

# Concentrations of Poorly Studied Technology-Critical Elements Ge, Zr, Mo, Sn, Sb, Hf, Ta, W and Ti in the Sediments of Three Northern Egyptian Lakes

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## Abstract

Thirty five Sediment samples were collected from three Northern Egyptian Lakes namely El Burullus, El Manzala and El Bardawil to evaluate for the first time the concentrations of poorly studied technology-critical elements, namely Ge, Zr, Mo, Sn, Sb, Hf, Ta, W and Ti, and how that may be linked to local water quality parameters and other geochemical factors. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was used to determine element's concentration. The results were compared with Upper Continental Crust (UCC) and Nile sediment (ANS). Lake sediments generally have relatively low contents of most elements. The average concentration of Zr, Hf, Ta and W are lower than UCC and ANS in three Lakes. El Bardawil Lake attained the lowest average concentration for all elements except Mo. Correlation analysis revealed that water quality and geochemical properties did not play any important role affecting the metal distribution and the studied metals are generally not affected by the complex lacustrine system of these lakes and their spatial distribution still not affected by the different anthropogenic activities.

## Keywords

Poorly-Studied Technology-Critical Metals, Sediments, Northern Egyptian Lakes, ICP-MS

## 1. Introduction

The Egyptian Mediterranean coast, which is considered to be between arid and

hyper-arid, has five coastal lakes, namely Mariut, Idku, El-Burullus, El-Manzala and El-Bardawil. These lakes constitute about 25% of the total Mediterranean wet lands. All of them are directly connected to the Sea except Lake Mariut. More than 20% of Egypt's total population lives along the northern coastal zone of the country, with more than 40% of its economic activities including industry, agriculture, tourism, petroleum and mining activities, and urban development concentrated along the coast [1] [2]. Unfortunately, many factors exist in Egypt which directly or indirectly threaten the Egyptian coastal lakes ecological system. The degradation of these coastline habitats due to the rapid development and urbanization lead to imbalance of the ecosystems and generate polluting elements that affect the quality of these frail and precious areas mainly due to the discharge of large quantities of agricultural, industrial, and municipal wastes through several drains and from factories around them [3]-[7]. Although the Egyptian Northern Lakes are productive ecosystems that provide a good quantity of fish, they are usually under pressure from industrial activities and potentially polluting activities developed around them. Therefore close monitoring are required to develop feasible approaches to mitigate fish contamination and the associated human health risks. The Egyptian government recently launched a program and established a strong policy framework to mitigate these lakes' pollution and prioritized minimizing the release of harmful contaminants within these lakes [6] [8] [9].

Among all the pollutants, heavy metals have received a paramount attention to environmental chemists [10] [11] [12] [13]. Because of their persistence and their tendency to be bio accumulated by the aquatic organisms, they are considered very harmful for the aquatic environment. The deleterious effects of some of the trace elements on living organisms were well documented in the Egyptian ecosystems [14]-[24]. Metal's concentrations in the aquatic environment were regulated by Egyptian Environmental Affairs Agency (EEAA) protocols and directives that determine their appropriate environmental concentrations according to Law No. 4 of 1994 Promoting the Law on the Environment and its Executive Regulation, Egypt (EEAA, 2005) [25].

Metal pollutants occur mainly from two sources in the aquatic environment; 1) natural sources such as volcanic eruptions, and soil erosion. 2) Some anthropogenic behavior. These two factors are the primary cause of disruption to the natural ecosystem and eventually to humans via the food chain [6] [26]-[32]. However, a significant number of trace elements in these studies are excluded. This is due to: 1) their low ambient concentrations, in general, the detection limits of the analytical procedures used and 2) the lack of any significant industrial role in the past has no apparent environmental consequences [33]. This situation is presently changing, since some of these non-regulated trace elements such as Tellurium (Te), Germanium (Ge), gallium (Ga), Indium (In), Niobium (Nb), Tantalum (Ta) etc. are now key of the development of new technologies, such as semiconductors, electronic displays, energy-related technologies or telecommunications technology. The extent of the environmental impact of the increasing

