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# Elemental Analysis of Waters and Sediments by External Beam PIXE

## Part 1. Vegoritis Lake, Greece†

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Lake Vegoritis, in the northwestern part of Greece, accepts the pollutants of the municipal and industrial activities in the area (lignite burning power plants and a nitrogen fertilizer factory). The PIXE external beam technique was used for the elemental analysis of water and sediment samples in order to determine the load of metals in the lake and obtain information for managing its environment. From our results it is deduced that the anthropogenic activities in the drainage basin do not have so far any significant impact on the Vegoritis Lake, as far as the elements studied are concerned. However, there is indication that other pollutants are introduced, mainly along the Solou stream, which cause precipitation of the metals.

## 1 INTRODUCTION

During the last two decades, a large number of research projects dealing with the analysis of inland water columns and sediments have been carried out (Bradford *et al.*, 1968; Shapiro *et al.*, 1971; Bruland *et al.*,

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1974; Kemp *et al.*, 1976; Förstner, 1976; Golterman, 1977; Förstner and Wittmann, 1979). The above projects were conducted for the purpose of determining the extent of metal pollution of the water bodies. Along with the anthropogenic contributions through municipal, agricultural and industrial effluent discharges, the natural sources of metal contribution are also taken into account.

Metals usually tend to accumulate in the sediments and from these, they may be released into water soluble forms through various processes of remobilization and attain the bioavailable form that can move up the biologic chain and reach the human beings.

The metal content of various lakes and rivers in Greece has not been studied until now. This study has been undertaken in order to obtain baseline information on the levels of accumulated metals in water columns and sediments of the Vegoritis Lake. This lake was selected as the study area, because it is the largest Greek lake, located in a relatively heavily industrialized and populated area used as the receiver of industrial and municipal effluents as well as a resort for recreational activities by Greeks and Yugoslavs.

Our previous studies (Mourkides *et al.*, 1978) have shown that part of the lake is highly polluted with ammonia due to the nitrogen fertilizer factory waste effluents, a fact that has lowered seriously the yield of the fish farming. Therefore it is important to determine all the pollutants, and to pinpoint all the pollution sources, so that they may be controlled and further disruption of the lake ecosystem be prevented.

The Proton Induced X-ray Emission (PIXE) method was used for the elemental analysis of the samples (Katsanos, 1980; Cahill, 1980). Since this method detects automatically and simultaneously all heavy elements, it was not necessary to predetermine which elements to look for.

## 2 EXPERIMENTAL

### 2.1 Site Description

Vegoritis, also known as Ostrovou Lake, is a warm oligotrophic lake (Mourkides *et al.*, 1978) changing to oligomesotrophic (Mourkides and Tsicritsis, unpublished data 1982). It is located in northwestern Macedonia (Figure 1); the geographical and other parameters are shown in Table I. It is the biggest lake in Greece, surrounded by a relatively heavily industrialized area. The area is rich in lignites and several coal-

