

ANALYSIS AND DISCUSSION OF FOUR TRACE METALS—COPPER,
LEAD, MERCURY, AND ZINC—TAKEN FROM SELECTED SITES
ALONG THE OTTAWA RIVER IN AND NEAR LIMA, OHIO.

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CONTENTS

	Page
Abstract.....	1
Introduction.....	2
Geology of the Local Area and Site Locations of the Eight Samples Taken.....	4
Local Geology.....	4
Sample Locations.....	4
Techniques of Study.....	8
Sampling Procedure.....	8
Sample Preparation.....	8
Atomic Absorption Process.....	10
Results.....	14
Possible Origins of the Trace Elements.....	18
Geological Pollution.....	18
Industrial and Waste Disposal Pollution.....	18
Chemical Pollution.....	19
Automobile Pollution.....	20
Desirable Levels of the Trace Elements in Humans and Water.....	22
Effects of the Trace Elements on Human Health.....	25
Copper Toxicity.....	25
Zinc Toxicity.....	25
Lead Toxicity.....	26
Mercury Toxicity.....	27
Discussion and Summary.....	29
Selected References.....	31

TABLES

Table	Page
I. Generalized Stratigraphic Sequence of the Rocks in Allen County, Ohio.....	7
II. Atomic Absorption Data for the Determination of Copper, Lead, Mercury, and Zinc in Both the Water and Sediment from Selected Sites in the Ottawa River.....	13
III. Trace Lead Content of Water and Sediment from Eight Sites in the Ottawa River in and near Lima, Ohio.....	14
IV. Trace Mercury Content of Water and Sediment from Eight Sites in the Ottawa River in and near Lima, Ohio.....	15
V. Trace Zinc Content of Water and Sediment from Eight Sites in the Ottawa River in and near Lima, Ohio.....	16
VI. Trace Copper Content of Water and Sediment from Eight Sites in the Ottawa River in and near Lima, Ohio.....	17
VII. Use of Copper, Lead, Zinc, and Mercury by Some of the Principle Industries in and around Lima, Ohio.....	21
VIII. Recommended Limits in Finished Water Supplies as Established by the U.S. Public Health Service.....	23

MAPS

Map		Page
I.	Map of Ohio Showing the Location of the Sampling Area Where Eight Water and Sediment Samples Were Taken from the Ottawa River in and near Lima, Ohio.....	6
II.	Location of Sample Sites along the Ottawa River.....	back pocket
III.	Location of Samples C, D, E, and F Within the City Limits of Lima, Ohio..	back pocket

ABSTRACT

Eight water and sediment samples collected from the Ottawa River in and near Lima, Ohio, were analyzed for their content of four trace metals—copper, lead, mercury, and zinc—by the atomic absorption process. The four following possible origins of these trace metals were considered: geological pollution, industrial and waste disposal effects, chemical pollution, and automobile exhaust emissions. A discussion as to the desirable levels that have been established for public water supplies and the trace metal requirements of the human metabolism is presented. The effects of these trace elements on the human metabolism is also considered, as copper and zinc are believed to be essential to human health but can be toxic, and lead and mercury are not important in the body's biochemical system but are considered cumulative poisons.

INTRODUCTION

Since before the birth of Christ man has recognized the fact that many trace substances can be detrimental to health, both by toxicity and deficiency. However, for centuries there was a lack of knowledge as to the origins of many of these trace elements and their functions in animal and plant metabolisms. The importance of trace substances in the human environment was finally recognized at the beginning of this century when Sir Frederick Hopkins promoted the idea of "accessory food factors" which we now label as vitamins. Efforts made during the last two centuries to understand and control the environment for the common good have resulted in spectacular changes in the life of the individual and the community, and the whole organization of society. Yet, man's rapid increase in industrialization and urbanization and the resulting increase in chemical pollution and ecological disruption has left him with a lack of understanding of the effects of these pollutants on his environment, as well as his health. Until recent years man had no sensitive and accurate analytical methods for the detection of trace substances, and subsequently, few studies were conducted to test the effects of trace elements on the human metabolism or to detect them in his environment.

A variety of trace elements occur in man's ecological system. Many are essential to the sustenance of both

plants and animals, some are known to be of no benefit, and the roles of others are questionable. On the other hand, all of them can be toxic when consumed in sufficient amounts. This study deals only with the detection of four of these trace elements—copper, zinc, lead, and mercury. Copper and zinc are considered to have an essential role in the body's biochemical process; lead and mercury are not known to have any essential role. The study involves the detection of these elements in eight samples of both water and sediment taken from selected areas along the Ottawa River as it passes through Lima, Ohio. An emphasis will be placed on the possible origins of the trace elements, their desirable levels in humans and water, and their effects on human metabolism.