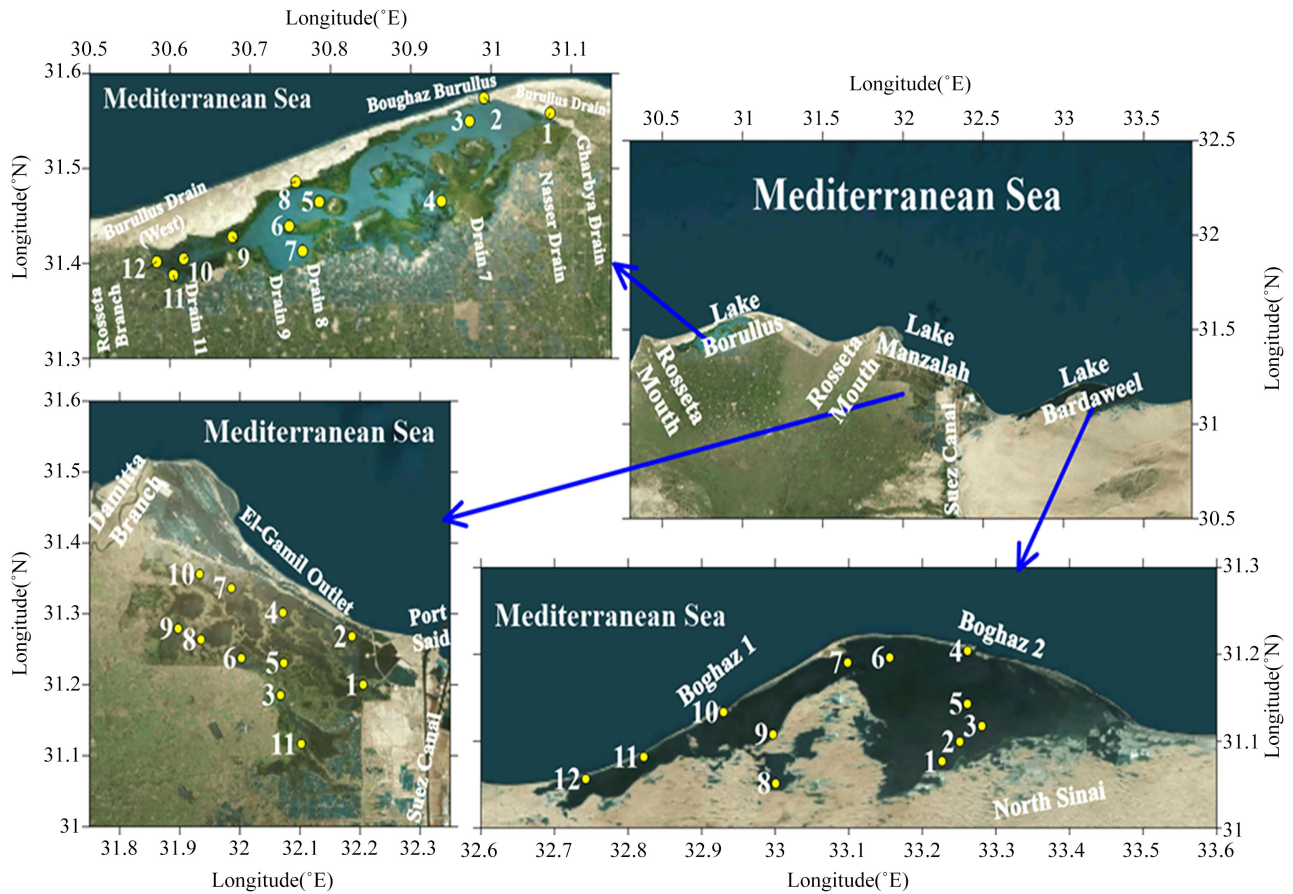
anthropogenic use of these elements needs therefore to be further assessed including knowledge of their concentrations in the environment along with better understanding of the chemical processes underlying its environmental behavior. A major reason for this scarcity in environmental studies relies in the fact that their analytical determination is still challenging [34] [35].