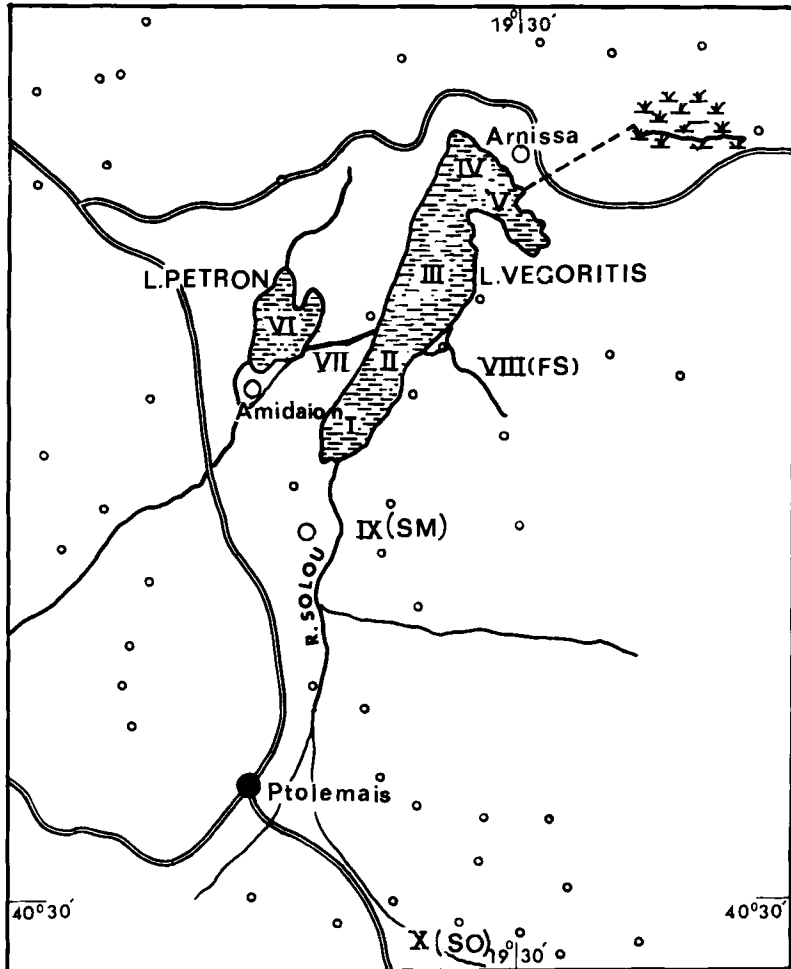


FIGURE 1 Map of the area around Vegoritis Lake.

fired electricity power plants have been established. Five sampling stations were chosen in the lake as shown in Figure 1, along with some additional stations in the connecting canals.

*Site I* This site is an inlet just off the mouth of the Solou stream. The Solou stream accepts the industrial and municipal waste of the surrounding area. During the last decade five factories have been in operation along the Solou stream. One is a nitrogen fertilizer producing

TABLE I  
Parameters of the Vegoritis Lake.

Latitude	40° 45'
Longitude	21° 47'
Sea level	520 m
Surface Area	60 km
Depth, max	55 m
Length, max	15.5 km
Width, max	6.4 km
Lake water pH	8-9

plant, one is a lignite mine, one is a lignite processing unit, and two units are coal-fired electric power plants. Among these units, the lignite mine plant is the only one which treats its wastes before discharging them into the Solou stream. One of the power plants pumps out water directly from the lake for its needs and discharges the waste into the Solou stream. The main source of municipal effluents is Ptolemais, a town of 25000 population. On the other hand, the stream Chrystophorou (CS), an unpolluted stream, discharges also its water into the Solou stream, thus diluting the pollutants.

*Site II* It is located across the mouth of the Farangi stream (FS) and a short canal that connects the small Petron Lake with the Vegoritis Lake. The FS collects mountain spring water and discharges it directly into the lake during the spring and summer months. The metal concentrations in the Farangi stream water are taken as the levels of lithogenic source.

*Site III* This site is located in the center of the Vegoritis Lake.

*Sites IV and V* These sites are located at the upper end of the Vegoritis Lake as indicated in Figure 1.

*Site VI* This site is located in the center of lake Petron. Lake Petron is the receiver of the municipal untreated sewage of Amyntaion, a town of 15000 people. During spring time and most of the summer its water passes through a canal to the Vegoritis Lake. This small lake contributes to the load of metals of Lake Vegoritis, and its status is an indication of the municipal pollution.

*Site VII* A site in the canal connecting Lake Petron with Lake Vegoritis.

*Site VIII* Farangi stream, considered as natural unpolluted water of lithogenic origin.

*Site IX* This station at Solou mouth (SM) has been chosen for monitoring the industrial pollutants which are collected by the Solou stream, just before entering the lake.

*Site X* Sampling at the origin of Solou stream (SO).

## 2.2 Sample Collection

From each of the first five stations surface and bottom water samples were taken, while surface water was also collected from stations VI to X. Grab samples of sediment were collected using an Ekman dredge sampler from sites I to VI and IX. The samples were emptied into small plastic storage bottles and transported to the laboratory for analysis. Polyethylene bottles were used for the collection and transportation of the water samples. All samples (water, sediment) were transported in an ice-box.

