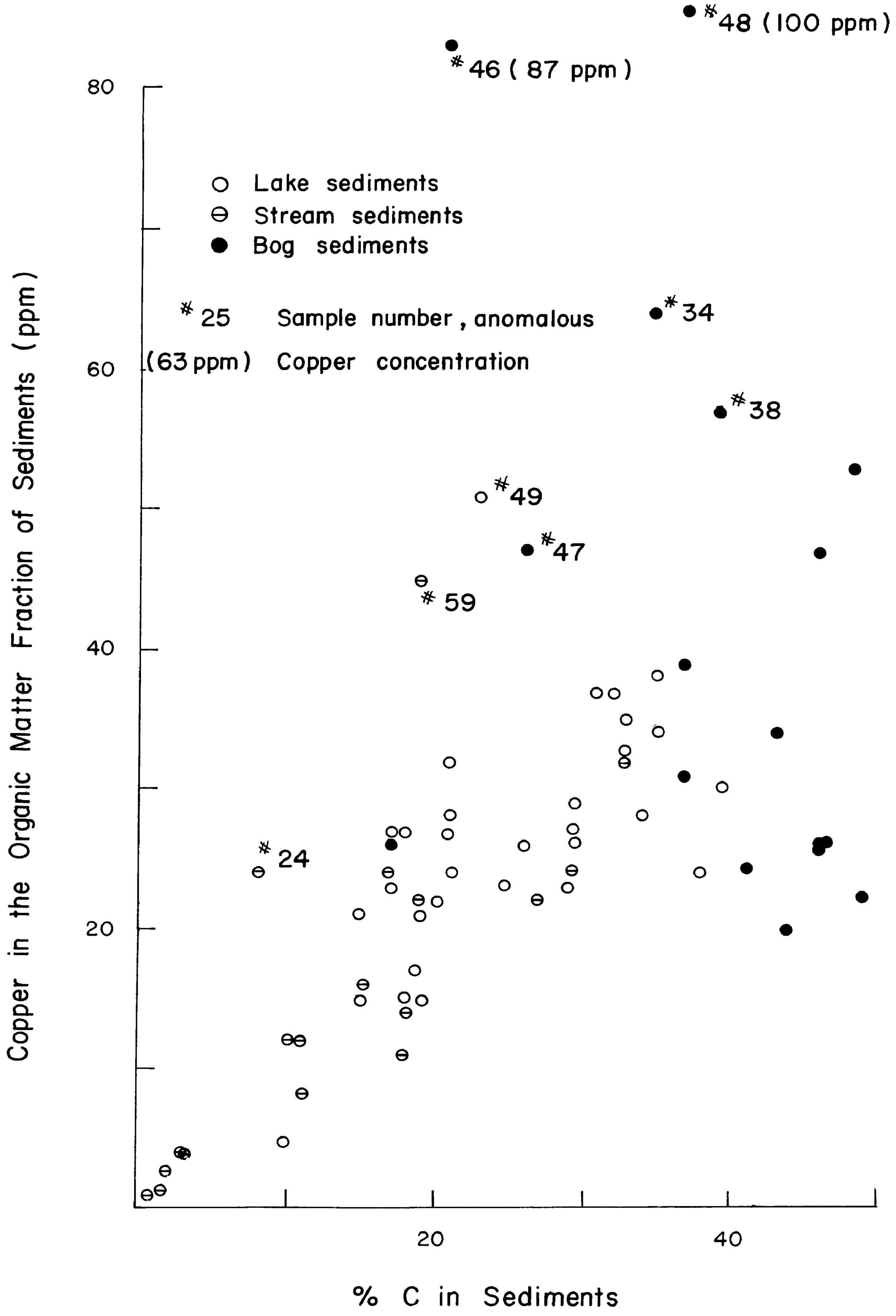

Figure 8 Concentration of copper in the organic matter fraction of sediments versus the percent carbon in sediments

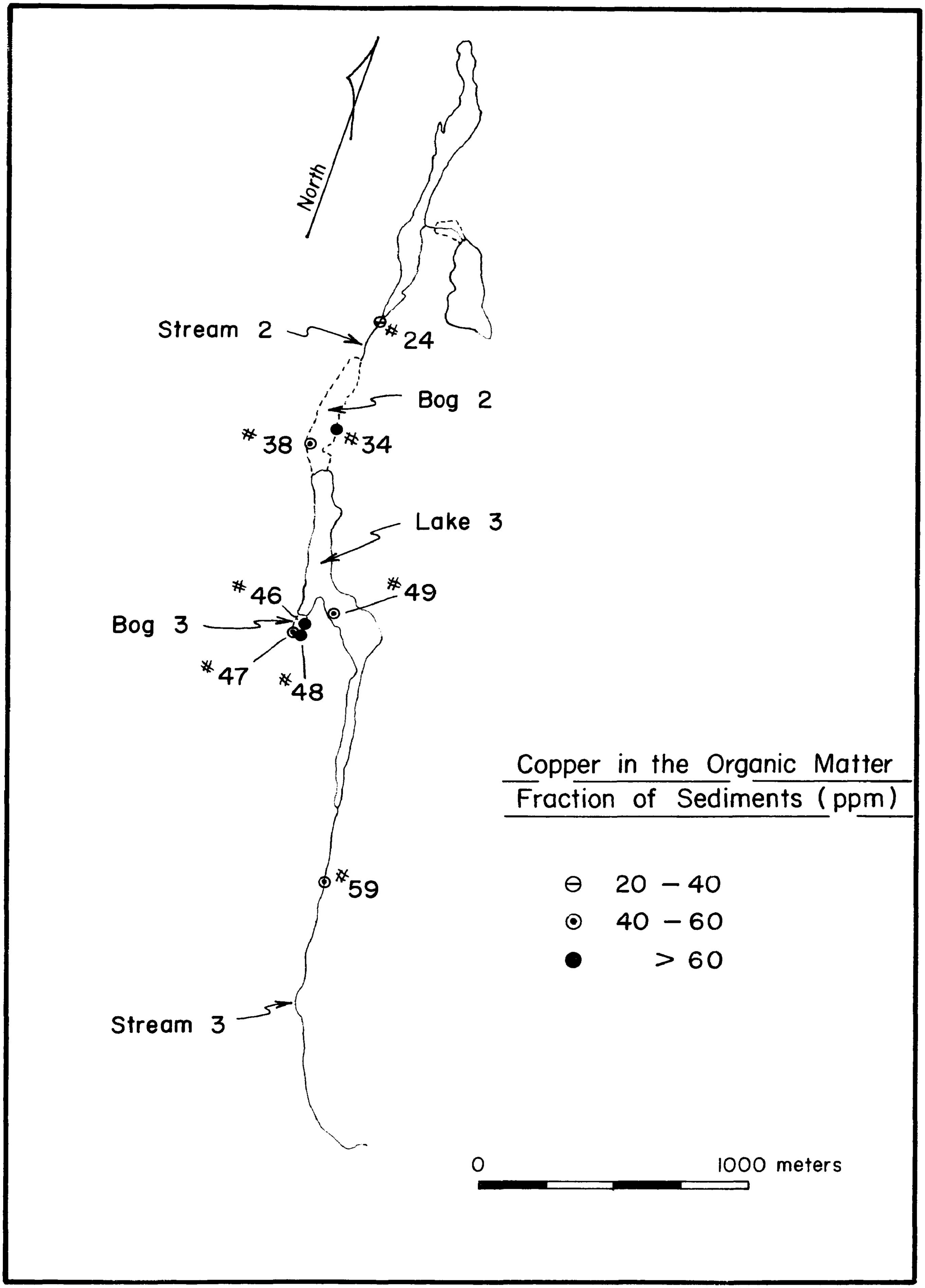

Figure 9 Locations of sediment samples containing potentially anomalous concentrations of copper in the organic matter fraction

7.2.3 Zinc

Zinc tends to partition into the component which is the most abundant in lake, stream and bog sediments and this varies considerably within and between each sedimentary environment. Amorphous iron hydroxides have been previously shown to be most abundant in near-shore inlet and outlet lake sediments where this fraction retains up to 68% of the total zinc (Map 4, Appendix VI, Table 22). Detrital minerals are also in relative abundance in near-shore sediments and retain up to 33% of the total zinc. In centre-lake basins organic matter is abundant and consequently up to 67% of the total zinc in these sediments is partitioned into this fraction. Organic matter also represents the dominant fraction in centre-bog sediments and retains up to 100% of the total zinc whereas in near-margin bog sediments up to 74% of the total metal is in detrital minerals. Amorphous iron hydroxides in bog sediments generally retain less than 21% of the total zinc in sediments even though this fraction is known to fix significant proportions of copper and iron. The physico-chemical conditions in local bogs may be restricting the availability of zinc to hydroxides in the same manner as manganese which does not occur as hydroxides in bogs because acidic pH makes this phase unstable. Detrital minerals are more common in streams than in lakes and bogs and up to 82% of the total zinc in stream sediments is retained in this fraction. Other fractions are relatively less abundant as indicated by the fact that organic matter retains a maximum of 49% and amorphous iron hydroxides a maximum of 59% of the total zinc in stream sediments.

If metal concentration data from all sedimentary environments are to be interpreted collectively in geochemical exploration, the application of zinc in hydroxide form is precluded since this phase is not present in bog sediments. In contrast organic matter and detrital minerals do retain significant proportions of zinc in lake, stream and bog sediments and therefore these fractions were considered to be of potential importance. Percent carbon in sediments influences the proportion of total zinc in organic matter (Figure 4). Therefore the effects of this factor on actual zinc concentrations have been investigated by plotting concentrations in organic matter versus percent carbon in sediments (Figure 10). Most of the bog sediments in the present study contain lower concentrations of zinc in organic matter than lake and stream sediments with the same percent carbon. For example, at 20.0% carbon, bogs contain approximately 8 ppm zinc in organic matter whereas lake and stream sediments contain approximately 15 ppm zinc. At 40.0% carbon bog sediments contain approximately 18 ppm zinc whereas lake and stream sediments approximately 34 ppm zinc in organic matter. Low concentrations of zinc in the organic matter fraction of bog sediments correspond to low proportions of zinc in hydroxides in these sediments and support the hypothesis that physico-chemical conditions in bogs restrict the general availability of zinc to sediments. In lake and stream environments restrictions on availability of zinc to sediments do not exist. This characteristic is of considerable importance to geochemical exploration. Although samples 28, 39, 44 and 49 contain high concentrations of zinc in organic matter (up to 66 ppm, Figure 11) they may not be

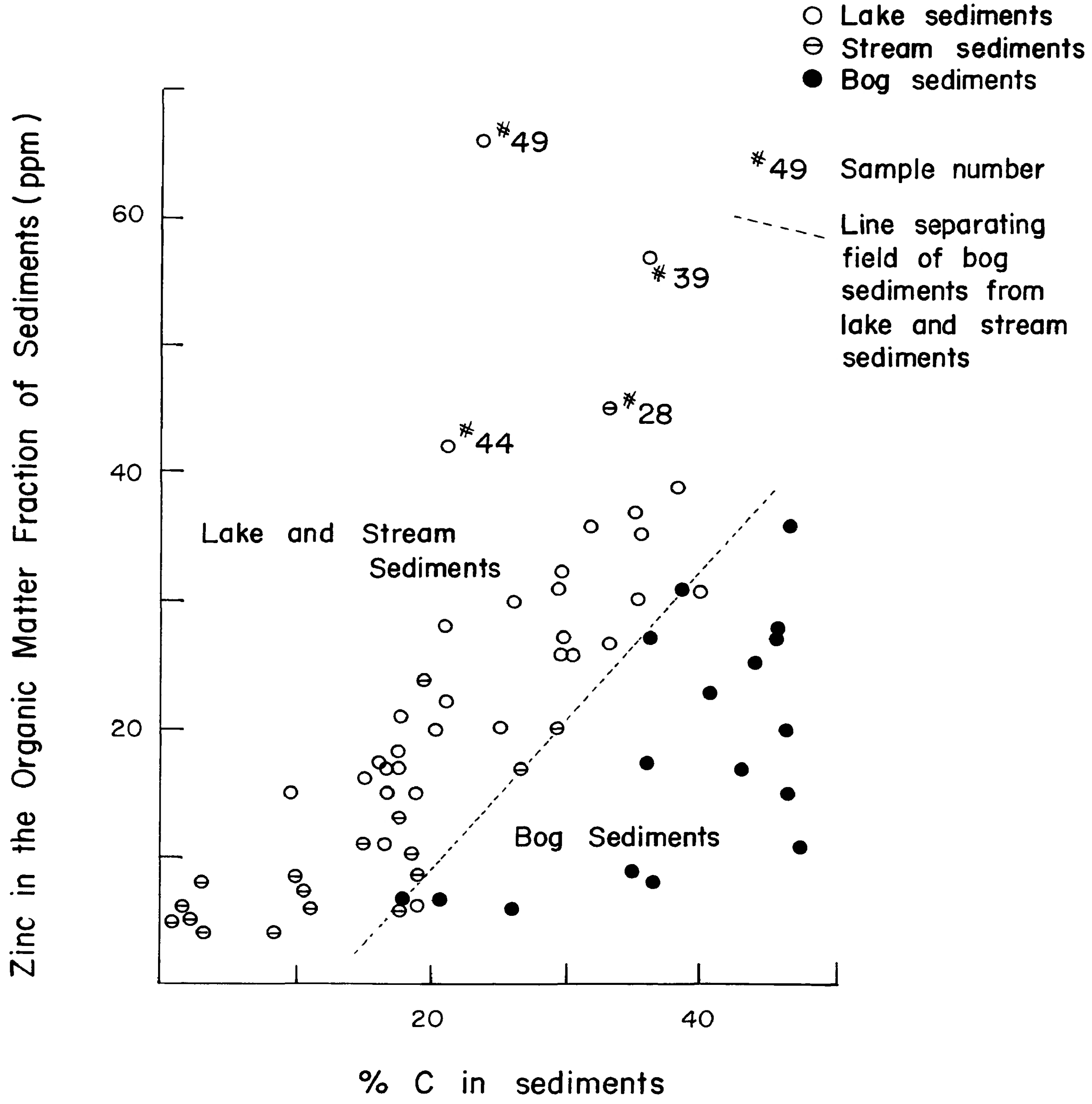

Figure 10 Concentrations of zinc in the organic matter fraction of sediments versus the percent carbon in sediments