A much more complete study of this kind is currently being conducted by Mr. Tom Schultz, a graduate student at the Ohio State University, under the direction of Dr. Wayne Pettyjohn, professor of Geology at the Ohio State University, both of whom have been invaluable aids to me in the research completed for this text.

GEOLOGY OF THE LOCAL AREA AND SITE LOCATIONS
OF THE EIGHT SAMPLES TAKEN

LOCAL GEOLOGY

The sampling area (see Map I) is a lowlying glaciated area covered with glacial deposits of clay, sand, and gravel. The bedrock surface beneath these deposits consists of carbonate rocks, mostly dolomitic limestones. However, small areas are underlain by shale or interbedded shales and thin limestones. A generalized stratigraphic sequence of the rock underlying the sample area is presented in Table I.

SAMPLE LOCATIONS

The following are the eight sample locations along the Ottawa River from which both water and sediment were taken. An overall picture of these locations can be seen on Maps II and III located in the envelope attached to the inside back cover of this folder.

1. Sample A is from a site east of Lima near the headwaters of the Ottawa River where Hog Creek intersects the river. The location is approximately 1.2 miles north of Lafayette, Ohio, off State Route 81.
2. Sample B is from a site east of Lima at the intersection of State Route 81 and Thayer Road where the two highways pass over the river.

3. Sample C is from a site located within the Lima city limits at the bridge on North Sugar Street that crosses the river approximately .75 miles west of Interstate 75 and about one mile from downtown Lima.
4. Sample D is from a site one hundred yards east of the intersection of North Pine Street and the Ottawa River, about .25 miles from the center of Lima.
5. Sample E is from a site fifty yards southeast of the intersection of North Shore Drive and South Metcalf Street, approximately .50 miles from the center of Lima.
6. Sample F is from a site at the intersection of the Erie Railroad overpass and the Ottawa River, behind Woodlawn Cemetery which is located in the southwest part of the city.
7. Sample G is from a site west of Lima, approximately .20 miles north of Allentown and fifty yards west of the Allentown-Elida Road.
8. Sample H is from a site west of Lima, approximately .30 miles northwest of Elida, Ohio, at the intersection of State Route 305 and the Ottawa River.



Map I. Map of Ohio showing the location of the sampling area where eight water and sediment samples were taken from the Ottawa River in and near Lima, Ohio.

System or Series		Group or Formation	Character of the Material
Quaternary	Recent		Clay, silt, fine sand and gravel deposited on flood plains of the major valleys.
	Pleistocene		Sands and gravels interbedded with less permeable glacial material.
			Till, largely clay, with buried sand or gravel beds in some places.
Devonian	Ohio		Hard; dense, carbonaceous shale.
	Columbus		Dolomitic limestone.
Silurian	Bass Is.		Massive to thin-bedded dolomite.
	Niagara		Massive to thin-bedded limestones and dolomites with shale beds.
	Clinton		Massive to irregularly-bedded limestone.
Ordovician	Richmond and Maysville		Interbedded soft shales and thin, hard limestones.

Table I. Generalized Stratigraphic Sequence of the rocks in Allen County, Ohio.

TECHNIQUES OF STUDY

SAMPLING PROCEDURE

The samples were collected from the Ottawa River in and near the city of Lima, Ohio, on February 5, 1972. An attempt was made to collect a range of samples from areas upstream, downstream, and in the city itself. At this time the river was almost completely frozen over with a layer of ice five to six inches thick. A hole was chipped in the ice, and immediately a plastic bottle was filled with the river water. Then, a pole approximately six feet long with a holder attached, allowing a large-mouth plastic bottle to be fastened to it, was lowered through the hole and the sediment sample taken. This invention of mine allowed sampling of the sediment in approximately five to six feet of water. Finally, the sample was marked with its location and readied for transport to the Ohio State University where preparation of the samples and analysis on the Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer was conducted during the week of March 19, 1972.

SAMPLE PREPARATION

In order to ascertain the trace element content of the eight sediment samples, two extracting agents were used. The first was ammonium acetate which was used to extract those trace ions that would be readily available to the

environment in a short amount of time. The second was a stronger extracting agent of hydrochloric acid and sulfuric acid which would extract a greater amount of trace metal ions reflecting the amount which might be available to the environment over longer periods of exposure.