## 2.3 Sample Preparation

The Proton Induced X-ray Emission (PIXE) method with external beam was used for the elemental analysis of the samples (Katsanos *et al.*, 1976 and 1978; Khan *et al.*, 1979). For this purpose the samples were treated as follows.

A portion of the sediment samples was dried at 120°C and a small amount (about 30 mg) from each was pressed into a pellet for the irradiation with protons.

For each water sample, a subsample of 250 ml was placed into a preweighed beaker, mixed with one ml of standard molybdenum solution and evaporated slowly (avoiding boiling) to dryness. The dry residue was then weighed, collected with a plastic spatula and about 30 mg of it was pressed into a pellet for irradiation. Molybdenum was used as the internal standard for checking the homogeneity of the dry residue and any possible loss during the evaporation of the samples.

## 2.4 PIXE Analysis

The samples were analysed at the Tandem van de Graaff accelerator of the NRC DEMOCRITOS. The PIXE set up with the external beam has been successfully developed in this laboratory (Katsanos *et al.*, 1976; Katsanos, 1978; Kahn *et al.*, 1979). A thin kapton foil (1 mg/cm<sup>2</sup>) was used as an exit window for the 2.5 MeV protons. The thick pellets

were placed at 45° relative to the incoming beam. The proton current (10–50 nA) was integrated at the window and the target, as described previously (Kahn, 1979). A Ge(in) detector with working resolution of about 180 eV, standard electronics for amplification, pile-up rejection and pulse analysis were used.

The Proton Induced X-ray Emission method with external beam offers many advantages, such as small sample requirements, simultaneous detection of all elements present (major and trace elements) with one irradiation, high sensitivity, good accuracy and precision.

### 3 RESULTS AND DISCUSSION

#### 3.1 Results

Distribution patterns of element concentration in a lake provide valuable clues as to the existence of a changing geochemical status in a drainage basin.

For the determination of the extent of pollution in a lake by means of the metal load in its water or sediment, it is of primary importance to establish the natural levels of these substances in the water body and in the sediment. The difference between these normal concentrations and the determined ones will show the total enrichment that has been caused by the anthropogenic influences. Förstner and Wittmann (1979) have reported a valuable compilation of such normal background values. This information will be used to some extent in the following discussion, along with the data, for reasons of comparison.

However, due to large variations which are observed in waters of different origin, the above mentioned levels can only be partially used, while one could probably derive better conclusions if the variations of the elemental concentrations at different sites of the region are studied. Especially useful could prove differences between the values of the concentrations at the origin of the streams, at points passing from industrial areas up to and the lake.

Thirteen elements (Ca, Ti, V, Fe, Mn, Cu, Pb, Zn, Ni, Sr, Cr, As, Br) have been detected and determined in the water samples from nine stations, whereas in the sediment samples the following fifteen elements have been determined: Ca, Ti, V, Fe, Mn, Cu, Pb, Zn, Ni, Sr, Cr, Ga, Rb, Y, Zr.

Table II summarises the results of the analysis for the lakes Vegoritis and Petron. Table III presents the ranges of the elemental concentrations in Vegoritis, the concentrations of the inflow streams and canal,

TABLE II  
Elemental concentrations in the water of lake Vegoritis. 'A' stands for surface water and 'B' for bottom. The solid residue (RES) and Ca are in ppm, all other elements in ppb. The errors are analytical standard deviations.