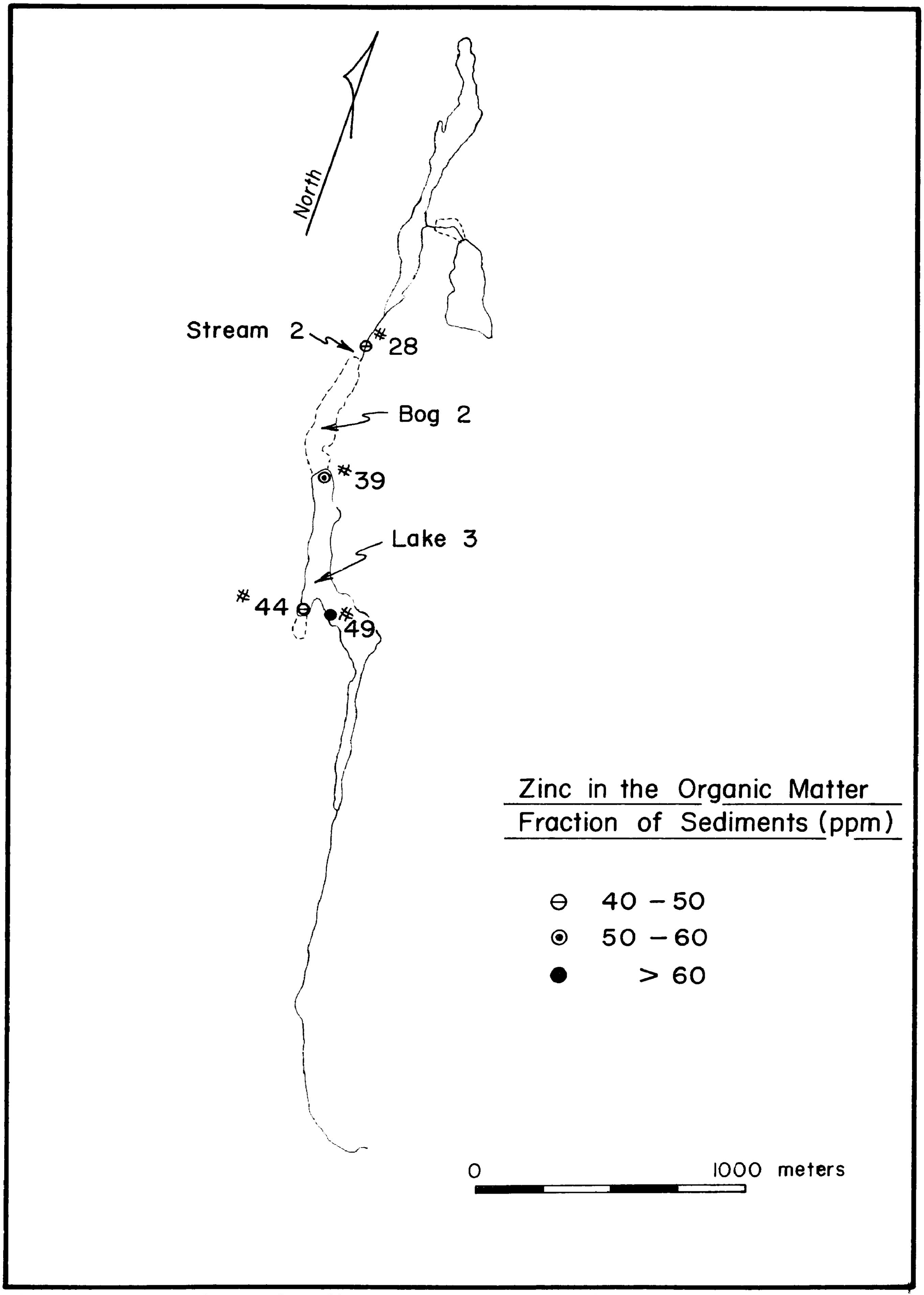

Figure 11 Locations of sediment samples containing potentially anomalous concentrations of zinc in the organic matter fraction

of much significance if the locations of the samples are considered. These sediments are from Stream 2 and Lake 3 and were collected adjacent to Bog 2 and 3, respectively (Map 4). High concentrations in organic matter in these sediments may therefore be attributed to the increased availability of zinc and not to a potential source of zinc mineralization. Since apparent anomalies of this type may occur only because zinc sorption occurs more readily into lake and stream sediments than into bog sediments, zinc concentrations in organic matter and/or hydroxides are of limited use in geochemical exploration. The only sediment fraction not affected by physico-chemical conditions is detrital minerals since this fraction and contained metals are of a primary nature. Zinc in detrital minerals could be of considerable importance to geochemical exploration since this fraction represents debris which is mechanically eroded from bedrock and soils. High zinc concentrations in this fraction could therefore be directly related to local mineralization. Specific examinations of detrital minerals have not been performed in this study which presently limits any interpretation regarding their potential significance to geochemical exploration.

7.2.4 Iron and Manganese

Iron and manganese are commonly analyzed for in geochemical exploration because many metals illustrate an association to iron and manganese (Coker, 1974). Sequential partial extractions indicate this association to be due to partitioning of metals into the amorphous-iron-manganese hydroxide fractions of sediments. The stability of iron and manganese hydroxides in sediments has been shown to be influenced by Eh-pH conditions in superjacent waters. Whereas iron is stable as hydroxides in lake, stream and bog sediments of the present study, manganese hydroxides are only stable in lake and stream sediments. Although significant proportions of total manganese in lake and stream sediment occur as amorphous manganese hydroxides, this fraction generally retains low proportions of uranium, copper, zinc and iron (Maps 2-6, Appendix VI, Tables 20-24). In contrast, amorphous iron hydroxides retain significant proportions of metals in lake, stream and bog sediments (Maps 2-6, Appendix VI, Tables 20-24). The limited role of manganese hydroxides in metal partitioning into lake, stream and soil sediments has been previously reported from the Nechako Plateau region, central British Columbia (Hoffman and Fletcher, 1978). Other fractions of sediments that contain iron and manganese include organic matter particularly in bog sediments and detrital minerals in stream sediments. Partitioning into organic matter and occurrence in detrital minerals is primarily influenced by the relative abundance of these fractions.

CHAPTER 8

CONCLUSIONS AND RECOMMENDATIONS

The present study has shown that partitioning of uranium, copper, zinc, iron and manganese are affected by the nature of component fractions within sediments and by the physico-chemical conditions in superjacent waters. In the present study important sediment fractions include organic matter, amorphous iron hydroxides and detrital minerals whereas carbonate minerals, amorphous manganese hydroxides and crystalline iron hydroxides are of limited significance. Important physico-chemical parameters include pH and Eh of the waters and percent carbon in the sediments.

Uranium shows a strong preference for partitioning into the organic matter fraction of lake, stream and bog sediments. The pH of superjacent waters affects the actual concentration of uranium in the organic matter fraction of sediments and dictates the relative proportion of metal partitioned into the humic and fulvic acid components of organic matter. The largest proportion of total uranium occurs in humic acid of sediments where pH in superjacent waters is less than 5.8. These pH conditions typically prevail in peat bogs and in bays of lakes situated adjacent to and down-drainage of bogs. In environments where pH is greater than 6.4, the largest proportion of total uranium in sediment is contained in fulvic acid. These conditions typically prevail in centre-lake basins and streams. The actual concentration of uranium in organic matter is high where pH is less than 5.8 or greater than 6.4 since under these conditions the combined humic and fulvic acids have high capacities to sorb metal. Between pH 5.8 and 6.4 sorption capacities for humic and fulvic acids are low and as a consequence actual concentrations of uranium in these acids are relatively low. Although the largest proportion of uranium in sediment is still in organic matter, significant amounts of metal are partitioned into other fractions. In near-inlet and outlet lake sediments, where pH ranges from 5.8 to 6.4 partitioning occurs into amorphous iron hydroxides whereas in stream sediments significant amounts of uranium remain in detrital minerals.

Copper also shows a strong preference for the organic matter fraction in lake, stream and bog sediments. The proportion and concentration of metal in this fraction increases proportionally as percent carbon in sediments increase and so percent carbon is considered to be the principle factor to influence copper partitioning. Stream sediments and near-inlet and outlet bog and lake sediments typically have low percent carbon and therefore low amounts of copper in organic matter. Amorphous iron hydroxides and detrital minerals are in relative abundance and fix, or contain significant amounts of copper in these sediments. Centre-lake and bog basin sediments have high percent carbon and as a result there are high proportions and concentrations of copper in organic matter and low proportions in detrital minerals and amorphous iron hydroxide fractions.

The Eh-pH conditions of superjacent waters control the solubilities of iron, manganese and zinc and thereby directly affect the availability and sorption of these metals into the organic matter and inorganic hydroxide fractions of sediment. In the present study, Eh-pH conditions ensure the availability of iron to sediments in all environments. The proportion and concentration of iron in individual sediment fractions is determined by their relative abundance. Although the largest proportions of iron in bog sediments is partitioned into organic matter, iron is also partitioned to form amorphous iron hydroxides and also occurs as detrital minerals particularly in near-shore sediments. In lake sediments amorphous iron hydroxides are in abundance and typically fix the largest proportion of iron with smaller amounts occurring in detrital minerals. The amount of iron in organic matter in lake sediments is negligible. Detrital minerals are common in stream sediments and contain considerable concentrations of iron. In contrast, relatively low amounts of iron are found in amorphous iron hydroxides and insignificant amounts in the organic matter fraction of stream sediments. The Eh-pH conditions in local bogs determine that zinc and manganese are stable in solution and therefore are not available for sorption into sediments. Since partitioning into the organic matter and/or hydroxide fractions is restricted, total concentrations in sediment are relatively low. In lakes and streams there are no Eh-pH restrictions on the availability of zinc and manganese and total metal concentrations in sediment are relatively high. Partitioning of these metals into organic matter, hydroxides and occurrence in detrital minerals is determined by the relative abundance of these sediment fractions. Manganese occurs in relative abundance in amorphous manganese hydroxides in lake and stream sediment. However, despite its presence, this fraction typically retains insignificant proportions of uranium, copper, zinc and iron.

The application of sediment sampling to the geochemical exploration for zinc, manganese and iron is precluded by the fact that Eh-pH conditions in superjacent waters affect the amount of these metals available to complex with most sediment fractions. The only fraction unaffected by physico-chemical conditions in the superjacent water is the detrital mineral fraction since it is of a primary nature. Metal concentrations in detrital minerals could be of considerable importance to geochemical exploration. Since this fraction represents mechanically eroded debris from bedrock and soils, high metal concentrations in this fraction could be related to local mineralization. Specific examinations of detrital minerals has not been performed during the present study but considering its potential significance such investigations are recommended as part of future studies. The examination of mineralogy and weight percent might provide valuable data concerning metal associations based on abundance and/or specific mineral types present in sediments.

Recommendations are proposed concerning the use of lake, stream and bog sediments in geochemical exploration for uranium and copper. Procedures have been based on partitioning characteristics and physico-chemical factors which influence metal partitioning in the present study. Recommendations are considered to be of a preliminary nature and their final acceptance hinges on further testing in other geologic - geographic environments.

Recommendations for uranium exploration are as follows:

1. The pH of superjacent waters should be routinely measured in conjunction with sampling of lake, stream and bog sediments.

2. Samples should be treated with sodium hypochlorite to selectively extract organic matter. Uranium concentrations in this fraction can be determined by standard fluorimetric techniques.
3. Uranium concentrations in organic matter of sediments should be interpreted with regard to three pH ranges in superjacent waters ($pH < 5.8$, $5.8 \leq pH \leq 6.4$, $p > 6.4$). This can be carried out by a graphic plot of uranium in organic matter versus pH. Concentrations will generally be highest in samples where pH is less than 5.8 and greater than 6.4 and comparatively low in samples where pH is between 5.8 and 6.4. In graphic analysis potentially important samples in each pH range will plot above the bulk of data.

Recommendations for copper exploration are as follows:

1. Lake, stream and bog sediment samples should be treated with sodium hypochlorite to selectively extract organic matter. Copper concentrations in this fraction can be determined by standard atomic adsorption spectrophotometric techniques.
2. Percent carbon should be determined on the whole sediment.
3. Copper concentrations in organic matter should be interpreted with regard to percent carbon in sediments. This can be done on a graphic plot of copper in organic matter versus percent carbon. Copper concentrations in organic matter are generally proportional to the percent carbon content of sediments. On the basis of known percent carbon values, copper concentrations in organic matter can be predicted. Potentially important samples may occur where actual concentrations exceed predicted concentrations.

Locations of samples containing high concentrations of copper and uranium can be recorded. The occurrence of samples within a confined geographic area(s) indicate targets that may be worthy of further investigation. By applying the recommended procedures, uranium and copper targets have been defined in lake, stream and bog sediments of the present study that correspond in location to known sources of metal in bedrock. Uranium targets reflect uranium mineralization in pegmatite that outcrops adjacent to and intersects Lake 1 and Bog 2 whereas copper targets reflect copper-bearing biotite gneiss in the vicinity of Lake 3. Consequently these procedures are considered to be of potential importance to geochemical exploration.