The following procedure was used in the preparation of each of the eight sediment samples for analysis by the atomic absorption spectrophotometer:

1. The sample was stirred for three minutes, and the resulting solution of suspended particles and water was divided into two petri dishes and two plastic test tubes which had been weighed and marked.
2. The test tubes and petri dishes were weighed and the weight noted.
3. The petri dishes were placed in an oven for twenty-four hours and then weighed.
4. The weight of the dried sediment was divided by the weight of the undried sediment and the resulting percentage multiplied by the weight of the contents in each test tube in order to obtain the actual dry weight of the sediment in each test tube.
5. Approximately 30 ml. of a one mole solution of ammonium acetate was added to the contents of one test tube, and the same amount of the extractant

containing 0.05 mole of hydrochloric acid and 0.025 mole of sulfuric acid was added to the other.

6. The test tubes were capped and placed in a shaker for thirty minutes.
7. The test tubes were then placed in a centrifuge for fifteen minutes.
8. The liquid was then poured off into two 100 ml. flasks—one designated "X" for the ammonium acetate extractant and one designated "Y" for the hydrochloric and sulfuric acid extractant.
9. Steps 5, 6, 7, and 8 were repeated twice.
10. The solutions were poured into plastic bottles and marked with their sample location and the type of extractant used.

No modification of the eight water samples was necessary, and consequently, they were aspirated directly into the atomic absorption unit for analysis.

ATOMIC ABSORPTION PROCESS

A short discussion of the atomic absorption process is essential for a proper appreciation of the value of the general method.

When a vapor containing free atoms of an element is illuminated by a light source that radiates light of a frequency characteristic of the element present in the vapor, the free atoms can absorb the resonant frequency.

The atom absorbs a photon of light, and the energy of the photon brings the atom to an excited state. The net energy of this state is equivalent to the energy of the absorbed photon. Each species of atom can exist only in certain excited states with definite energies, and only photons of definite energies can be absorbed. Photons absorbed by the atoms are only those with wavelengths corresponding to the lines of the atomic absorption spectrum of the element. Thus, by measuring the amount of absorbed light, a determination of the concentration of an element in a vapor can be made.

The analysis of the samples was conducted on the Perkin-Elmer Model 303 Atomic Absorption Spectrophotometer using an air-acetylene mixture as fuel for the flame. A one mole solution of ammonium acetate was used as a blank for those samples which were extracted with the ammonium acetate, and a mixture containing 0.05 mole hydrochloric acid and 0.025 mole sulfuric acid was used as a blank for the other solutions. Doubly distilled demineralized water was used as a blank for the water samples. The slit sizes, wavelengths, flame colors, noise suppressions, scale expansions, and standards used for the determination of each element are listed in Table II.

The methods of calculating the parts per million in solution and in the dry sediment will not be discussed here, but may be obtained from a publication, number 303-0152 "Analytic Methods for the Atomic Absorption Spectrophoto-

meter," published by the Perkin-Elmer Corporation.

The findings as to the trace metal content for lead, mercury, zinc, and copper are listed in Tables III, IV, V,, and VI, respectively.

*due to the large concentrations of zinc in some samples a scale expansion of 3 had to be used.

Element	Slit Size	Wave-length	Flame Color	Noise Suppression	Scale Expansion	Standards mg/l
Cu	4	3274.0	light blue	5	30	0.1 & 0.05
Pb	"	2833.1	"	"	10	0.1 & 0.5
Hg	"	2536.5	"	"	30	2.5 & 1.0
Zn	"	2139.0	"	"	10 & 3*	0.1 & 0.05

Table II. Atomic absorption data for the determination of copper, lead, mercury and zinc in both the water and sediment from selected sites in the Ottawa River.

*trace—detectable amount but not measurable

Sample Number	River Water	River Sediment			
		ammonium acetate extracting agent		hydrochloric and sulfuric acid extracting agent	
		mg/l in solution (ppm)	mg/l in dry sediment (ppm)	mg/l in solution (ppm)	
A	0.000	0.044	0.412	0.062	0.573
B	"	0.000	0.000	0.068	0.668
C	"	0.477	25.368	0.187	29.641
D	"	0.129	1.013	0.199	1.137
E	"	0.056	1.820	0.195	6.388
F	"	0.077	22.039	0.703	200.985
G	"	0.056	4.587	0.201	16.646
H	"	0.075	2.442	0.129	4.231

Table III. Trace lead content of water and sediment from eight selected sites in the Ottawa River in and near Lima, Ohio.