SITE	RES	Ca	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	As	Br	Sr	Pb
IA	221	19 ± 2	2 ± 1	0.7 ± 0.3	0.7 ± 0.4	4 ± 0.4	0.7 ± 0.3	0.9 ± 0.3	0.9 ± 0.3	14 ± 1	1.0 ± 0.3	55 ± 3	123 ± 10	0.2 ± 0.2
IIA	233	18 ± 2	5 ± 2	3 ± 2	1.6 ± 0.5	0.5 ± 0.3	3 ± 0.3	0.7 ± 0.3	0.5 ± 0.2	7 ± 1	0.9 ± 0.3	52 ± 3	82 ± 6	2.3 ± 1.0
IIIA	209	18 ± 2	4 ± 2	1 ± 1	0.6 ± 0.3	0.4 ± 0.3	30 ± 2	0.4 ± 0.2	0.4 ± 0.2	34 ± 2	0.4 ± 0.1	54 ± 3	110 ± 10	2.0 ± 1.0
IVA	200	20 ± 2	1 ± 0.5	2 ± 1	0.2 ± 0.2	0.4 ± 0.3	8 ± 0.6	0.4 ± 0.2	0.6 ± 0.3	3 ± 1	0.6 ± 0.2	52 ± 3	128 ± 10	0.8 ± 0.5
VA	202	28 ± 2	4 ± 2	3 ± 2	0.2 ± 0.2	nd	6 ± 0.5	0.6 ± 0.3	0.8 ± 0.3	9 ± 1	nd	41 ± 3	86 ± 6	2.0 ± 1.0
IB	214	16 ± 1	1 ± 0.5	3 ± 2	nd	0.9 ± 0.4	6 ± 0.5	0.9 ± 0.4	0.1 ± 0.1	6 ± 1	1.3 ± 1	63 ± 4	120 ± 10	0.6 ± 0.3
IIIB	253	26 ± 2	3 ± 2	3 ± 5	0.5 ± 0.3	0.8 ± 0.4	16 ± 12	0.3 ± 0.2	0.5 ± 0.2	18 ± 2	1.6 ± 2	60 ± 3	138 ± 13	1.2 ± 0.5
IIIB	237	14 ± 2	2 ± 1	4 ± 2	1.0 ± 0.4	0.5 ± 0.3	5 ± 0.5	1.4 ± 0.5	1.2 ± 0.4	7 ± 1	2.1 ± 2	67 ± 4	83 ± 6	2.4 ± 1.0
IVB	200	24 ± 2	9 ± 2	5 ± 2	0.4 ± 0.3	2.4 ± 0.6	90 ± 5	1.2 ± 0.4	1.6 ± 0.5	19 ± 2	1.0 ± 0.3	48 ± 3	146 ± 12	0.4 ± 0.2
VB	182	28 ± 2	1 ± 0.5	2 ± 1	nd	nd	40 ± 3	1.6 ± 0.5	0.4 ± 0.2	46 ± 3	1.2 ± 0.3	46 ± 3	131 ± 10	0.4 ± 0.3

TABLE III  
Elemental concentrations in water of the Vegoritis area, background levels by Föstner and Wittmann, and ranges in river waters by Habib-Minski. 'A' stands for surface water, 'B' for bottom. The solid residue and Ca are in ppm, all others in ppb.

RESIDUE	Ca	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn	As	Br	Sr	Pb
LAKE { A	200-233	1-5	1-3	0.2-1.6	0-0.7	3-30	0.4-0.7	0.4-0.9	3-34	0-1	41-55	82-123	0.2-2.3
RANGE { B	182-253	1-10	2-5	0-1.0	0-2.4	5-90	0.3-1.6	0.1-1.6	6-46	1-21	46-67	83-146	0.4-2.4
CANAL	343	7	nd	0.7	1.7	14	1.4	0.3	2	0.3	69	72	0.7
PETRON	305	24	17	nd	20.0	265	3.0	1.5	40	1.2	50	156	4.0
FARANGI	177	53	14	6	10.0	354	0.3	1.0	19	2.0	10	121	2.3
SO	336	67	1	10	7.7	3.4	16	4.0	nd	6	13.0	9.0	246
SM	282	45	2	10	1.4	2.2	22	2.8	0.2	17	8.0	9.0	184
FÖSTNER		<1	0.9	0.5	<5	<30	0.3	1.8	10	2.0	—	50	0.2
HABIB { MIN	2	3	0.01	0.1	0.02	10	0.02	0.2	0.2	0.2	0.05	3	0.06
MAX	120	18	20	6	130	1400	27	30	100	230	55	1000	120



TABLE IV  
Elemental concentrations in sediments. The values for Ca, Ti, Mn and Fe are in  $\text{g}\cdot\text{kg}^{-1}$ , all other values in ppm.