Sediments are the primary habitat and source of food for benthic organisms. Sediment pollution is directly or indirectly detrimental to aquatic organisms, and may even have further adverse impacts on terrestrial organisms and human beings as a result of bioaccumulation. The main objectives of this study are to analyze the concentrations, better understand the occurrence and the distribution patterns of poorly studied metals, namely Germanium (Ge), Zirconium (Zr), Molybdenum (Mo), Tin (Sn), Antimony (Sb), Hafnium (Hf), Tantalum (Ta), Tungsten (W) and Titanium (Ti), in the sediments of three Northern Egyptian Lakes. Based on this study, an evaluation of the current gaps in our knowledge of these elements in sediments of three Egyptian lakes will be provided to indicate directions for future research. This study was the first study to determine the concentrations of these elements in the sediments of the three lakes, and thus it provides base-line information that is the beginning of a database that can be used in assessing human-induced changes. Therefore, this study offers a valuable data for better monitoring of technology critical elements in three northern Egyptian lakes which will be helpful for environmental managers and policymakers.

## 2. Materials and Methods

### 2.1. Study Area, Site Selection and Sampling

The northern lakes play an important role in the Egypt's economy, not only because of producing more than 40% of the nation's fish catch but also for being resting areas for migrating birds [8] [9]. More than 75% of the harvest of Egyptian lake production is from them. Moreover, they have an ecological importance due to a variety of biodiversity inhabiting the lakes and their hydrologic attributes. Changes in environmental conditions along with other human-induced stresses and interferences have played an important role in the degradation of the lake and the consistency of the water and increase all the biological productivity along the lakes causing less diverse system. Sediment samples were collected from different locations (**Table 1** & **Figure 1**) by using a van-veen grab sampler. In addition to sediment samples, water samples were collected by completely submersing the sample bottle, opening the lid and filling the bottle, and then recapping the bottle underwater. In addition, the reproducibility of the sampling procedure was verified through collection of 3 replicate samples. Water quality parameters such as water temperature, salinity, pH, dissolved oxygen (DO), biological oxygen demand (BOD) and chemical oxygen demand (COD) were measured on each aqueous sample immediately after collection.



**Figure 1.** Map showing the positions of sampling stations of the three Egyptian Northern lakes.

**Table 1.** Locations of the sampling sites in El-Burullus, El-Manzala, and El-Bardawil Egyptian Northern Lakes during 2019.

Lake Stations	El-Burullus Lake		El-Manzala Lake		El-Bardawil Lake	
	Lat.	Long.	Lat.	Long.	Lat.	Long.
1	31°33'29.9"	31°04'25.3"	31°12'1.2"	32°12'18.0"	31°04'37"	33°13'36"
2	31°34'27.6"	30°59'28.4"	31°16'15.6"	32°11'11.4"	31°05'58"	33°15'03"
3	31°32'59.0"	30°58'23.5"	31°11'7.8"	32°04'3.0"	31°07'03"	33°16'51"
4	31°27'56.1"	30°56'17.5"	31°18'6.6"	32°04'17.4"	31°12'15"	33°15'41"
5	31°27'53.3"	30°47'10.0"	31°13'51.0"	32°04'22.8"	31°08'35"	33°15'40"
6	31°26'21.2"	30°44'54.9"	31°14'15.6"	32°00'9.0"	31°11'47"	33°09'20"
7	31°24'46.9"	30°45'54.9"	31°20'10.8"	31°59'9.0"	31°11'26"	33°05'54"
8	31°29'9.0"	30°45'24.4"	31°15'50.4"	31°56'6.6"	31°03'50"	33°00'02"
9	31°25'41.0"	30°40'40.2"	31°16'45.6"	32°53'49.8"	31°06'28"	32°56'49"
10	31°24'17.5"	30°37'1.9"	31°21'22.2"	31°55'58.8"	31°08'01"	32°55'47"
11	31°23'15.5"	30°36'15.3"	31°07'0.6"	32°06'7.8"	31°04'55"	32°49'17"
12	31°24'6.3"	30°35'0.4"			31°03'24"	32°44'33"

## 2.2. Materials

### 2.2.1. Reagents and Apparatus

All chemicals and mineral acids were of superpure quality (Merck, Darmstadt, Germany). All glassware and plastics were cleaned by soaking overnight in a 10% (w/v) HNO<sub>3</sub> solution and then rinsed with deionized water before use. All solutions were prepared by using ultrapure water (18.2 MΩ cm). The entire procedure was accomplished in a Class-100 laboratory. Reagents and procedural blanks were also determined in parallel to the sample treatment.