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APPENDIX I

CHRONOLIGIC BIBILIOGRAPHY OF PREVIOUS PAPERS REPORTING THE USE OF PARTIAL AND SEQUENTIAL PARTIAL EXTRACTIONS

Year	Author(s)	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1916	Gortner	Soils	organic matter	hydrochloric acid, sodium hydroxide		general investigation of nature of soil organic matter
1919	Oden	Soils	fulvic-humic acids, humin fraction of soil organic matter	sodium hydroxide	-	general investigation of nature of soil organic matter
1924	Eden	Soils	humic acid	hydrochloric acid, sodium hydroxide	-	colorimetric analysis of humus
1924	Robinson & Holmes	Soils	iron oxides	oxalic acid	-	general investigation of iron oxides
1931	Troell	Soils	organic matter	sodium hypobromite, sodium hydroxide	-	analysis of mineral colloid constituents of organic matter
1935	Drosdoff	Soils	iron oxides	sodium acid oxalate	-	
1942	Allison & Scarseth	Soils	iron oxides	sucrose		general investigation of iron oxides
1946	Marshall & Jeffries	Soils	iron oxides	oxalic acid, hydrochloric acid	-	
1949	Bremner & Lees	Soils	organic matter	sodium pyrophosphate	-	examination of electrophoretic properties and hydrolysis products of organic matter
1949	Deb	Soils	iron oxides	sodium dithionite	-	application of dithionite to estimate free iron oxide in soils
1950	Schofield	Soils	iron oxides	oxalic acid	-	general investigation of the nature of soil iron oxides
1953	Aguilera & Jackson	Soils	iron oxides	sodium dithionite, citrate, bicarbonate	-	removal of iron oxides observed to increase the sensitivity of x-ray diffraction analysis

Year	Author(s)	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1954	Mitchell & McKenzie	clay soils	iron oxides	sodium hydrosulphite	-	details extraction method and effects of pH and temperature
1956	Jackson	soils	carbonates, organic matter, iron oxides	sodium acetate, hydrogen peroxide, sodium dithionite, citrate bicarbonate	-	removal of carbonates organic matter and iron oxides observed to enhance x-ray diffraction analysis
1957	White	soils	iron oxides, clay silicate minerals, carbonates	sodium dithionite, citrate bicarbonate, ammonium acetate	Zn	zinc association in clay mineral lattices and iron oxides
1958	Goldberg & Arrhenius	marine sediments	iron-manganese oxide minerals	ethyl diamine tetraocetate (EDTA)	Na,K,Mg,Ca, Sr,Ba,B,Si, Al,Y,Ti,V, Cr,Cu,Fe, Mn,Ni,Pb,Mo	removal of readily soluble phases of pelagic sediments and study of metal content of these components
1958	Schnitzer et al	soils	organic matter sodium	pyrophosphate hexametaphosphate borate fluoride chloride bromide iodide carbonate hydroxide hydrochloric acid, hydrofluoric acid EDTA		to define conditions of maximum extraction of organic matter by comparing reagents
1960	Mehra & Jackson	clay soils	iron oxides	sodium dithionite citrate bicarbonate	-	report on effectiveness of extraction technique
1963	Coffin	clays	iron oxides	sodium citrate sodium hydrosulphite	-	report on extraction method
1963	deEndredy	soils	iron oxides	acid ammonium oxalate	-	one of first reports on extraction method using ammonium oxalate

Year	Author	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1963	Anderson	soils	organic matter	sodium hypochlorite	-	one of preliminary reports on use of sodium hypochlorite to remove organic matter
1963	LeRiche & Weir	soils	iron oxides	ammonium oxalate	Co,Cr,Cu, Ga,Mn,Ni, Pb,Sr,Ti, V,Zr	trace metal analysis in iron oxide component of soils
1966	McKeague & Day	soils	iron oxides	dithionite oxalate	Fe,Al	extraction of Fe and Al in iron oxides as an aid in differentiating classes of soil extraction carried out in darkness
1967	Chester & Hughes	marine sediments	carbonate minerals, manganese oxides, silicate minerals	acetic acid, hydroxylamine, hydrochloride, nitric acid	Fe,Mn, Ni,V	partitioning of trace elements among component fractions of marine sediments
1967	McKeague	soils	iron organic complexes, amorphous iron inorganic complexes, crystalline iron complexes	sodium pyrophosphate, oxalate dithionite	-	comparison of methods to remove iron organic and inorganic complexes
1969	Kemp	lake sediments	bitumen, humic-fulvic acids, Kerogen	benzene, methanol, acetone, sodium hydroxide, sodium pyrophosphate, perchloric acid	-	study of organic matter
1970	Lavkulich & Weins	soils	organic matter	sodium hypochlorite, hydrogen peroxide	-	comparison of effectiveness of hypochlorite and peroxide
1971	Zwarich & Mills	soils	carbonates	sodium acetate, sulphuric acid	-	comparison of effectiveness of sodium acetate and sulphuric acid

Year	Author	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1972	Chao	soils	manganese oxides	hydroxylamine hydrochloride	-	details extraction procedure for removal of Mn oxides
1972	Gamble & Daniels	soils	iron oxides	acid ammonium oxalate, dithionite	Fe,Si	partitioning of metals in iron oxides
1972	Presley et al	marine sediments	carbonates, iron manganese oxides, organic matter, silicates	acetic acid, hydroxylamine hydrochloride, hydrogen peroxide, hydroflouric nitric perchloric acids		study of component fractions of marine sediments
1972	Schnitzer & Khan	soils	humic-fulvic acid, humin fraction	sodium hyponitrate		details extractive technique
1973	Baker	soils	humic acid	sodium hyponitrate	Cu,Zn,Pb, Fe,Bi,Sb, Ni,Co,Ba	role of humic acid in metal migration
1973	Gibbs	river sediments	exchangeable, organic matter, iron manganese oxides, silicates	magnesium chloride, sodium dithionite sodium hypochlorite, nitric acid	Fe,Ni,Co, Cr,Cu,Mn	partitioning of metals in river sediments
1974	Chao & Anderson	stream sediments	iron manganese oxides	dithionite, hydroxylamine hydrochloride	Ag	scavenging of Ag by iron manganese oxides
1975	Schaef	lake sediments	humic-fulvic acid, humic components	sodium hyponitrate	Cu,Pb,Zn, Mn	partitioning of elements between organic phases of lake sediments
1976	Chao & Theobold	soil and stream sediments	amorphous & crystalline iron manganese oxides, sulphides, silicates	hydroxylamine hydrochloride, sodium dithionite, potassium perchlorite, hydrochloric nitric acid, hydroflouric nitric acid	Co,Ag	reports on method to fractionate 5 phases of iron manganese components 1) amorphous Mn oxides 2) amorphous Fe oxides 3) crystalline Fe " 4) sulphides 5) silicates

Year	Author	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1976	Grieve & Fletcher	deltaic sediments	carbonate, iron oxides, silicates	magnesium chloride, hydroxylamine, hydrochloride, nitric perchloric acid	Co,Cu,Fe, Mn,Ni,Pb, Zn	partitioning of metals in component fractions of sediments
1976	Meineke & Klaysmat	soils and lake sediments	silicates, sulphides, organic matter, clay minerals, Fe-Mn hydroxides	nitric acid (hot & cold) nitric-hydrochloric acid (hot & cold) EDTA, ammonium citrate, hydroxylamine hydrochloride, ascorbic acid, hydrogen peroxide	Cu,Ni,Zn	extractability of metal by various digestion methods
1977	Gatehouse et al	soils	carbonates, Mn hydroxides, organic matter, iron hydroxides, silicates	ammonium acetate, hydroxylamine hydrochloride, hydrogen peroxide, hydrazine chloride, hydrochloric nitric, hydroflouric acid		
1977	Gibbs	stream sediments	exchangeable, iron manganese oxides, organic matter, silicates	magnesium chloride, sodium, dithionite, sodium hypochlorite, nitric acid	Cr,Mn,Fe, Co,Ni,Cu	partitioning of trace elements in stream sediments
1977	Huang et al	soils	carbonate, organic matter, iron-manganese oxides	sodium acetate, hydrogen peroxide, sodium dithionite, ammonium oxalate	Si,Al,Fe	partitioning of trace elements in soils
1978	Carpenter et al	stream sediments	iron manganese oxides		Mn,Fe,Cu, Zn,Pb,Co, Ni	partitioning of trace elements in streams and application to exploration

Year	Author	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1978	Dijkstra et al	stream sediments	iron manganese oxides	hydroxylamine hydrochloride, sodium dithionite citrate	Zn	
1978	Hoffman & Fletcher	soil, stream, lake sediments	organic matter, carbonate, Mn oxides, Fe oxides (amorphous), Fe oxides (crystalline), silicates	sodium hypochlorite, hydrochloric acid, hydroxylamine hydrochloride, acid ammonium oxalate, sodium dithionite, nitric perchloric acid	Cu,Zn,Mo, Fe,Mn	partitioning in sediments and application to exploration
1978	Huang	lake sediments	carbonate, organic matter, Fe-Mn sesquioxide	ammonium acetate, hydrogen peroxide, citrate dithionite bicarbonate		
1979	Huang & Liaw	lake sediments	carbonate, organic matter, Fe-Mn sesquioxide	ammonium acetate, hydrogen peroxide, citrate dithionite bicarbonate	As	study of distribution and fractionation of Arsenic Precambrian and Prairie Lakes
1979	Tessier et al	stream sediments	exchangeable, carbonate, iron manganese oxides, organic matter, silicates	magnesium chloride, sodium acetate, sodium sulphate, sodium citrate, hydroxylamine hydrochloride, hydrogen peroxide, hydroflouric-perchloric acid	Cd,Co,Cu, Ni,Pb,Zn, Fe,Mn	partitioning in stream sediments environmental impact
1980	Bogle	stream and lake sediments	organic matter, manganese hydroxides, amorphous iron hydroxides, crystalline hydroxides, silicates	sodium hypochlorite, hydroxylamine, hydrochloride, nitric acid	Cu,Pb,Zn, Fe,Mn,Cu, Mg	

Year	Author	Type of Sediment(s) Examined	Component Fraction(s) Extracted	Reagent(s) Used	Elements Analyzed	Scope of Study
1980	Sopuck, Lehto et al	lake sediments	organic matter, carbonate, manganese oxides (amorphous) iron oxides (amorphous) iron oxides (crystalline), silicates	sodium hypochlorite, hydrochloric aicd, hydroxylamine, hydrochloride acid ammonium oxalate, hydrazine chloride, hydroflouric-nitric-hydrochloric acid	Cu,Ni,Zn, U,Pb,Fe, Co,As	
1980	Sopuck, Lehto et al	lake sediments			Cu,Zn,Ni, Co,U,Pb, Fe	

APPENDIX II

DESCRIPTION OF EXTRACTION PROCEDURES

APPENDIX II

I. SEQUENTIAL PARTIAL EXTRACTION PROCEDURES

Treatment #1

Sodium Hypochlorite Treatment to Dissolve Organic Matter - Metal Complexes

References (Anderson, 1963; Lavkulich and Wiens, 1970; Hoffman and Fletcher, 1978)

Reagents Required:

500 ml of reagent grade sodium hypochlorite freshy adjusted to pH 9.5 with 6M hydrochloric acid.

Procedure:

Weigh exactly 1.000g of sample into a 50 ml nalgene centrifuge tube. Add 20 ml sodium hypochlorite and stir. Allow the sample to sit for 4 hours homogenizing it at 2 hour intervals. Place the tube in a preheated waterbath at 80°C for 3 hours to decompose excess hypochlorite. Centrifuge the tube at 2400 rpm for 10 minutes. Pour off the extract into a 100 ml volumetric flask. Repeat the above extraction twice. This will give 60 ml of extract. Dilute the extract to 100 ml with H_2O. Save the extract for analysis. Wash the residue using 20 ml of H_2O. Shake and centrifuge solution for 5 minutes at 2400 rpm. Pour off and discard the H_2O. Repeat the washing once. Save the residue for the next treatment.

Comments:

In very organic-rich sediments addition of hypochlorite is commonly followed by a vigorous reaction which may result in sample loss. In these samples it is therefore recommended that hypochlorite be added in two aliquots of 10 ml each. Extractant solutions are commonly dark brown after the first extraction and yellow after the end of the third. Spectral interference in sodium hypochlorite extrants hampers copper analysis by atomic adsorption spectrophotometry. Contamination produces an apparent 25 ppm copper concentration in blank hypochlorite solution. Copper concentration in solutions containing sample must therefore be computed as the difference between values detected in sample solutions and values detected in blank sodium hypochlorite (Appendix II (III and IV)).

Treatment #2

Water-Hydrochloric Acid Treatment to Dissolve Carbonate, Clay-Metal Complexes

Reference (Hoffman and Fletcher, 1978)

Reagents Required:

1 litre distilled water acidified to pH 3.0 with 1.5M hydrochloric acid.

Procedure:

Add 20 ml distilled water - HCl solution to the residue from Treatment #1. Agitate and allow solution to sit for 30 minutes. Centrifuge tube for 10 minutes at 2400 rpm. Pour off liquid into a 100 ml volumetric flask. Repeat the extraction twice. This will give 60 ml of extract. Dilute the extract to 100 ml with H_2O. Save the extract for analysis. Wash the residue using 20 ml H_2O. Shake and centrifuge solution for 5 minutes at 2400 rpm. Pour off and discard the H_2O. Repeat the washing once. Save the residue for the next treatment.

Treatment #3

Hydroxylamine Hydrochloride Treatment to Dissolve Amorphous Manganese Hydroxide - Metal Complexes

References (Chester & Hughes, 1967; Chao, 1972; Gatehouse et al, 1977; Hoffman and Fletcher, 1978)

Reagents Required:

Dissolve 3.47 g of hydroxylamine hydrochloride salt in 500 ml of distilled water. Adjust the pH of the solution to 3.0 with the 1.5M hydrochloric acid used in Treatment #2.

Procedure:

Add 20 ml of hydroxylamine hydrochloride solution to the residue from Treatment #2. Shake and allow the reaction to continue for 30 minutes agitating the sample every 10 minutes. Centrifuge the tube at 2400 rpm for 10 minutes. Pour off the extract into a 100 ml volumetric flask. Repeat the above extraction twice. This will give 60 ml of extract. Dilute the extract to 100 ml with H_2O. Save the extract for analysis. Wash the residue using 20 ml H_2O. Shake and centrifuge solution for 5 minutes at 2400 rpm. Pour off and discard the H_2O. Repeat the washing once. Save the residue for the next treatment.

Treatment #4

Acid Ammonium Oxalate Treatment to Dissolve Amorphous Iron Hydroxide - Metal Complexes

References (LeRiche and Weir, 1963; McKeague and Day, 1966;
Gamble and Daniels, 1972; Huang et al, 1977;
Hoffman and Fletcher, 1978)

Reagents Required:

Dissolve 24.9 g of ammonium oxalate monohydrate and 12.6 g of oxalic acid dihydrate in 1 litre of distilled water.

Procedure:

Add 20 ml of acid ammonium oxalate to the residue from Treatment #3. Allow the reaction to continue for 4 hours in darkness agitating the samples at 1/2 hour intervals. Centrifuge the tube at 2400 rpm for 10 minutes. Pour off the extract into a 100 ml volumetric flask. Repeat the above extraction twice. This will give 60 ml of extract. Dilute the extract to 100 ml with H_2O. Save the extract for analysis. Wash the residue using 20 ml H_2O. Shake and centrifuge solution for 5 minutes at 2400 rpm. Pour off and discard H_2O. Repeat the washing once. Save the residue for the next treatment.