SITE	Ca	Ti	Mn	Fe	V	Cr	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Pb
I	133	3.8	1.0	52	50	290	190	53	94	13	91	131	17	117	70
II	134	3.5	0.9	46	200	240	185	32	98	15	92	129	15	107	70
III	135	2.9	1.4	46	200	360	190	21	85	18	85	120	18	80	75
IV	133	3.2	1.3	50	120	400	212	42	86	12	90	124	22	90	75
V	120	2.6	0.9	40	140	220	130	50	107	20	123	129	23	103	70
VI	220	1.0	0.5	17		100	10	20	30						
IX	87	2.4	0.7	37		620	400	50	150						

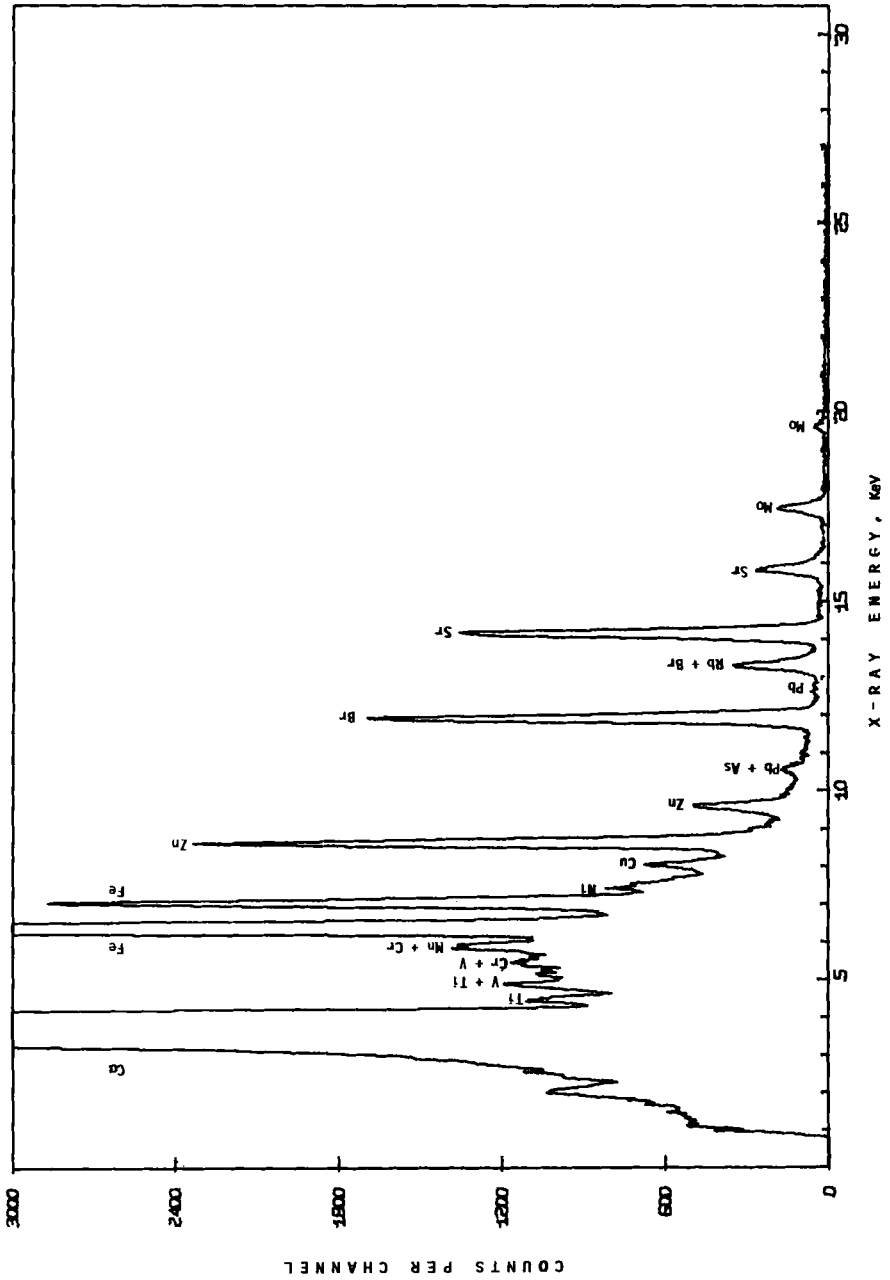


FIGURE 2 The X-ray spectrum of a water residue sample.

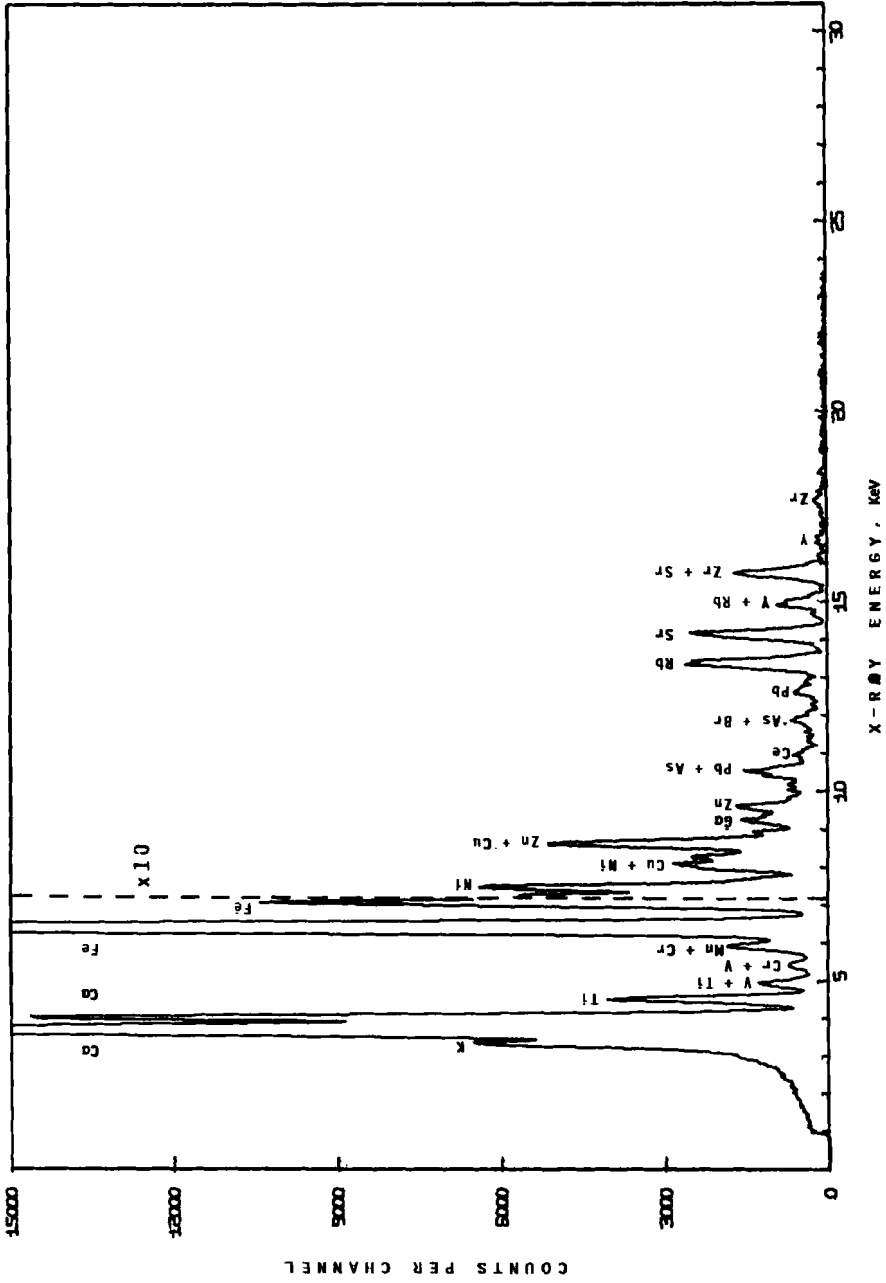


FIGURE 3 The X-ray spectrum of a sediment sample.

as well as the normal background values of Förstner and Wittmann, for reasons of comparison. Finally, Table IV shows the results of the corresponding sediment samples.