*trace—detectable amount but not measurable

Sample Number	River Water	River Sediment			
		ammonium acetate extracting agent		hydrochloric and sulfuric acid extracting agent	
		mg/l in solution (ppm)	mg/l in dry sediment (ppm)	mg/l in solution (ppm)	
A	0.000	1.405	13.025	1.491	13.821
B	"	1.000	9.823	1.093	10.733
C	"	0.634	33.700	2.846	151.366
D	"	1.405	10.996	2.591	20.271
E	"	0.754	24.733	1.907	62.538
F	"	trace*	trace	0.533	152.263
G	"	1.076	85.195	1.979	163.514
Hi	"	0.879	28.732	1.031	33.688

Table IV. Trace mercury content of water and sediment from eight selected sites in the Ottawa River in and near Lima, Ohio.

*trace—detectable amount but not measurable

Sample Number	River Water	River Sediment		
		ammonium acetate extracting agent	hydrochloric and sulfuric acid extracting agent	hydrochloric and sulfuric acid extracting agent
	mg/l in solution (ppm)	mg/l in dry sediment (ppm)	mg/l in solution (ppm)	mg/l in dry sediment (ppm)
A	trace*	0.051	0.286	2.655
B	"	0.051	0.334	3.276
C	0.002	0.122	0.214	11.369
D	trace	0.187	1.015	7.943
E	0.002	0.471	0.649	21.294
F	0.004	0.332	4.710	1345.660
G	0.113	0.091	0.405	33.452
H	0.059	1.854	3.774	123.338

Table V. Trace zinc content of water and sediment from eight selected sites in the Ottawa River in and near Lima, Ohio.

*trace—detectable amount but not measurable

Sample Number	River Water	River Sediment		
		ammonium acetate extracting agent	hydrochloric and sulfuric acid extracting agent	hydrochloric and sulfuric acid extracting agent
	mg/l in solution (ppm)	mg/l in dry sediment (ppm)	mg/l in solution (ppm)	mg/l in dry sediment (ppm)
A	0.000	0.028	0.043	0.394
B	"	trace*	0.013	0.130
C	"	"	0.006	0.298
D	"	0.011	0.021	0.164
E	0.002	trace	0.010	0.325
F	0.000	"	0.014	0.843
G	"	0.028	0.037	4.181
H	0.011	0.010	0.016	0.444

Table VI. Trace copper content of water and sediment from eight selected sites in the Ottawa River in and near Lima, Ohio.

POSSIBLE ORIGINS OF THE TRACE ELEMENTS

GEOLOGICAL POLLUTION

Much of the trace element content in the water and sediment of a stream can be attributed to the surrounding parent rock. It has been suggested that geological pollution is quantitatively the greatest contributor to abnormal amounts of trace substances in soils. Thus, when the surrounding soil is eroded and washed into the local stream, the trace elements become incorporate with the stream sediment.

In a study conducted by Warren, Delavault, and Cross in Canada, the following values were considered normal in soils: copper ranging from 5 to 70 ppm (parts per million); zinc, from 35 to 300 ppm; lead, from 2 to 10 ppm; and for mercury less than .01 ppm.

Samples of the limestone bedrock underlying the northwestern part of Ohio were analyzed for trace metal content by Birsa, and concentrations of copper ranging from 7.05 to 11.4 ppm and of lead ranging from 27.1 to 41.6 ppm were found with an average of 8.01 ppm and 33.2 ppm respectively.

INDUSTRIAL AND WASTE DISPOSAL POLLUTION

Even before the beginning of the Industrial Revolution in the nineteenth century, man used his waterways as a means of transporting waste material from around him. The

city of Lima is characterized by a variety of industries, many of which use copper, lead, mercury, and zinc in some way or another. No attempt will be made to single out any one industry as a polluter; yet, the majority of these firms are potential polluters. The discharge of effluent containing these trace metals either by accident or with full knowledge of the company, along with such possibilities as leakage from holding ponds, waste disposal pits, pipes, tanks, or sewers could cause a marked increase in the trace element content of the Ottawa River.

The air is another means of transporting trace metal pollution. The burning of coal and the possibilities of the trace metals in the ashes and soot from industries in the area could easily act as a source for trace substances in the river.

Table VII has been prepared to show those elements that are being used by some of the principle industries in and around Lima, Ohio. This table is only an attempt to show the variety of uses of the four elements studied and not an accusation of who is responsible for any pollution.