Treatment #5

Hydrazine Chloride Treatment to Dissolve Crystalline Iron Hydroxide - Metal Complexes

Reference (Gatehouse et al, 1977)

Reagents Required:

Dissolve 100 g of 99% hydrazine hydrate in 1.8 litres H_2O. Adjust pH to 4.5 with conc. hydrochloric acid and then bring volume up to 2 litres with distilled water.

Procedure:

Add 10 ml of hydrazine chloride solution to the residue from Treatment #4. Heat sample in a preheated water bath at 90°C for 3 hours mixing periodically. Add another 10 ml of hydrazine chloride, return the sample to the water bath and leave overnight (8-12 hours). Centrifuge the sample at 2400 rpm for 10 minutes or until supernatant becomes clear. Pour off the liquid into a 100 ml volumetric flask. Repeat the above extraction twice. This will give 60 ml of extract. Dilute the extract to 100 ml and save for analysis. Wash the residue as outlined in treatments #1, 2 and 3. Save the residue for the next treatment.

Treatment #6

Hydrofluoric-Nitric-Perchloric Acid Treatment to Dissolve Residual Detrital Mineral - Metal Complexes

(also used as a total digestion procedure)

Reference (Tessier, 1979)

Reagents Required:

15:5:2 Hydrofluoric:Nitric:Perchloric acid solution, a 5% solution of Nitric acid, and concentrated hydrochloric acid.

Procedure:

Weight exactly 0.5000 g of thoroughly dried residue from extraction #5* into a 20 ml teflon beaker. Add a couple drops of 5% nitric acid and swirl the beaker to ensure that the sample is completely wetted. Fill the beaker with the solution of hydrofluoric:nitric:perchloric acid and set on an electric hot plate at 90°C for 8 to 12 hours. Raise the temperature of the hot plate to 150°C and allow the sample to thoroughly dry. In the final stages of drying, while perchloric acid fumes will be observed. Once these fumes disappear the sample is dry. Remove samples from the heat, add 3 ml of concentrated hydrochloric acid and allow to simmer for 5 minutes. Fill remaining volume of beaker with de-ionized water and allow to simmer for 10 minutes. Transfer to volumetric flasks. Dilute to 100 ml and save for analysis.

*Note: where this procedure is used as method of total digestion the 0.5000 g portion of sample is not residue from extraction #5 but is instead a freshly weighed aliquot of whole sample.

II. PARTIAL EXTRACTION PROCEDURES TO SEPARATE HUMIC-FULVIC ACID COMPONENTS OF SEDIMENT ORGANIC MATTER

Reference (Schnitzer and Khan, 1972; Schaef, 1975)

Reagents Required:

0.5N sodium hydroxide, 6.0N and 1.0N hydrochloric acid, nitrogen gas

Procedure:

Weight exactly 10.0000 g of dried sample into a 250 ml centrifuge bottle. Add 100.0 ml of 0.5N sodium hydroxide and shake gently to dissolve or wet all sample. Bubble nitrogen gas through the solution for approximately 5 minutes capping the bottle immediately afterwards. Allow to stand for 18 hours shaking occasionally. Centrifuge the mixture at 2000 rpm for 30 minutes. Decant the solution (humic-fulvic component) into a 250 ml centrifuge bottle. Remove insoluble residue (humin component) and save. Acidify the decanted solution to pH 2.0 with addition of 6.0N and 1.0N hydrochloric acid. The 1.0N hydrochloric acid should be used as pH 2.0 is approached since the pH sensitivity of the system increases. At pH 2.0 two phases will be present: a humic acid precipitate and a fulvic acid supernatant. These are separated and saved after centrifuging the mixture at 2000 rpm for 30 minutes. The above extraction is repeated twice on the insoluble humin residue. The fulvic acid solutions and humic and humin residues are placed in a vented fumehood and allowed to evaporate to dryness. Samples are then thoroughly dried at 40°C, homogenized and dissaggregated using an agate mortar and pestle. 0.5000 g aliquots of dried and dissaggregated sample are subjected to total digestion procedures as described in Appendix II(I) in preparation for analysis.

III. COMMENTS ON THE QUALITY OF REAGENTS USED

Reagents used in the sequential partial extraction procedures were all of analytical grade. Blank solutions of each reagent were prepared in identical manner to solutions prepared from sediment samples and routinely analyzed for possible metal contamination. Only blank solutions of sodium hypochlorite indicate an apparent contamination equalling approximately 20 ppm copper. The nature of this contamination was further investigated by preparing a stock solution of sodium hypochlorite in the laboratory (Appendix II (IV)) and comparing copper values in this solution to values determined in commercially available sodium hypochlorite. Copper concentrations were observed to be similar in each of the solutions. Since the laboraty grade sodium hypochlorite was prepared from pure reagents containing no copper, apparent contamination can only be attributed to a spectral interference phenomena. The application of sodium hypochlorite in sequential partial extractions therefore requires that blank solutions be prepared for each batch of samples to be analyzed. Copper values in samples are then determined by calculating the difference between copper values analyzed in samples and values detected in blank sodium hypochlorite.

IV. PROCEDURES FOR PREPARATION OF A STOCK SOLUTION OF SODIUM HYPOCHLORITE

A sodium hypochlorite solution is prepared as follows:

Reagents Required:

32 gm sodium hydroxide, 150 ml de-ionized water, 150 g ice prepared from de-ionized water, chlorine gas

Procedure:

Dissolve 32 g of sodium hypochlorite in 150 ml of water. Cool the solution to normal temperature and add 150 g of crushed ice. Pass a rapid stream of chlorine gas through the solution until the weight has increased by 21 - 24 g.

Note: Commercially available sodium hypochlorite was used in the present study since both it and laboratory prepared solutions show similar interference phenomena.

APPENDIX III

ANALYTICAL PROCEDURES

APPENDIX III

ANALYTICAL PROCEDURES

I. ATOMIC ADSORPTION SPECTROPHOTOMETRIC ANALYSIS

Introduction and Theory of Operation

Concentrations of Cu, Zn, Fe and Mn have been determined by the author at Lakehead University facilities using Perkin Elmer Model 303 and Model 2380 atomic adsorption spectrophotometers. Initial preparation for analysis requires that the samples be brought into solution. In the present study samples were dissolved according to sequential partial extraction and total digestion techniques described in Appendix II(I).

Sample solutions are introduced into the instrument by the nebulizer and are vapourized by means of a flame fueled by a mixture of air/acetylene in this study. Temperatures of the flame are sufficient to reduce droplets of the sample solution into atomic form. Atomic species in the flame are in turn illuminated by a hollow cathode lamp composed of the same element that is being analyzed. Atoms in the flame will adsorb at a distinct frequency incident energy radiated from the hollow cathode lamp. Remnant radiation from the lamp is then isolated by means of a monchromator and it's reduced intensity measured on a detector. The amount of adsorption of light energy in the flame is proportional to the concentration of the element in the vapour and the measurement of decrease in energy received by the detector permits a quantitative determination of the element concentration.

Prior to analysis of sample solutions the instrument must first be calibrated. Calibration requires the use of solution standards. For the present study standard stock solutions for each of Cu, Zn, Fe and Mn were prepared using high purity metals and methods outlined in the Perkin Elmer Manual Analytical Methods for Atomic Adsorption Spectrophotometry (1976). For each element to be analyzed up to seven solution standards were prepared by step-wise dilution of standard stock solutions. Standards are made up so that metal concentrations encompass the entire range of values expected in the samples. The solution with the highest concentration is labelled the reference having an absorbance value greater than or equal to the highest expected metal concentration in samples.

Operation Procedures for the Model 303 Instrument

Operation of the Model 303 instrument is as follows:

(a) The appropriate element lamp is installed, the power switches are engaged and the instrument is allowed to reach optimum operating conditions during a minimum fifteen minute period.

(b) Current milliampere, ultraviolet or visible wavelength and slit settings are adjusted according to lamp specifications.

(c) Amplifier gain is adjusted to a peak deflection setting by fine clockwise rotation of the wavelength and gain controls and by final alignment adjustments of the lamp.

(d) Adsorption and zero knobs are both set to null point readings.

(e) Air-fuel flow rates and mixtures, burner head height and nebulizer aspiration rates are adjusted to optimum settings according to Perkin-Elmer specifications.

(f) Calibration is performed by aspirating and observing peak absorbance values for each of the seven solution standards. Final absorbance is recorded as a value equal to the difference between absorbance for standards and absorbance for high quality deionized water.

(g) Subsequent analyses of sample solutions are conducted in a similar manner in groups of five samples; the first and fifth sample being the reference standard and samples 2, 3 and 4 being sample solutions. Two readings per sample are recorded and averaged. Frequent repetitive analysis of the reference standard is performed to monitor instrument drift.

(h) Linear regression is performed on absorbance data from the suite of solution standards via Fortran program load XRF (KH Poulsen). This data is subsequently transposed into actual element concentrations in another step of the same program.

Operation Procedures for the Model 2380 Instrument

The analytical operation of the Model 2380 instrument is as follows:

(a) Verify that either the plastic spoiler or impact head is properly installed in the burner assembly. In the present study, use of hydrofluoric acid in Extraction #6 and Total Digestions (Appendix II) required that the plastic spoiler be used. Hydrofluoric acid will attack the impact bead made of pyrex. The plastic spoiler was therefore used in all applications to standardize the analytical set-up.

(b) The appropriate lamp is installed, the power switches are engaged and the instrument is allowed to reach an optimum operating temperature over a minimum one minute period.

(c) Current milliampere, ultraviolet or visible wavelength and slit settings are adjusted according to lamp specifications.

(d) Amplifier gain is adjusted to a peak setting of 75 on the lamp energy digital display. This is accomplished by fine clockwise adjustment of the gain control knob and final lamp alignment adjustments.

(e) The model 2380 instrument is equipped with an automatic zero mechanism on the keyboard panel so that adjustment of adsorption and zero knobs is not necessary as in the Model 303.

(f) Air-fuel flow rates and mixtures, burner head height and nebulizer aspiration rates are adjusted to optimum settings according to Perkin-Elmer specifications.

(g) Calibration of the Model 2380 is performed with either a single solution standard if the expected metal concentration in samples falls within the linear range of the instrument or by using two or three standards depending on the degree to which the linear range is exceeded. The linear working range for each element is listed in the Perkin-Elmer Manual Analytical Methods in Atomic Adsorption Spectrophotometry. The Model 2380 is capable of calibrating directly in units of concentration. The concentration of the solution standard is set at the maximum expected value for samples and entered by the digital keyboard. A blank solution of high quality de-ionized water is aspirated, the integration time is set and the automatic zero control is engaged. The solution standard is then aspirated and the read key is engaged. Completion of the calibration cycle will coincide with a read-out on the digital display of the metal concentration in the standard. With the calibration curve established the instrument is capable of converting absorbance values directly into concentration values which are then displayed.

(h) Subsequent analyses of sample solutions are conducted in groups of five samples; the first and fifth sample being the standard and samples 2, 3 and 4 being sample solutions. Five readings per sample are performed and averaged through the built in averaging program of the instrument.

Comments on Analysis of Iron

For iron analysis, extraction solutions were diluted by a factor of 50x with high quality de-ionized water and spiked with 10 ml of strontium nitrate solution. Strontium nitrate is prepared by mixing 36 gm strontium nitrate to 1 litre of de-ionized water and acts to suppress spectral interference particularly by alkali compounds. Addition of strontium nitrate to ammonium oxalate extractant solutions (Extraction #4, Appendix II) was observed to produce a white precipitate. By analyzing identical solutions with and without the precipitate, it was discovered that the presence of the precipitate had no effect on the concentration of Fe detected in the solution. Therefore to avoid possible clogging of the nebulizer and burner head assembly, strontium nitrate was not routinely added to ammonium oxalate solutions.

Comments on Analysis of Sodium Hypochlorite Extracts

The high concentrations of sodium in sodium hypochlorite extractants require frequent cleaning of the burner head assembly to avoid possible clogging. This task should be performed with a dilute solution of nitric acid after completing each sample batch.

II. FLUORIMETRIC ANALYSIS

Introduction and Theory of Operation

Concentrations of uranium have been determined by the author by fluorimetric analysis at Saskatchewan Research Council (SRC) facilities, Saskatoon, Saskatchewan using a Jarrell-Ash fluorimeter. In preparation for fluorimetric analysis, solid samples must first be brought into solution. In the present study, samples were dissolved according to sequential partial extraction and total digestion procedures described in Appendix II(I). Fluorimetric analysis hinges on the principle that uranium bearing sample solutions fused with a sodium fluoride flux will fluoresce when exposed to ultraviolet radiation. The intensity of fluorescence measured in the fluorimeter is proportional to the concentration of uranium in the sample.

The effects cf quenching must always be considered when performing fluorimetric analysis. Quenching effectively reduces sample fluorescence and has been attributed to the presence of interfering elements including iron and manganese. Quenching is particularly apparent in samples containing high concentrations of iron and manganese. At SRC, effects of quenching have been compensated for by the addition of a transmittance module to the fluorimeter. In the transmittance mode, ultra violet light is transmitted through the fused sample. Intensity of transmitted light is proportional to the amount of iron and manganese present which tends to increase the relative opacity of fused discs. Numerical correction factors have been determined for various values of transmittance. These correction factors are then applied to fluorescence readings recorded while operating the instrument in the fluorescence mode. Actual uranium concentrations in samples are calculated by the following formula:

$$U\ \text{ppm} = \left(\frac{0.300}{a - b} \times c\right) \times [(S.F. \times C.F.) - b]$$

0.300 = a constant value equal to mg/ml U

a = mean fluorescence determined from at least two solution standards analyzed with each batch of samples

b = mean fluorescence determined from at least two blank solutions analyzed with each batch of samples

c = ratio of final volume of sample after dissolution/initial weight of sample*

S.F. = measured fluorescence value of sample

C.F. = correction factor applied according to measured transmittance value of sample

*in the present study the ratio is 100/1.

Procedures for Uranium Analysis

Routine analysis of uranium is performed according to the following procedure:

1. 0.200 ml of sample solution are pipetted onto platinum dishes and allowed to evaporate in a convection oven.
2. Dishes containing evaporated samples are placed on a sample fusion rotary burner and heated by propane induced flame to 500°C for

1.5 minutes. Ashing of sodium hypochlorite extractant solutions by this technique is unsuitable since samples are observed to explode out of the platinum dishes. The high sodium salt and organic content of extracts are considered to be the source of the problem. Therefore, sodium hypochlorite extractant solutions are ashed in a muffle furnace where temperatures can be gradually increased to 500°C. This method is observed to be a satisfactory alternative.