A typical X-ray spectrum of a water residue sample is shown in Figure 2, while Figure 3 shows a typical X-ray spectrum of a sediment sample.

### 3.2 Discussion

In the following discussion it must be remembered that the flow rate at the Solou mouth (SM) is ten times higher than the one at the Canal. On the other hand, the Farangi stream (FS) flows only during the spring months.

*Farangi stream* The most natural water in the area under study is probably the water of Farangi stream. The differences in the elemental concentrations which appear relative to the background values given by Förstner *et al.* (Table III) are therefore accepted to be due to lithogenic reasons. Similar differences are also observed for the drinking water of Athens which comes from a lake in the center of Greece.

*Petron Lake* Next, one may examine the trace elements in Petron Lake (Table III) which accepts only municipal effluents, but no industrial pollutants. In this particular lake low Ca and Cr concentrations are observed. On the other hand, Ti, Mn, Sr, Zn, Br and Pb are found in relatively high concentrations. Their presence may be due to lithogenic reasons or to municipal pollution. In fact the concentration of these latter elements in the sediment of Petron Lake (Table IV) is much less than their concentration in Vegoritis Lake, while the concentration of Ca is high. Therefore the differences are most probably due to anthropogenic reasons.

*Petron canal* The water here appears very poor in all trace elements with the exception of Br which has the highest concentration among all the samples under study. This observation cannot be easily explained.

*Solou stream* Comparing the water at the origin (SO) of Solou stream (before passing through the industrial and municipal areas) to the Farangi stream water, the main differences observed are low concentrations of Ti and Fe and high concentrations of Ni and As. This could probably be explained from the fact that the stream starts from an area which is a drained lake.

The stream passes through several industrial and municipal areas before reaching the Solou mouth (SM) which discharges to the lake. At this point it is noticed that the concentrations of Ca, Sr and Cr and to a lesser degree Mn, Ni and As have decreased, while the concentration of Zn has increased. The concentrations of V and Br remain constant and several other elements such as Ti, Fe and Pb do not show any significant changes.

The fact that there are elements, e.g. Cr, with lower concentrations at SM than at SO could imply that water enters the Solou stream as it proceeds to the lake, thus diluting the metal concentrations. This water could be coming from side springs or due to the water pumped from Vegoritis Lake for use in the factories (like the Ptolemais electric plant). If this assumption is true, then the magnitude of metal pollution is much higher than indicated from the tables. If Cr were taken as a base, the dilution factor would be 5.5.

Another reason for the decrease of elemental concentrations from SO to SM could be the discharge into the stream of chemicals which cause the precipitation of several elements. Indeed examining the composition of the sediment at SM (Table IV, Station IX) one observes high concentrations of Ni and Cr as well as Zn, but low concentration of Ca. Therefore, it would appear that the precipitation of Ni and Cr is fast, which for Ca and Zn it is a slow process and it occurs mainly in the lake. In any way, the numbers indicate a high degree of Zn enrichment of the Solou stream. Zinc partly precipitates in the stream and partly enters the lake.

*Vegoritis Lake* The results for the water analysis of the lake are in Table II. They are compared with the other sources in Table III, while the results for the sediments of the lake are in Table IV.

There are five collecting sites in the lake (Figure 1). The reader is reminded that Site I is near the entrance of the Solou stream and near the point where water is pumped out for the Ptolemais electric power plant, while the other stations proceed to the north-east where an underground canal comes from an area of drained marshes.