CHEMICAL POLLUTION

The area bordering the Ottawa River outside the city of Lima is almost all farm land, and everything from corn to soybeans is cultivated. The use of agricultural chemicals to combat plant deficiencies and diseases and

also to control harmful insects is probably responsible for some of the trace metal pollution in the river. The elements are incorporated into the soil after spraying, and because of erosion and runoff, they are subsequently carried towards the river.

AUTOMOBILE POLLUTION

Recently much attention has been given to the fact that the use of tetraethyl lead in gasoline can give rise to marked pollution in areas near heavily traveled highways. The vicinity of Lima, Ohio, is crisscrossed by many major and minor highways (not unlike any other part of the United States) and, therefore, high concentrations of lead may be expected. How much pollution is caused is questionable, as no facts and figures are available to date.

INDUSTRIES	Cu	Pb	Zn	Hg
Manufacturers of electrical goods	X	X	X	X
Manufacturers of power shovels, locomotives and aircraft parts	X	X	X	X
Oil refineries		X		
Manufacturers of rubber goods		X		
Manufacturers of automobile bodies	X	X		X
Manufacturers of enamel goods		X		
Manufacturers of agriculture chemicals	X	X	X	
Manufacturers of electroplated goods	X		X	

Table VII. Use of copper, lead, zinc and mercury by some of the principle industries in and around Lima, Ohio.

DESIRABLE LEVELS OF THE TRACE ELEMENTS
IN HUMANS AND WATER

As early as 1914 the Public Health Service adopted and published the first drinking water standards to protect the health of the traveling public. These standards have been revised many times since, but no exact levels for the trace elements have been established, mainly because of the lack of knowledge surrounding their effects. The establishment of normal or safe levels involves many factors. One is the fact that different species of plants and animals may differ widely in their sensitivity or tolerance to different trace elements. With regard to agriculture, the variations and interactions of water, climate, plants, and soils preclude the establishment of a simple set of standards to evaluate all water quality characteristics. Industries also have various requirements which will also affect the establishment of water quality criteria.

The recommended limits established by the United States Public Health Service are for finished water and not for the raw water supply. However, the presence of trace substances in the finished water is directly related to the trace element content of the raw water supply; thus, the defined treatment processes generally do not remove the trace metals. In addition, all established water quality criteria ignore the possibility of trace elements in the stream sediment where most trace metals

accumulate. The recommended limits in finished water supplies of the four elements with which this study deals, as proposed by the United States Public Health Service, are listed below in Table VIII.

*the U.S. Department of Health has established a mercury tolerance rating of zero in water.

Element	Permissible Criteria mg/l	Desirable Criteria mg/l
copper	1.0	virtually absent
lead	0.05	absent
mercury	none	none
zinc	5.0	virtually absent

Table VIII. Recommended limits in finished water supplies as established by the U.S. Public Health Service.

Of the four metals studied, only copper and zinc are considered to be essential to most plants and animals. The only standards that have been established are those for the amount necessary to prevent copper deficiency. The amount varies with the species: for man the normal copper requirement of the body as determined by balance studies has been found to be about 2 mg. a day, and it has been estimated that the adult body contains between 100 and 150 mg. of copper. The normal zinc requirement

has been established at about 1.5 mg. a day with 2.2 g. being the estimated content in the adult body.

EFFECTS OF THE TRACE ELEMENTS ON HUMAN HEALTHCOPPER TOXICITY

Human metabolism is dependent on copper; it is essential for pigmentation, and it is an integral part of several proteins. However, the human metabolism may be grossly disturbed by too much copper. The amount of copper in the human system increases in acute as well as chronic infectious diseases. It has been found that in cases of acute leukemia, extremely high values for whole blood, plasma, and cell copper content have been observed. Increases in copper content of blood have also been observed in cases of malignancy, Hodgkin's disease, certain types of anemia, various types of liver disease, schizophrenia, and Wilson's disease.

An interesting fact about Wilson's disease is that the disorder is inherited as a recessive trait and shows that man has had to develop a mechanism which prevents copper poisoning. Man is able to maintain a net-zero balance of copper, despite an intake of 2 to 5 mg. of it daily.

ZINC TOXICITY

Man's metabolism is dependent on zinc for the formation of RNA and certain proteins, and zinc is essential for the activity of certain metallo-enzymes. It is also considered important in the bone healing processes and for

the treatment of atherosclerosis.

Even though zinc toxicity is not common, the human metabolism can be greatly upset by abnormal amounts. An interesting fact is that certain manifestations of zinc toxicity resemble those which are characteristicly a result of zinc deficiency, namely anemia and subnormal growth which will eventually lead to death.