3. After ashing, commercially prepared sodium fluoride pellets are added to each of the platinum dishes and the samples are fused at 500°C for 3 minutes on the sample fusion rotary burner.
4. Samples are allowed to cool, fluorescence and transmittance values are immediately read on the fluorimeter, appropriate correction factors are applied and actual uranium concentrations are calculated.

III. CHN ANALYSIS

Introduction and Theory of Operation

Carbon determinations on lake, stream and bog sediments were performed on a Perkin Elmer Model 240 Elemental Analyzer by staff of the Science Instrumentation Laboratory, Lakehead University. The instrument determines element content by detecting and measuring their combustion products, namely, CO_2. Combustion occurs in pure oxygen under static conditions. Combustion products are analyzed automatically in a self-integrating, steady-state thermal conductivity analyzer and results are recorded in bar graph form on a 0-1 mV recorder.

Operation Procedures for the Model 240 Instrument

Analyses are performed according to the following procedure outlined in the Perkin Elmer Manual Model 240 Elemental Analyzer.

1. 0.0004 grams of sample are weighed into a platinum boat of known weight, placed in the magnetically operated ladle and inserted into the sample entrance fitting.
2. The Start button is engated and the entire system flushed with helium at a high flow rate for 90 seconds.
3. The combustion train is then flushed with oxygen for 30 seconds.
4. After the inject panel light comes on, the sample is moved into the combustion chamber and the combust button is engaged. Procedures following this step are fully automatic.
5. Combustion occurs at approximately 950°C. During the high heat interval combustion products are flushed from the combustion train into a 300 ml spherical glass mixing volume where they are homogenized over 90 seconds. While the sample gases are mixing, helium flows through the system and the "zero" output with no sample in the detector is determined. When mixing is complete sample gases are allowed to expand to atmospheric pressure and then displaced through the detectors where the signal output is recorded over 30 seconds. The difference in microvolts between each "read" and "zero" output on the bar graph is in direct proportion to the concentration of the gas measured. Actual carbon concentrations are computed with standard formulae.

IV. ACCURACY AND PRECISION OF ATOMIC ADSORPTION AND FLUORIMETRIC ANALYSES

Analytical accuracy and precision of atomic adsorption spectrophotometry and fluorimetry were determined in the present study by a series of standard samples and a suite of replicate analyses, respectively.

Analytical accuracy is considered to be a measure of the ability of an analytical method to reflect the true concentration of an element. Sample MRG-1 was employed as a standard sample to determine analytical accuracy in atomic adsorption spectrophotometry. MRG-1 is a sample of Mount Royal Gabbro. A petrographic description and assay analysis is provided by Faye, 1978. Two specific weight fractions of MRG-1 were selected to match the range of metal values expected in the sediment samples. One weight fraction was chosen to match the upper limit, the second weight fraction to match a value approximately 1/10 the upper limit. Chosen concentration levels for each metal are as follows: copper, 100 and 10 ppm; zinc, 240 and 20 ppm; manganese, 600 and 60 ppm; and iron, 5.0% and 0.5%. Weighed fractions of MRG-1 were subjected to a total digestion procedure as described in Appendix II. Solutions of MRG-1 were inserted into sample batches at a frequency of approximately 10% for each element analyzed. Sample LS-1 a homogenized lake sediment is used at Saskatchewan Research Council facilities to determine accuracy of fluorimetric analysis. Fractions of LS-1 were prepared for analysis by total digestion procedures and inserted into sample batches at a frequency of approximately 5%.

Since standard samples are not presently available for sequential partial extraction procedures accuracy of individual extractions could not be established. Accuracy as reported is considered to be a measure of instrument accuracy.

Analytical precision refers to the limits within which analyses are reproducible. In the present study, a suite of replicate samples comprised of one lake, stream and bog sediment has been applied to a test of analytical precision. Each sample of the replicate suite has been separately subjected to sequential partial extractions and analyzed ten times over the course of the study. Precision has been determined by calculating the coefficient of variation of observed metal values about mean values determined for each element and each extraction. Data are summarized in Chapter 4.2.2, Tables 5, 6 and 7.

APPENDIX IV

SUMMARY OF STATISTICAL METHODS USED

APPENDIX IV

SUMMARY OF STATISTICAL METHODS

Statistical methods employed in the present study are as follows:

I. THE MANN WHITNEY STATISTICAL TEST

The Mann Whitney statistic can be used to test the hypothesis that two populations are equal against the alternative that they differ (Mann & Whitney, 1947; Kreyszig, 1970). In the present study it has been used to determine whether or not analytical data derived from Model 303 and Model 2380 Atomic Adsorption Spectrophotometers differ. From a suite of replicate samples, eight analyses of copper, zinc, iron and manganese were derived from the Model 303 and two from the Model 2380 instrument. In applying the statistic, data from both instruments have been pooled. Data from Model 2380 was labelled n_1, data from Model 303 n_2. Values from n_1 and n_2 were arranged in order of size from smallest to largest and assigned rank scores of 1, 2, 3, etc. In the case of similar values for a number of observations, a mean rank score was determined for the sequence. The sum of rank scores for the smaller of the two populations (n_1) is determined and assigned the value w. This value is compared to critical values c_1 and c_2 which have been published for specific sample populations sizes n_1 and n_2. If the calculated value of w lies within the range of the two critical values, the populations can be considered equal. Critical values are read from tables published in Kreyszig (1970). For the present study $n_1 = 2$, $n_2 = 8$ and critical values c_1 and c_2 are 3 and 19 at the 95% confidence interval. Calculated values of w for copper, zinc, iron and manganese in all extractions of replicate samples are listed in Table 3 and the results are summarized in Chapter 4.2.1.

II. THE SUBSTITUTE t-Test

The substitute t-test can be used to determine the significance between mean values of two populations of equal size (Miller and Kahn, 1962; Dixon and Massey, 1969). In the present study it has been used to determine the significance between mean total metal values calculated as a sum of all sequential partial extractions and mean total values determined from a total digestion of the same replicate sample. In this way it has been used to test the reliability of the sequential partial extraction method on the premise that the summed value of all extractions should equal the total metal value of the sample. The statistic is not recommended for sample populations where n exceeds 10 and involves the use of the mean ($\bar{x}$) and the range (w) of observed values. The statistic is computed by the formula:

$$\tau d = \frac{\bar{x}_1 - \bar{x}_2}{1/2\ (w_1 + w_2)}$$

where $\bar{x}_1$ is the mean of one population (in the present study data from total digestions)

$\bar{x}_2$ is the mean of the second population (in the present study data from the sum of sequential partial extractions)

w_1 is the range of observed values in population 1

w_2 is the range of observed values in population 2

Calculated τd values are compared to critical values determined for specific population sizes published in Table form in Dixon and Massey, 1969. If calculated τd values are less than determined values for a specific confidence interval the means of the two populations can be considered to be statistically equal. In the case of the present study critical values of τd are $\pm$ .304 at the 95% confidence interval. The substitute t-test is summarized in Chapter 4.2.3 and Table 8.

III. OTHER STATISTICS

The following statistics employed in the present study have been computed from formulae as shown.

Arithmetic Mean ($\bar{x}$) - $\frac{\Sigma x}{n}$

Standard Deviation (σ_n) - $\frac{\Sigma x^2 - \frac{(\Sigma x)^2}{n}}{n}$

Coefficient of Variation - $\sigma n/\bar{x} \times 100$

x - observed values

n - number of observed values

σn - standard deviation

$\bar{x}$ - arithmetic mean

APPENDIX V

DATA FROM REPLICATE ANALYSIS

SEDIMENT TYPE	ELEMENT ANALYZED	REPLICATE ANALYSIS	TOTAL: TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SEQUENTIAL PARTIAL EXTRACTIONS: SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	D.I. WATER - HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES, AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
LAKE	U	*1 1	175	167	133	7	1	25		1
		2	179	169	140	5	1	22		1
		3	162	165	142	3	1	19		1
		4	173	168	146	4	1	17		
		5	165	158	130	1	1	25		1
		6	182	174	147	1	1	24		1
		7	177	166	139	1	1	24		1
		8	158	165	138	1	1	24		1
		9	178	153	119	7	*2	25		2
		10	188	181	149	5		26		1
	U	1	38	38	31			6		1
		2	47	42	35			6		1
		3	40	37	30			6		1
		4	44	40	35	1		6		
		5	39	41	32	1		7		1
		6	46	44	36	1	1	5		1
		7	41	39	31	1		6		1
		8	50	37	29			8		1
		9	46	44	37	1		6		
		10	43	48	39			8		1
	U	1	181	172	171				1	
		2	173	172	171				1	
		3	186	181	178	1		1	1	
		4	175	179	176	1		1	1	
		5	164	162	161				1	
		6	176	178	177				1	
		7	178	178	176	1			1	
		8	155	157	155	1			1	
		9	154	154	153					1
		10	182	170	168			1		1

METAL CONCENTRATION (ppm) AND EXTRACTION TYPE

*1 Analyses 1 and 2 are from Model 2380, 3-10 from Model 303 spectrophotometers.
*2 Blank columns indicate values less than the detection limit.

Table 14 Uranium concentrations in replicate lake, stream and bog sediment samples

SEDIMENT TYPE	ELEMENT ANALYZED	REPLICATE ANALYSIS	METAL CONCENTRATION (ppm) AND EXTRACTION TYPE							
			TOTAL	SEQUENTIAL PARTIAL EXTRACTIONS						
			TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	D.I. WATER - HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES, AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
LAKE	Cu	1	86	86	55	2	2	22	4	1
		2	83	86	49	3	3	25	5	2
		3	89	83	47	2	3	26	4	1
		4	88	80	46	2	2	24	5	1
		5	82	96	52	1	5	31	5	1
		6	88	90	49	3	3	30	5	1
		7	89	91	53	2	3	28	4	1
		8	88	95	54	2	4	30	4	1
		9	96	88	51	2	3	23	6	3
		10	89	86	47	1	6	22	8	3
STREAM	Cu	1	46	46	12		3	15	5	11
		2	46	45	13		2	14	5	11
		3	50	44	13		3	15	6	7
		4	46	42	13		1	16	5	7
		5	52	45	14	1	1	16	6	8
		6	52	45	12		3	16	5	9
		7	43	46	16		2	13	8	7
		8	49	51	12	2	3	16	4	14
		9	47	51	12	3	2	15	6	13
		10	44	47	12		3	15	4	13
	Cu	1	55	55	43	1	2	3	2	4
		2	54	54	39	1	2	4	4	4
		3	58	55	40		3	5	4	3
		4	52	55	41	1	4	3	2	4
		5	50	58	46		2	4	2	4
		6	56	58	45		2	4	3	4
		7	47	50	38	1	2	3	2	4
		8	49	57	45		2	5	2	3
		9	53	53	42		2	4	2	3
		10	46	47	38		2	3	2	2

Table 15 - Copper concentration in replicate lake, stream and bog sediment samples

SEDIMENT TYPE	ELEMENT ANALYZED	REPLICATE ANALYSIS	METAL CONCENTRATION (ppm) AND EXTRACTION TYPE TOTAL TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SEQUENTIAL PARTIAL EXTRACTIONS SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	D.I. WATER - HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES, AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
LAKE	Zn	1	252	256	53		21	142	18	22
		2	270	258	47		26	150	17	18
		3	264	250	45		22	146	20	17
		4	252	266	47		21	153	22	23
		5	268	263	42	2	41	146	13	19
		6	254	269	49		43	144	15	18
		7	238	253	45	1	42	130	16	19
		8	228	241	42		35	133	14	17
		9	228	240	66		27	108	16	23
		10	237	243	54		36	125	12	16
	Zn	1	92	99	11			38	8	42
		2	101	90	8			32	5	45
		3	94	101	9			40	4	48
		4	96	93	12			30	6	45
		5	98	90	8	1		28	8	45
		6	94	90	12	1		27	7	43
		7	90	96	8			33	4	51
		8	90	90	11	1	1	31	4	42
		9	97	96	8		2	38	5	43
		10	95	99	9	2	3	40	5	40
	Zn	1	32	33	17		3	5	4	4
		2	29	35	19		3	4	5	4
		3	26	23	15		1	1	5	1
		4	25	33	22		1	1	8	1
		5	32	32	18		4	3	3	4
		6	29	29	16		3	4	3	3
		7	28	36	19		4	5	3	5
		8	30	32	17		4	4	4	3
		9	36	33	16	1	5	6	3	3
		10	34	29	16		4	3	3	3

Table 16 Zinc concentrations in replicate lake, stream and bog sediment samples

SEDIMENT TYPE	ELEMENT ANALYZED	REPLICATE ANALYSIS	METAL CONCENTRATION (%) AND EXTRACTION TYPE							
			TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SEQUENTIAL PARTIAL EXTRACTIONS					
					SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	0.1 N. WATER - HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES, AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
	Fe	1	1.75	1.65	0.06		0.04	0.83	0.24	0.48
			1.60	1.75	0.11		0.07	0.71	0.25	0.61
			1.66	1.57	0.06		0.03	0.72	0.25	0.51
			1.74	1.72	0.08		0.05	0.75	0.22	0.62
		5	1.57	1.61	0.09		0.05	0.83	0.20	0.44
		6	1.73	1.62	0.10		0.04	0.82	0.22	0.44
		7	1.58	1.62	0.05		0.07	0.95	0.16	0.39
		8	1.68	1.65	0.08		0.05	0.85	0.18	0.49
		9	1.64	1.63	0.07		0.03	0.86	0.20	0.47
		10	1.62	1.68	0.09		0.01	0.88	0.24	0.46
	Fe	1	4.47	4.62	0.09	.15	.06	1.72	.18	2.42
		2	4.42	4.65	0.11	.16	.05	1.72	.22	2.39
		3	4.67	4.71	0.11	.11	.04	1.70	.24	2.51
		4	4.58	4.66	0.09	.22	.09	1.72	.16	2.38
		5	4.72	4.77	0.10	.15	.08	1.65	.15	2.64
		6	4.43	4.78	0.05	.11	.08	1.74	.18	2.62
		7	4.74	4.47	0.07	.11	.07	1.77	.17	2.58
		8	4.75	4.41	0.11	.14	.09	1.86	.22	1.99
		9	4.63	4.50	0.08	.15	.07	1.44	.15	2.61
		10	4.51	4.54	0.10	.15	.06	1.52	.19	2.62
	Fe		0.57	0.53	0.29	.04	.03	.10	.03	.04
		2	0.49	0.49	0.17	.03	.04	.13	.07	.05
		3	0.50	0.46	0.24	.02	.04	.09	.04	.03
		4	0.54	0.48	0.22	.03	.05	.09	.06	.03
		5	0.54	0.47	0.24	.01	.05	.07	.08	.02
		6	0.42	0.53	0.29	.02	.04	.10	.05	.03
		7	0.49	0.56	0.28	.05	.03	.11	.04	.05
		8	0.52	0.50	0.25	.03	.04	.09	.05	.04
		9	0.49	0.57	0.27	.02	.05	.12	.08	.03
		10	0.45	0.53	0.24	.06	.05	.11	.07	.04