The solid residue of the water is highest at Site II and to a lesser degree at Site I, indicating the introduction of solids mainly from the canal (taking into account its low flow rate) and secondly from the Solou stream. This assumption is in accordance with the solid residues of these waters (Table III). It must be also noted that the amount of the solid residue of the water is reduced towards the north of the lake.

The lake water is rich in Br which is most likely coming from the

canal, but has low concentrations of Ca, Mn, Ni, Sr, Cr and As. In the average one observes higher concentrations of the elements in the bottom water of the lake than in the surface water. This may be due to the higher pH values of the surface water (Mourkides and Tsiouris, unpublished data).

Another important observation is that the Ca concentration increases from south to north in the water samples, while it decreases in the sediments, in the same direction. This could be again due to various chemicals introduced into the lake.

Since sediment may be regarded to reflect the condition in an aquatic system, sediment analysis gives a new dimension by being employed as a tool to trace man-made pollution influences in inland waters and the coastal marine environment.

Most of the metals in the water precipitate into the sediment if the pH is increased (Dean *et al.*, 1972; Pande and Das, 1979). Therefore, due to the high pH values of the Vegoritis waters (Mourkides *et al.*, 1978) the concentrations of metallic contents in sediments are expected to be much higher than in our lake waters.

Comparing the values of the lake sediments of remote areas (Förstner and Wittmann, 1979, p. 136) with our values we notice our concentrations to be in the range of their report, except for chromium. Their chromium values range between 20–190 ppm while the sediments of the Lake Vegoritis have 220–400 ppm chromium and 620 ppm at the Solou stream. This means that either the area is rich in ores containing chromium or chemicals are introduced in the Solou stream and cause chromium's precipitation. The corresponding value of the Lake Petron is only 100 ppm.

The fact that the values in the elements of Vegoritis Lake sediment are in the range of the reported values (Förstner and Wittmann, 1979, p. 136), implies that the pollution of the lake is in its first steps and should be controlled before it is too late.

#### 4 CONCLUSIONS

The anthropogenic activities in the drainage basin do not have any significant impact on the Vegoritis Lake yet, as far as the elements studied are concerned. However, this will not be so, if the industry continues to operate without treatment of their effluents. On the other hand, there is indication that pollutants are introduced mainly along the Solou stream, which cause precipitation of the metals. The calcium

content of the water and of the sediment protects the lake water to some degree and renders the sediment a sink for the pollutants.

Bertine and Goldberg (1971) reported that lignite burning power plants are typical sources of metal pollution, i.e. zinc, arsenic, nickel, vanadium, chromium, cadmium and mercury. So one may conclude that lignite-fired electric generator plants of this area pollute the lake with zinc, arsenic, vanadium, nickel and perhaps with iron, titanium and strontium. Bromine, manganese and lead are industrial pollutants of this area. Samples were not analysed for mercury and cadmium. In addition, the lake gets polluted from municipal activities with nickel, zinc, iron, lead, strontium, bromine and manganese. Wittmann and Förstner (1976) reported zinc as a domestic pollutant, while Schleichert and Helmann (1973) found zinc in domestic and industrial effluents. Lead is considered to be a municipal pollutant (Förstner and Wittmann, 1979, p. 161).

Lithogenic sources pollute the Lake Vegoritis with titanium, copper, zinc, manganese, iron, strontium, chromium, lead and arsenic.

The levels of the studied elements in this investigation are not in any excess of the permissible values established for fish farming and drinking purpose (USNRC, 1977).

Previous data for comparison are lacking with regard to metals in water and sediment from this area. This work is preliminary and more extensive studies should be conducted here before any firm conclusions can be made. Such investigations are under way and cover a greater portion of the area. At the same time we plan to study the variation of the pollution with time by taking core sediments and slicing them into thin layers. The more interesting parts will be the layers of the upper 1–2 cm, corresponding to the last 10–20 years. This procedure will be possible with a new sampler we have purchased, while our results for the sediments in this paper represent average values for depths 0 to 20 cm. The study will include the toxic elements mercury and cadmium as well. Finally, waste water of the industrial and municipal sources are also under study.

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