The interest in zinc as a toxic agent arises principally from its interactions with other trace elements, notably copper. Zinc by itself is relatively nontoxic to humans, but the competitive zinc-copper antagonism illustrates the danger in abnormal zinc consumption. Zinc can cause a reduction of either the absorption of copper from the gastrointestinal system or the utilization of copper within the tissues, or both. It has also been observed that with high levels of zinc in the diet, the amount of copper in the liver is greatly reduced, suggesting that the accumulation of zinc is displacing the copper.

LEAD TOXICITY

Lead is a cumulative poison, and the absorption of minute quantities over a long period of time may produce disastrous results to the human metabolism. The poisonous effects of lead were recorded as early as the fifth century B.C. by Hippocrates. It has been suggested that the use of lead cups and pipes by the Romans may have been

a significant factor in the degeneration of the Roman Empire. The symptoms of lead poisoning are pain in the abdomen, constipation, loss of appetite, thirst, a nervous prostration known as lead palsy, epileptic fits, and total paralysis.

Most people become tolerant of lead to some degree, and a balance between absorption and secretion is developed. This balance may be upset by undernourishment, disease, or by a sudden increase in absorption of the poison.

Lead is distributed among the tissues of the body in a manner which suggests that its presence may be the result of contamination and that it probably has no essential role in the human metabolism.

MERCURY TOXICITY

Mercury, like lead, is considered a cumulative poison. Chronic mercury poisoning results from the absorption or ingestion of small amounts of mercury over extended periods of time.

The effects of mercury poisoning were realized many years ago. The expression "mad as a hatter" was coined in the nineteenth century when a high incidence of hatmakers became mentally ill because of their continual contact with mercury used in treating felt. Between 1953 and 1960 approximately one hundred and ten people died or were

disabled in Japan after eating fish caught in Minamata Bay, into which a plastics plant had been discharging mercury. In 1969 a discovery that many of the streams and lakes of the United States contained mercury brought an upsurge of attention to the possible health effects of mercury.

The symptoms of mercury poisoning are loss of appetite, salivation, gingivitis, nutritional disturbances, increasing renal damage and anemia. Even very slight amounts of mercury can cause headaches and numbness and possibly impairment of speech and vision or even severe emotional disturbances.

Tests by the Japanese have shown that the mercury tolerance levels vary from one individual to another, and in cases of pregnant women, the mercury concentrates in the unborn child, resulting in an increase in nervous disorders and deformation. Mercury is distributed among the human tissues in such a haphazard manner as to suggest that it is not essential in human metabolism.

DISCUSSION AND SUMMARY

The results of this study show that none of the eight water samples exceeded the water quality criteria as established by the United States Public Health Service. The trace copper content was found lower than expected in view of the bedrock trace copper content data supplied by Birsa and those figures considered normal in soils by Warren, Delavault and Cross. Lead was found to be in excess of 200 ppm at one location which is twenty times that considered normal in soils and approximately seven times greater than the average content that has been found in the bedrock. The range of the trace lead content in the sediment may indicate some contributing factor other than geological pollution, especially the sample containing over 200 ppm. The mercury content of the river sediment is very high with three samples showing more than 150 ppm and two others with more than 60 ppm. These values are extremely high if compared to the amount considered normal in soils by Warren and his associates and are probably due to industrial and/or chemical pollution. The trace zinc content for seven of the eight samples fell within the range considered normal by Warren et al, but a surprising amount, greater than 1300 ppm, was discovered in one sample which definitely indicates that an abnormal amount of trace zinc pollutants have been contributed at some time.

Due to the limited scope of this study, it would be unfair to speculate as to who was responsible for any of these abnormal amounts of trace metals in the Ottawa River. A much greater number of samples would be needed, and only then could the source of the pollutants be narrowed down to any particular cause; even under these conditions a danger of making specific accusations exists, in view of the fact that the origins of trace elements might still overlap.

More studies need to be made, but in order to assure that studies of this kind are properly conducted, there must be some sort of certified and standardized method of study established. There is also a need for studies involving the movement of trace substances in the environment, the absorption of trace metals by different plants and animals, the interaction of the trace metals among themselves, and the long-term effects of the trace pollutants on man's ecological system. Furthermore, there is a need for an established limit as to the trace metal content of stream sediment, as well as that of soils, plants, and animals. Until action has been taken in these areas, studies such as mine cannot be expected to provide answers but merely to provide leads for future critical investigations.

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