Table 17 Iron concentrations in replicate lake, stream and bog sediment samples

			METAL CONCENTRATION (ppm) AND EXTRACTION TYPE							
			TOTAL	SEQUENTIAL PARTIAL EXTRACTIONS						
SEDIMENT TYPE	ELEMENT ANALYZED	REPLICATE ANALYSIS	TOTAL DIGESTION IN HYDROFLUORIC-NITRIC-PERCHLORIC ACID	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	D.I. WATER - HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (MN HYDROXIDES, AMORPHOUS) #3	ACID AMMONIUM OXALATE (FE HYDROXIDES AMORPHOUS) #4	HYDRAZINE CHLORIDE (FE HYDROXIDES XTALLINE) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
	Mn	1	264	269	96		68	57	9	39
		2	275	268	130	2	48	45	8	35
		3	266	277	111	1	56	50	14	45
			268	272	111		53	53	13	42
		5	272	267	105	2	58	53	10	39
		6	266	274	112	1	52	57	13	39
		7	278	259	92		69	53	8	37
			260	268	95		68	57	10	38
		9	278	274	100		65	56	11	42
		10	261	280	119		53	61	11	36
	Mn	1	576	569	85		58	110	32	284
		2	568	562	75	2	67	112	30	276
		3	578	563	83		66	99	30	285
		4	566	560	82	1	75	104	27	271
		5	560	564	80		78	106	26	274
		6	570	579	72		80	116	27	281
		7	580	573	72	1	73	115	28	284
		8	540	576	115		78	116	30	237
		9	567	541	82		79	99	19	262
		10	572	553	81		78	101	18	275
	Mn	1	61	54	36	3	1	2	4	8
		2	55	55	38	2	3	3	2	7
		3	51	51	34	1	1	4	5	6
		4	56	51	27	3	5	2	6	8
		5	58	59	41		2	3	3	10
		6	57	61	39		2	5	5	10
		7	60	54	36	2	2	2	4	8
		8	62	54	41		2	1	2	8
		9	60	60	41		1	4	4	10
		10	54	49	38		1	2	2	6

Table 18 Manganese concentrations in replicate lake, stream and bog sediment samples

APPENDIX VI

	Sample Number	Physico-Chemical Parameters (measured at sediment-water interface)					% Carbon Content (determined on whole sediment)
		pH	(+millivolts) oxidation reduction potential	micromhos/cm conductivity	(°C) Temp.	(ppm) Dissolved Oxygen	
Lake 1	1	7.2	620	100	16.5	9.7	9.9
	2	7.1	610	100	16.0	9.8	39.7
	3	7.0	620	100	15.5	9.7	31.7
	4	7.0	560	90	16.5	9.7	33.1
	5	6.5	600	110	15.0	9.1	33.0
	6	7.1	590	100	16.0	9.6	33.5
	7	7.0	530	110	16.5	9.8	30.8
	8	7.2	520	100	17.0	9.8	32.6
	9	7.1	490	100	17.0	9.8	29.8
	10	7.0	490	120	17.0	9.6	29.8
	11	7.2	540	100	17.0	9.9	29.3
Bog 1	12	6.8	630	100	16.5	9.2	46.2
	13	6.5	620	100	14.0	5.0	45.6
	14	6.3	510	110	10.5	2.8	44.0
	15	6.5	560	110	7.5	3.0	47.7
Stream 1	16	6.4	620	110	10.5	3.5	2.7
Lake 2	17	5.6	650	100	12.5	8.5	21.0
	18	6.0	700	90	15.0	8.8	17.9
	19	5.9	690	90	14.5	8.3	17.8
	20	5.9	660	90	14.5	8.5	17.4
	21	6.0	700	80	15.0	8.8	16.8
	22	6.0	670	75	14.8	8.7	21.0
	23	6.4	610	80	14.6	8.7	15.0

Table 19 Physico-Chemical parameters and percent carbon content in lakes, streams and bogs

Table 19 - continued

	Sample Number	Physico-Chemical Parameters (measured at sediment-water interface)					% Carbon Content (determined on whole sediment)
		pH	(+millivolts) oxidation reduction potential	micromhos/cm conductivity	(°C) Temp.	(ppm) Dissolved Oxygen	
Stream 2	24	6.4	695	90	13.5	5.6	8.4
	25	6.6	650	90	13.0	5.0	19.2
	26	6.6	6.5	90	13.0	4.8	29.2
	27	6.6	565	90	12.0	5.0	26.5
	28	7.1	560	90	11.5	6.4	32.9
	29	7.6	535	90	11.5	6.6	15.4
Bog 2	30	5.1	510	150	13.5	1.7	36.8
	31	5.2	510	100	12.5	3.4	37.4
	32	5.3	545	130	13.0	1.7	43.1
	33	4.9	545	130	12.0	1.5	46.3
	34	4.9	555	110	10.5	4.1	35.1
	35	5.2	545	100	11.5	2.4	46.3
	36	5.2	555	100	11.5	2.5	47.4
	37	5.4	515	110	11.5	1.3	41.1
	38	4.7	485	110	11.0	2.4	39.2
Lake 3	39	6.1	640	90	17.0	7.2	35.9
	40	6.0	660	90	18.0	7.2	38.2
	41	5.8	690	100	11.0	7.3	29.9
	42	6.1	600	160	7.0	7.2	26.3
	43	6.3	620	190	6.0	7.4	24.9
	44	6.0	675	100	14.0	6.7	21.1
Bog 3	45						17.5
	46	Parameters not measured in Bog 3					20.7

Table 19 - continued

		Physico-Chemical Parameters (measured at sediment-water interface)					% Carbon Content (determined on whole sediment)
	Sample Number	pH	(+millivolts) oxidation reduction potential	micromhos/cm conductivity	(°C) Temp.	(ppm) Dissolved Oxygen	
Bog 3	47						25.9
	48						36.8
Lake 3	49	6.3	270	180	7.5	7.3	22.8
	50	6.4	670	100	14.0	7.2	20.1
	51	6.4	630	110	14.5	7.3	17.4
	52	6.1	360	150	8.5	7.4	18.4
	53	6.4	645	110	15.5	7.5	19.3
	54	6.5	645	110	15.5	7.6	14.9
	55	6.5	650	110	15.0	7.4	20.9
	56	6.5	660	110	15.0	7.5	18.8
	57	6.4	660	110	15.0	7.6	19.3
Stream 3	58	5.9	615	90	14.5	4.2	17.9
	59	6.0	580	100	17.0	7.5	19.1
	60	6.1	640	90	16.5	8.5	11.1
	61	6.1	610	110	16.0	7.1	10.3
	62	6.2	595	120	15.5	6.8	1.7
	63	6.3	630	120	16.5	7.0	3.3
	64	6.2	580	120	16.0	7.1	0.5
	65	6.2	630	130	15.0	7.4	2.1
	66	6.1	620	120	17.0	9.5	10.9
	67	6.0	580	100	20.5	8.3	18.9
	68	5.8	610	120	18.5	6.8	18.4

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
		URANIUM CONCENTRATION (ppm)	1	2	1	2	1	2	1	2	1	2	1	2
Lake 1 Sediments	1	65	65	100	-		-		-		-		-	-
	2	88	74	84	-		-		14	16	-		-	-
	3	86	75	87	1	1	-		10	12	-		-	-
	4	116	112	97	-		-		4	3	-		-	-
	5	94	88	94	-		-		6	6	-		-	-
	6	95	90	95	-		-		5	5	-		-	-
	7	187	182	97	-		-		5	3	-		-	-
	8	139	134	96	-		-		5	4	-		-	-
	9	164	162	99	-		-		2	1	-		-	-
	10	110	105	96	-		-		5	4	-		-	-
	11	125	119	95	-		-		6	5	-		-	-
Bog 1 Sediments	12	93	90	97	-		1	1	2	2	-		-	
	13	58	56	97	-		1	1	1	1	-		-	
	14	49	46	94	-		1	2	2	4	-	-	-	-
Stream 1 Sediment	15	42	41	98	-		1	2	-	-	-	-	-	
	16	30	24	80	-		1	3	3	10	-		2	7
	17	37	33	91	-	-	-	-	3	9	-	-	1	-
Lake 2 Sediments	18	30	27	90	-	-	-	-	3	10	-	-	-	-
	19	44	38	86	-	-	-	-	6	14	-	-	-	-
	20	34	28	82	-	-	1	3	5	15	-	-	-	-
	21	31	26	84	-	-	-	-	5	16	-	-	-	-
	22	32	23	72	-	-	-	-	9	28	-	-	-	-

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

II. Table 20 Uranium concentrations in component fractions of lake, stream and bog sediments

Table 20 - continued

SEDIMENT TYPE	SAMPLE NUMBER	URANIUM CONCENTRATION (ppm) SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Lake 2	23	23	41 / 80	- / -	- / -	10 / 20	- / -	- / -
Stream 2 Sediments	24	39	37 / 95	- / -	- / -	2 / 5	- / -	- / -
	25	32	32 / 100	- / -	- / -	- / -	-	-
	26	28	22 / 79	-	-	6 / 21	-	- / -
	27	42	40 / 95	-	-	2 / 5	-	- / -
	28	53	43 / 81	1 / 2	-	8 / 15	-	1 / 2
	29	37	29 / 78	-	-	8 / 22	-	- / -
Bog 2 Sediments	30	123	122 / >99	- / -	- / -	1 / 1	- / -	- / -
	31	336	335 / 99	- / -	- / -	1 / <1	- / -	- / -
	32	275	270 / 98	- / -	1 / <1	3 / 1	- / -	1 / <1
	33	156	155 / 99	- / -	- / -	- / -	- / -	1 / 1
	34	142	140 / 99	- / -	- / -	2 / 1	- / -	- / -
	35	234	230 / 98	- / -	1 / <1	1 / <1	1 / <1	1 / <1
	36	202	200 / 99	- / -	- / -	1 / <1	- / -	1 / <1
	37	1865	1845 / 99	3 / 1	8 / <1	7 / <1	1 / <1	2 / <1
	38	992	980 / 99	2 / <1	4 / <1	5 / <1	- / -	1 / <1
Lake 3 Sediments	39	149	143 / 96	-	-	5 / 3	-	1 / 1
	40	87	85 / 98	-	-	1 / 1	-	1 / 1
	41	94	90 / 96	-	-	4 / 4	-	-
	42	76	64 / 84	3 / 4	-	9 / 12	-	-
	43	72	39 / 54	4 / 6	1 / 1	28 / 39	-	-
	44	92	90 / 98	-	-	2 / 2	-	-

1 / 2	
1	metal concentration in ppm unless otherwise stated
2	metal concentration expressed as a % of sum

Table 20 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	URANIUM CONCENTRATION (ppm) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
			1	2	1	2	1	2	1	2	1	2	1	2
Bog 3 Sediments	45	28	25	89	-		1	4	1	4	-		1	4
	46	32	29	91	-		-		1	3	-		2	6
	47	34	30	88	-		-		1	3	1	3	2	6
	48	196	195	99	-		-		1	.1	-		-	
Lake 3 Sediments (cont'd)	49	131	99	76	7	5	-		25	19	-		-	
	50	60	49	82	2	3	-		9	15	-		-	
	51	74	69	93	-		-		4	5	-		1	2
	52	62	48	77	-		-		14	23	-		-	
	53	64	47	73	2	4	-		15	23	-		-	
	54	60	54	90	1	2	-		3	5	1	2	1	2
	55	98	81	83	-		1	1	16	16	-		-	
	56	38	29	76	-		-		8	21	-		1	3
	57	66	58	88	-		1	1	7	11	-		-	
Stream 3 Sediments	58	32	30	94	-		-		1	3	-		1	3
	59	147	145	98	-		-		1	1	-		1	1
	60	46	41	89	-		-		1	2	2	4	2	4
	61	44	42	95	-		-		1	3	-	-	1	3
	62	11	8	73	-		-		-		1	9	2	18
	63	13	10	77	-		-		1	8	1	8	1	8
	64	6	4	66	-		-		-		-		2	33
	65	15	10	66	-		1	7	1	7	-		3	20
	66	25	23	92	-		-		1	4	-		1	4

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 20 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	URANIUM CONCENTRATION (ppm)					
			SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Stream 3	67	48	38 79	-	-	9 19	1 2	-
	68	29	28 97	-	-	-	-	1 3

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

COPPER CONCENTRATION (ppm)

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Lake 1 Sediments	1	46	5	11	1	2	3	6	9	20	6	13	22	48
	2	56	30	54	-	-	3	5	12	21	6	11	5	9
	3	48	28	58	-	-	3	6	9	19	5	11	3	6
	4	62	38	62	2	3	4	6	9	15	5	8	4	6
	5	54	33	61	2	4	4	7	8	15	4	7	3	6
	6	58	35	60	3	5	3	5	9	16	5	7	3	5
	7	83	37	45	3	4	4	5	14	17	5	6	20	23
	8	62	37	60	3	5	2	3	12	19	5	8	3	5
	9	41	23	56	2	5	2	5	9	22	4	10	1	2
	10	49	26	53	2	4	4	8	11	23	5	10	1	2
	11	51	27	53	3	6	2	4	11	23	4	8	4	8
Bog 1 Sediments	12	39	26	67	2	5	2	5	4	10	5	13	-	-
	13	43	26	60	3	7	4	9	4	9	5	12	1	2
	14	37	20	54	4	11	4	11	4	11	5	13	-	
	15	31	21	68	2	6	2	6	2	6	4	13	-	
Stream 1	16	45	4	9	2	4	3	7	10	22	4	9	22	49
Lake 2 Sediments	17	64	32	50	-	-	4	6	22	34	4	6	2	3
	18	58	27	40	-	-	4	6	21	31	5	7	1	1
	19	68	27	40	-	-	5	7	28	41	5	7	2	3
	20	56	23	41	-	-	5	9	20	36	6	11	2	4
	21	59	26	44	-	-	5	8	23	39	5	8	-	
	22	62	24	39	-	-	5	8	26	42	5	8	2	3

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

II. Table 21 Copper concentrations in component fractions of lake, stream and bog sediments

Table 21 - continued

			COPPER CONCENTRATION (ppm)											
SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Lake 2	23	84	15	18	-	-	3	4	57	68	7	8	2	2
Stream 2 Sediments	24	52	24	46	1	2	5	10	8	15	6	12	8	15
	25	47	24	51	1	2	5	11	6	13	6	13	5	11
	26	49	24	49	3	6	2	4	8	16	9	18	3	6
	27	43	22	51	2	5	4	9	5	12	6	14	4	9
	28	57	32	56	2	4	4	7	8	14	8	14	4	7
	29	47	16	34	1	2	2	4	13	28	8	17	7	15
Bog 2 Sediments	30	53	31	58	-	-	5	9	9	17	7	13	1	2
	31	77	39	51	2	3	7	9	22	29	5	6	2	3
	32	59	34	58	2	3	7	12	7	12	7	12	2	3
	33	47	26	55	-	-	8	17	5	11	7	15	1	2
	34	103	64	62	3	3	5	5	16	16	7	7	8	8
	35	70	53	75	-	-	8	11	3	4	5	7	1	1
	36	65	46	70	-	-	7	11	5	8	6	9	1	2
	37	49	24	49	-	-	8	16	8	16	7	14	2	4
	38	80	57	71	1	1	8	10	5	6	7	9	2	3
Lake 3 Sediments	39	60	34	56	-		5	8	6	10	12	20	3	5
	40	41	24	59	1	2	3	7	4	10	7	17	2	4
	41	48	29	60	1	2	3	6	5	10	7	15	3	6
	42	48	26	54	2	4	2	4	8	17	6	13	4	8
	43	46	23	50	2	4	2	4	11	24	5	11	3	7
	44	50	28	56	1	2	5	10	8	16	6	12	2	4

1	2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	COPPER CONCENTRATION (ppm) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
			1	2	1	2	1	2	1	2	1	2	1	2
Bog 3 Sediments	45	71	26	37	-	-	4	6	12	17	3	4	26	37
	46	168	87	52	3	2	7	4	30	18	2	1	39	23
	47	91	47	52	2	2	2	2	17	19	1	1	22	24
	48	125	100	80	1	1	4	3	13	10	1	1	6	5
Lake 3 Sediments (cont'd)	49	79	51	65	2	3	3	4	14	18	6	8	3	4
	50	43	22	51	2	5	2	5	9	21	4	9	4	9
	51	52	21	40	-		4	8	16	31	6	12	5	10
	52	44	15	34	-		3	7	20	45	5	11	1	2
	53	44	21	48	1	2	4	9	11	25	4	9	3	7
	54	43	16	37	1	2	4	9	15	35	5	12	2	5
	55	61	27	44	2	3	4	7	24	39	4	7	-	-
	56	63	17	27	-		3	5	37	59	5	8	1	2
	57	41	15	37	1	2	3	7	16	39	5	12	1	2
Stream 3 Sediments	58	26	11	42	-	-	4	15	5	19	5	19	1	4
	59	70	45	64	1	1	2	3	14	20	5	7	3	4
	60	40	8	20	1	2	2	5	12	30	4	10	13	33
	61	43	12	28	-		3	7	12	28	4	9	12	28
	62	22	1	5	1	5	2	9	6	27	3	14	9	41
	63	21	4	19	1	5	2	10	5	24	3	14	6	29
	64	27	1	4	1	4	2	7	5	19	4	15	14	52
	65	25	3	12	1	4	3	12	5	20	3	12	10	40
	66	44	12	27	2	5	4	9	7	16	5	11	14	32

1 / 2	
1	metal concentration in ppm unless otherwise stated
2	metal concentration expressed as a % of sum

			COPPER CONCENTRATION (ppm)					
SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Stream 3	67	76	22 / 29	-	1 / 1	2 / 55	4 / 5	7 / 9
	68	41	14 / 34	- / -	2 / 5	10 / 24	2 / 5	13 / 32

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

ZINC CONCENTRATION (ppm)

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS Mn HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS Fe HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
			1	2	1	2	1	2	1	2	1	2	1	2
Lake 1 Sediments	1	97	15	15	-	-	10	10	33	34	7	7	32	33
	2	75	31	41	-	-	4	5	29	39	7	9	4	5
	3	65	36	55	-	-	-	-	29	45	-	-	-	-
	4	70	27	39	-	-	4	6	18	26	13	19	8	11
	5	72	37	51	-	-	-	-	15	21	17	24	3	4
	6	64	30	47	-	-	-	-	25	39	6	9	3	5
	7	103	26	25	-	-	-	-	48	47	21	20	8	8
	8	109	35	32	-	-	2	2	50	46	7	6	15	14
	9	86	32	37	-	-	1	1	36	42	5	6	12	14
	10	107	26	24	-	-	-	-	51	48	17	16	13	12
	11	107	31	29	-	-	-	-	36	34	18	17	22	21
Bog 1 Sediments	12	39	36	92	-	-	-	-	-	-	3	8	-	-
	13	28	28	100	-	-	-	-	-	-	-	-	-	-
	14	32	25	78	-	-	-		3	9	4	13	-	
	15	12	11	92	-	-	-	-	-	-	-	-	1	8
Stream 1	16	78	8	10	-		3	4	14	18	8	10	45	58
Lake 2 Sediments	17	158	28	18	2	1	20	13	47	30	34	22	27	17
	18	80	21	26	2	2	8	10	29	36	7	9	13	16
	19	106	17	16	-	-	11	10	60	57	5	5	13	12
	20	104	17	16	-	-	18	17	43	41	4	4	22	21
	21	85	11	13	-	-	9	11	36	42	4	5	25	29
	22	120	28	23	5	4	23	19	53	44	3	2	8	7

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

II. Table 22 Zinc concentrations in component fractions of lake, stream and bog sediments

Table 22 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	ZINC CONCENTRATION (ppm)											
			SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
			1	2	1	2	1	2	1	2	1	2	1	2
Lake 2	23	213	16	8	5	2	16	8	145	68	8	4	23	11
Stream 2 Sediments	24	104	4	4	-		7	7	7	7	10	10	76	73
	25	90	24	27	-		13	14	16	18	6	7	31	34
	26	72	20	28	-		9	13	19	26	3	4	21	29
	27	70	17	24	-		6	9	12	17	3	4	22	31
	28	91	45	49	-		-		30	33	5	5	11	12
	29	89	11	12	-		1	1	31	35	4	4	42	47
Bog 2 Sediments	30	24	8	33	-	-	-	-	5	21	-		11	46
	31	48	27	56	-	-	1	2	6	13	3	6	11	23
	32	43	17	40	5	12	2	5	5	12	6	14	9	21
	33	20	15	75	-	-	-	-	-	-	5	25	-	
	34	41	9	22	2	5	-	-	6	15	2	5	22	54
	35	39	27	69	-	-	3	8	2	5	7	18	-	-
	36	26	20	77	-	-	3	12	1	4	2	8	-	-
	37	32	23	72	1	3	1	3	3	9	2	6	2	6
	38	47	31	66	-	-	1	2	5	11	6	13	4	9
Lake 3 Sediments	39	113	57	50	-	-	11	10	27	24	7	6	11	10
	40	58	39	67	-	-	4	7	5	9	3	5	7	12
	41	58	27	47	-	-	3	5	14	24	4	7	10	17
	42	88	30	34	-	-	7	8	34	39	4	5	13	15
	43	82	20	24	-	-	7	9	46	56	-		9	11
	44	147	42	29	-	-	8	5	43	29	6	4	48	33

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 22 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	ZINC CONCENTRATION (ppm) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Bog 3 Sediments	45	67	7	10	1	1	1	1	5	7	3	4	50	74
	46	67	7	10	1	1	1	1	7	10	4	6	47	70
	47	53	6	11	1	2	2	4	5	9	4	8	35	66
	48	48	17	35	3	6	4	8	9	18	4	8	11	23
Lake 3 Sediments (cont'd)	49	234	66	28	-	-	27	12	108	46	5	2	28	12
	50	66	21	32	-	-	-	-	30	45	1	2	14	21
	51	125	17	14	2	2	13	10	56	45	5	4	32	26
	52	118	18	15	-	-	9	8	52	44	24	20	15	13
	53	74	15	20	-	-	6	8	45	61	3	4	5	7
	54	103	15	15	2	2	9	9	40	39	4	4	33	32
	55	144	22	15	-	-	22	15	85	59	8	6	7	5
	56	123	6	5	-	-	13	11	72	59	10	8	22	18
	57	96	9	9	2	2	12	13	47	49	7	7	19	20
Stream 3 Sediments	58	64	13	20	4	6	8	13	4	6	10	16	25	39
	59	54	10	19	-	-	5	9	3	6	2	4	34	63
	60	66	7	11	-	-	3	5	6	9	4	6	46	70
	61	66	8	12	-	-	6	9	6	9	2	3	44	67
	62	56	6	11	4	7	2	4	5	9	2	4	37	66
	63	44	6	14	1	2	4	9	5	11	1	2	27	61
	64	84	5	6	-	-	3	4	5	6	2	2	69	82
	65	61	5	8	-	-	5	8	4	7	1	2	46	75
	66	95	6	6	3	3	4	4	5	5	4	4	73	77

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 22 - continued

SEDIMENT TYPE	SAMPLE NUMBER	ZINC CONCENTRATION (ppm)						
		SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
Stream 3	67	127	9 / 7	1 / 1	4 / 3	75 / 59	8 / 6	30 / 24
	68	62	62 / 10	- / -	1 / 2	8 / 13	4 / 6	43 / 69

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	IRON CONCENTRATION (%)											
			SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Lake 1 Sediment	1	2.94	.08	3	.12	4	.05	2	.75	26	.17	6	1.77	60
	2	1.05	.12	11	.12	11	-		.46	44	.13	12	.22	21
	3	1.25	.08	6	.19	15	.02	2	.55	44	.10	8	.31	25
	4	1.19	.13	11	-	-	.08	7	.36	30	.18	15	.44	37
	5	0.74	.21	28	.02	3	.05	7	.21	28	.05	7	.20	27
	6	1.22	.24	20	.12	10	.05	4	.34	28	.05	4	.42	34
	7	1.79	.14	8	.26	15	-		.30	17	.30	17	.79	44
	8	1.52	.08	5	-	-	.08	5	.40	26	.20	13	.76	50
	9	1.75	.21	12	.02	1	-	-	.69	39	.21	12	.62	35
	10	2.00	.18	9	.09	1	-		.54	27	.39	20	.80	40
	11	2.08	.05	2	.12	6	.02	1	.54	26	.37	18	.98	47
Bog 1 Sediment	12	0.97	.25	26	.28	29	<.01	1	.19	20	.18	19	.06	47
	13	1.04	.38	37	.02	2	.14	13	.15	14	.34	33	<.01	1
	14	1.71	.79	46	.05	3	.02	1	.53	31	.29	17	.03	2
	15	0.63	.32	51	.08	13	.02	34	.02	3	.16	25	.03	5
Stream 1	16	7.59	.62	8	.16	2	.13	2	4.49	59	.27	4	1.92	25
Lake 2 Sediment	17	1.38	.18	13	.05	4	.07	5	.40	29	.50	36	.18	13
	18	0.95	.18	19	.02	2	.04	4	.44	46	-	-	.27	28
	19	1.03	.14	14	-		.07	7	.69	67	-	-	.13	13
	20	1.47	.08	5	.05	3	.08	5	.53	36	.60	41	.13	9
	21	1.40	.14	10	-	-	.07	5	.38	27	.19	14	.62	44
	22	1.76	.11	6	.05	3	.11	6	.90	51	.18	10	.41	23

1	
	2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

II. Table 23 Iron concentrations in the component fractions of lake, stream and bog sediments

Table 23 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	ZINC CONCENTRATION (%)											
			SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		AMMONIUM OXALATE ACID (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
			1	2	1	2	1	2	1	2	1	2	1	2
Lake 2	23	5.46	.18	3	-	-	.02	<1	3.19	58	.84	15	1.23	23
Stream 2 Sediments	24	2.83	.01	<1	-	-	-	-	.20	7	.04	1	2.58	91
	25	1.89	.08	4	-	-	-	-	.37	20	.06	3	1.38	73
	26	2.44	.15	6	.04	2	.07	3	1.32	54	.12	5	.74	30
	27	1.74	.17	10	-	1	.10	4	.53	52	.08	4	.86	29
	28	2.30	.24	10	.02	1	.09	4	1.20	52	.09	4	.66	29
	29	4.21	.08	2	.02	<1	.12	3	1.86	44	.14	3	1.99	47
Bog 2 Sediments	30	0.77	.13	17	-	-	.01	1	.22	29	.03	4	.38	49
	31	0.72	.19	26	-	-	.03	4	.16	22	.06	8	.28	39
	32	0.73	.25	34	<.01	<1	.15	21	.14	19	.05	7	.13	18
	33	0.45	.24	53	-	-	.04	9	.09	20	.05	11	.03	6
	34	1.15	.15	13	-	-	.03	3	.24	21	.05	4	.68	59
	35	0.45	.29	64	<.01	<1	.04	9	.05	18	.03	7	.04	9
	36	0.39	.25	77	-	-	.03	8	.06	16	.02	5	.03	8
	37	0.80	.29	36	-	-	.04	5	.25	31	.04	5	.18	23
	38	0.70	.22	31	-		.05	7	.21	30	.04	6	.18	26
Lake 3 Sediments	39	0.84	.17	20	.01	1	.05	6	.38	45	.05	6	.18	21
	40	0.78	.25	32	.03	4	.07	9	.28	36	.06	8	.09	12
	41	0.93	.14	15	.03	3	.03	3	.48	51	.03	3	.22	24
	42	1.34	.10	7	.01	1	.03	2	.84	63	.07	5	.29	22
	43	2.46	.11	4	-	-	.10	4	1.92	78	.07	3	.26	11
	44	1.56	.08	5	.01	1	.03	2	.46	29	.03	2	.95	61

1	2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 23 - continued

IRON CONCENTRATION (%)

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS Mn HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS Fe HYDROXIDES)		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Bog 3 Sediments	45	3.09	.07	2	.04	1	.06	2	.30	10	.09	3	2.53	82
	46	2.31	.05	2	.05	2	.08	3	.16	7	.07	3	1.90	83
	47	2.10	.21	10	.04	2	.06	3	.28	13	.07	3	1.44	69
	48	0.72	.39	54	-		.03	4	.25	35	.04	6	.01	1
Lake 3 Sediments (cont'd)	49	1.48	.05	3	-	-	.05	3	.86	58	.05	3	.47	32
	50	1.27	.05	4	-		-		.79	62	.04	3	.39	31
	51	2.42	.08	3	-	-	.11	5	.48	20	.26	11	1.49	62
	52	2.09	.21	10	-		.16	8	1.02	49	.08	4	.62	30
	53	1.43	.04	3	-		.07	5	.96	67	.03	2	.33	23
	54	2.25	.08	4	-		.08	4	.46	20	.43	19	1.20	53
	55	1.76	.05	3	.08	5	.01	1	.63	36	.31	18	.68	39
	56	3.85	.05	1	.02	1	-		2.17	56	.38	10	1.23	32
	57	1.26	.11	9	-		.07	6	.69	55	.17	13	.22	17
Stream 3 Sediments	58	1.66	.08	5	.09	5	.04	2	.26	16	-	-	1.19	72
	59	2.10	.21	10	-	-	.04	2	.31	15	.12	6	1.42	68
	60	2.25	.05	2	.03	1	.05	2	.27	12	.05	2	1.80	80
	61	2.82	.07	2	.02	1	.03	1	.50	18	.05	2	2.15	76
	62	2.16	.06	3	.03	1	.05	2	.36	17	.06	3	1.60	74
	63	2.67	.06	2	.01	<1	.09	3	.25	9	.06	2	2.20	82
	64	3.95	.06	2	.02	<1	.09	2	.17	4	.08	2	3.53	89
	65	2.50	.07	2	.01	<1	.06	2	.19	8	.08	3	2.09	84
	66	3.68	.08	2	.04	1	.09	2	.21	6	.18	2	1.12	87

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 23 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	IRON CONCENTRATION (%) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
	67	4.18	.10 2	.02 1	.09 2	2.67 64	.18 4	1.12 27
	68	2.37	.12 5	- -	.03 1	.38 .16	.08 3	1.76 74

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	MANGANESE CONCENTRATION (ppm) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Lake 1 Sediments	1	602	127	21	18	3	33	5	101	17	24	4	299	50
	2	314	181	58	2	1	33	11	69	22	12	4	17	5
	3	305	199	65	5	2	25	8	45	15	14	5	17	6
	4	207	103	50	4	2	14	7	36	17	19	9	31	15
	5	184	123	67	2	1	14	8	25	14	8	4	12	7
	6	199	118	59	-	-	14	7	28	14	15	8	24	12
	7	338	155	46	6	2	36	11	52	15	28	8	61	18
	8	301	151	50	3	1	28	9	49	16	24	8	46	15
	9	453	256	57	4	1	42	9	45	10	24	5	82	18
	10	466	217	47	7	2	49	11	93	20	42	9	58	12
	11	434	224	52	3	1	43	10	73	17	32	7	59	14
Bog 1 Sediments	12	1106	937	85	31	3	31	3	67	6	37	3	3	< 1
	13	551	474	86	14	3	17	3	29	5	15	3	2	< 1
	14	623	531	85	12	2	22	3	37	6	19	3	2	< 1
	15	119	94	79	12	10	6	5	1	1	5	4	1	1
Stream 1	16	905	76	9	26	3	270	30	459	51	39	4	35	4
Lake 2 Sediments	17	119	42	35	-	-	16	13	18	15	6	5	37	31
	18	121	42	35	1	1	20	17	20	17	8	7	31	26
	19	126	42	33	-	-	20	16	25	20	8	6	31	25
	20	140	48	34	-	-	22	16	32	23	8	6	30	21
	21	134	43	32	2	1	21	16	26	19	7	5	35	26
	22	213	77	36	-	-	53	25	41	19	10	5	32	15

[1] metal concentration in ppm unless otherwise stated

[2] metal concentration expressed as a % of sum

II. Table 24 Manganese concentrations in component fractions of lake, stream and bog sediments

Table 24 - continued

			MANGANESE CONCENTRATION (ppm)											
SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Lake 2	23	1558	100	6	-	-	778	50	520	33	48	3	112	7
Stream 2 Sediments	24	321	24	8	1	<1	19	6	24	8	10	3	243	76
	25	461	136	30	1	<1	22	5	27	6	11	2	264	57
	26	773	401	52	4	1	102	13	156	20	26	3	84	11
	27	322	150	46	2	1	23	7	36	11	13	4	98	30
	28	759	497	65	2	<1	74	10	85	11	27	4	73	10
	29	539	115	21	3	1	61	11	99	18	23	4	237	44
Bog 2 Sediments	30	134	63	47	1	1	4	3	2	1	4	3	60	45
	31	194	140	72	3	2	6	3	3	2	1	<1	41	21
	32	194	162	84	4	2	7	4	1	<1	1	<1	19	10
	33	59	34	58	3	5	1	2	8	14	8	14	5	8
	34	146	70	48	1	1	5	3	1	1	10	7	59	40
	35	244	210	86	4	1	4	1	5	2	12	5	9	4
	36	149	120	81	2	1	3	2	5	3	13	9	6	4
	37	144	90	63	1	1	4	3	6	4	10	7	33	23
	38	84	43	51	4	5	1	1	2	2	5	6	29	35
Lake 3 Sediments	39	270	187	69	2	1	24	9	23	9	16	6	18	7
	40	235	178	76	2	1	15	6	16	7	7	3	17	7
	41	292	186	64	-	-	33	11	30	10	12	5	30	5
	42	330	179	54	-	-	62	19	42	13	12	4	35	10
	43	544	260	48	3	1	150	28	94	17	13	2	23	4
	44	319	105	33	3	1	26	8	34	26	13	4	138	43

[1] metal concentration in ppm unless otherwise stated

[2] metal concentration expressed as a % of sum

Table 24 - continued

SEDIMENT TYPE	SAMPLE NUMBER	MANGANESE CONCENTRATION (ppm)												
		SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	SODIUM HYPOCHLORITE (ORGANIC MATTER) #1		DI WATER-HYDROCHLORIC ACID (CARBONATE) #2		HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3		ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4		HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5		HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6	
Bog 3 Sediments	45	333	10	3	-		3	1	5	2	3	1	312	94
	46	186	17	9	-	-	3	2	5	3	5	3	156	84
	47	137	19	14	1	1	3	2	5	4	2	1	107	78
	48	133	88	66	4	3	9	7	14	10	3	2	15	11
Lake 3 Sediments (cont'd)	49	269	130	48	3	1	48	18	45	17	8	3	35	13
	50	243	96	40	1	<1	40	16	46	19	7	3	53	22
	51	272	60	22	-	-	31	11	34	13	9	3	138	51
	52	372	98	26	-	-	109	29	107	29	11	3	47	13
	53	237	101	43	2	1	39	17	44	19	4	2	47	20
	54	258	39	15	1	<1	31	12	44	17	11	4	132	51
	55	206	68	33	-	-	45	22	49	24	7	3	37	18
	56	590	108	18	-	-	167	28	200	34	23	4	92	16
	57	187	68	36	-	-	34	18	37	20	10	5	38	20
Stream 3 Sediments	58	265	35	13	2	1	8	3	13	5	7	3	200	75
	59	329	62	19	1	<1	16	5	26	8	10	3	214	65
	60	331	10	3	1	<1	7	2	12	4	3	1	298	90
	61	338	12	4	1	<1	3	1	10	3	4	1	308	91
	62	343	16	5	2	1	19	6	10	3	2	1	294	86
	63	484	17	4	1	<1	17	4	8	2	3	1	438	90
	64	584	12	2	-	-	9	2	8	2	5	1	550	94
	65	377	13	4	-		9	2	9	2	-		346	92
	66	456	13	3	3	1	15	3	5	1	5	1	415	91

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Table 24 - continued

SEDIMENT TYPE	SAMPLE NUMBER	SUM OF SEQUENTIAL PARTIAL EXTRACTIONS 1+2+3+4+5+6	MANGANESE CONCENTRATION (%) SODIUM HYPOCHLORITE (ORGANIC MATTER) #1	DI WATER-HYDROCHLORIC ACID (CARBONATE) #2	HYDROXYLAMINE HYDROCHLORIDE (AMORPHOUS MN HYDROXIDES) #3	ACID AMMONIUM OXALATE (AMORPHOUS FE HYDROXIDES) #4	HYDRAZINE CHLORIDE (XTALLINE HYDROXIDES) #5	HYDROFLUORIC-NITRIC-PERCHLORIC ACID (RESIDUAL DETRITAL) #6
	67	508	72 / 14	6 / 1	150 / 30	167 / 33	22 / 4	91 / 18
	68	271	22 / 8	3 / 1	8 / 3	15 / 6	6 / 2	217 / 80

1 / 2

1 metal concentration in ppm unless otherwise stated

2 metal concentration expressed as a % of sum

Sample Type	Sample Number	pH in Superjacent Water	Uranium Concentration in Sediment		
			Fulvic Acid	Humic Acid	Humin Fraction
Bog 2 Sediments	31	5.2	90 / 30	200 / 66	13 / 4
	31 (replicate)	5.2	105 / 32	218 / 66	4 / 1
	37	5.4	566 / 31	1212 / 67	26 / 2
	38	4.7	353 / 36	601 / 62	17 / 2
Lake 3 Sediments (adjacent to Bog 2)	39	6.1	47 / 31	86 / 56	20 / 13
	40	6.0	26 / 31	52 / 62	5 / 6
	41	5.8	21 / 24	63 / 71	5 / 5
	41 (replicate)	5.8	26 / 29	61 / 69	2 / 2
Lake 3 Sediments (distant to Bog 2)	43	6.3	20 / 29	18 / 26	31 / 45
	52	6.1	18 / 32	21 / 37	18 / 32
	53	6.4	20 / 34	17 / 29	22 / 37
	55	6.5	40 / 43	34 / 36	20 / 21
	56	6.5	15 / 44	9 / 26	10 / 29
	56 (replicate)	6.5	17 / 49	10 / 29	8 / 23

1 / 2

1 metal concentration in ppm
2 metal concentration expressed as % of sum of fulvic acid and humic acid and humin

III. Table 25 Uranium concentrations in fulvic acid, humic acid and humin in sediments

			Copper Concentration in Sediment		
Sample Type	Sample Number	pH in Superjacent Water	Fulvic Acid	Humic Acid	Humin Fraction
Bog 2 Sediments	31	5.2	10 / 18	20 / 35	27 / 47
	31 (replicate)	5.2	9 / 17	19 / 36	25 / 47
	37	5.4	5 / 9	33 / 58	19 / 33
	38	4.7	9 / 16	26 / 44	24 / 40
Lake 3 Sediments (adjacent to Bog 2)	39	6.1	12 / 24	20 / 39	19 / 37
	40	6.0	7 / 18	17 / 45	14 / 37
	41	5.8	9 / 17	23 / 44	20 / 38
	41 (replicate)	5.8	9 / 18	24 / 47	18 / 35
Lake 3 Sediments (distant to Bog 2)	43	6.3	11 / 20	17 / 31	26 / 48
	52	6.1	4 / 8	13 / 27	31 / 65
	53	6.4	7 / 13	18 / 35	27 / 52
	55	6.5	10 / 15	20 / 29	38 / 56
	56	6.5	4 / 6	16 / 22	52 / 72
	56 (replicate)	6.5	6 / 8	18 / 25	47 / 66

1 / 2

1 metal concentration in ppm

2 metal concentration expressed as % of sum of fulvic acid and humic acid and humin

III.Table 26 Copper concentrations in fulvic acid, humic acid and humin in sediments

			Zinc Concentration in Sediment		
Sample Type	Sample Number	pH in Superjacent Water	Fulvic Acid	Humic Acid	Humin Fraction
Bog 2 Sediments	31	5.2	19 / 35	10 / 18	26 / 47
	31 (replicate)	5.2	20 / 38	10 / 19	22 / 42
	37	5.4	14 / 56	6 / 24	5 / 20
	38	4.7	19 / 49	9 / 23	11 / 28
Lake 3 Sediments (adjacent to Bog 2)	39	6.1	54 / 43	19 / 15	53 / 42
	40	6.0	24 / 39	13 / 21	24 / 39
	41	5.8	21 / 33	9 / 14	33 / 52
	41 (replicate)	5.8	24 / 48	7 / 14	29 / 58
Lake 3 Sediments (distant to Bog 2)	43	6.3	19 / 20	11 / 11	66 / 69
	52	6.1	14 / 11	11 / 8	106 / 81
	53	6.4	12 / 14	6 / 7	67 / 79
	55	6.5	13 / 9	9 / 6	117 / 84
	56	6.5	5 / 4	3 / 3	109 / 93
	56 (replicate)	6.5	6 / 5	3 / 2	114 / 93

1 / 2

1 metal concentration in ppm
2 metal concentration expressed as % of sum of fulvic acid and humic acid and humin

III. Table 27 Zinc concentrations in fulvic acid, humic acid and humin in sediments

Sample Type	Sample Number	pH in Superjacent Water	Manganese Concentration in Sediment					
			Fulvic Acid		Humic Acid		Humin Fraction	
Bog 2 Sediments	31	5.2	104	55	33	17	52	28
	31 (replicate)	5.2	102	56	31	17	48	27
	37	5.4	61	43	24	17	57	40
	38	4.7	24	30	12	15	44	55
Lake 3 Sediments (adjacent to Bog 2)	39	6.1	153	55	36	13	89	32
	40	6.0	126	61	30	14	52	25
	41	5.8	119	46	42	16	98	38
	41 (replicate)	5.8	133	45	48	16	112	38
Lake 3 Sediments (distant to Bog 2)	43	6.3	190	35	83	15	267	49
	52	6.1	54	14	35	9	293	77
	53	6.4	78	24	32	10	216	66
	55	6.5	50	22	25	11	147	66
	56	6.5	91	15	29	5	501	81
	56 (replicate)	6.5	91	15	23	4	487	81

1 metal concentration in ppm
2 metal concentration expressed as % of sum of fulvic acid and humic acid and humin

III.Table 28 Manganese concentrations in fulvic acid, humic acid and humin in sediments