Inductively coupled plasma-mass spectrometry ICP-MS (Varian 810/820-MS ICP Mass Spectrometers-USA) was applied. The ICP-MS operating parameters were set as follows: Plasma gas, 16.00 L·min<sup>-1</sup>; Auxiliary gas, 2.00 L·min<sup>-1</sup>; Nebulizer gas, 1.00 L·min<sup>-1</sup>.

### 2.2.2. Field Procedures and Analysis

Measurements of temperature (T), salinity (S), and pH were performed in situ during sampling. A salinometer (Thermo Fisher Eutech Salt, USA) was used to measure the salinity of water, and a pH meter (Thermo Fisher Orion Star A221, USA) was used for the pH measurement. The contents of dissolved oxygen/hydrogen sulfide (DO/H<sub>2</sub>S); biological oxygen demand (BOD) and chemical oxygen demand (COD) were determined according to APHA [36]. In the sediment samples, granulometric analysis was based on determination of sandy and muddy fraction by separating the coarser (both sand and gravel) fractions above 4Φ (0.063 mm), from finer (mud) fraction below (0.063 mm) involving the sieving of raw sediments on a standard sieve (4Φ) mounted on an electric shaker machine (Labor-technique) and 10 minutes was applied as a standard time of sieving according to Folk [37]. The dry sediments were used for determination of the total organic carbon (TOC) according to Schumacher [38], and water contents were determined by drying at 105°C for constant weight. Total phosphorus and inorganic phosphorus as well as total nitrogen were determined according to Aspila *et al.* [39].

Approximately 100 mg of powdered sediments digested to a mixture of 10 mL of HCl (37%,  $\rho = 1.19$  g/mL), 10 mL of HNO<sub>3</sub> (65%,  $\rho = 1.42$  g/mL), 10 mL of HF (40%,  $\rho = 1.49$  g/mL), and 10 mL of HClO<sub>4</sub> (70%,  $\rho = 1.68$  g/mL) at 180°C in a microwave oven (ETHOS TOUCH CONTROL, Milestone Inc., Via Fatebenefratelli, 1/5 24010 Sorisole (BG), Italy). The obtained suspension liquid was then filtered using a membrane filter and nine elements namely, Germanium, Zirconium, Molybdenum, Tin, Antimony, Hafnium, Tantalum, Tungsten and Titanium were analyzed using ICP-MS according to Caballero-Gallardo *et al.* [40].

## 2.3. Method Validation and Quality Control Studies

In order to guarantee the accuracy of the results, adequate quality assurance/quality control (QA/QC) was adopted in all aspects of the study. Quality control (QC) and quality assurance (QA) for quantitative ICP-MS included triplicate

analyses, dilution checks, reference solutions, analysis spikes, an interference check, and calibration checks were applied. Triplicate field sediment samples were taken at each site and an average of the resulting element concentrations was used to represent that site. Precision was determined by three replicate analyses of one sample and expressed as a coefficient of variation (CV). The results of the precision were agreed within 10%. The accuracy of the method was tested by five replicates of a standard reference material (GBWO7301) stream sediment, China. In general, quantitative recoveries for Ge, Zr, Mo, Sn, Sb, Hf, Ta, W and Ti using ICP-MS 810/820-USA fall within  $\pm 20\%$ .

### 3. Results and Discussion

#### 3.1. Water Quality of the Three Egyptian Northern Lakes

The water quality of the three Egyptian Northern Lakes has become a focus of various studies due to the influence of the severe anthropogenic activities resulting from the excessive discharges of different agricultural, industrial, and sewage effluents. Other factors may also exert local control on the physicochemical properties are land use changes and urban development, leading to ecological stress and algal blooms [3] [6] [7] [26].

A summary of selected chemical properties is presented in **Table 2**. Our investigations on the water quality parameters, namely water temperature, salinity, conductivity, pH, DO, H<sub>2</sub>S, BOD and COD, revealed a noticeable variations in hydrochemical characteristics of different water samples collected from the three northern Egyptian lakes. The temperature of the three lake waters showed obvious differences. The mean water temperatures in the three lakes, *i.e.*, El-Burullus, El-Manzala and El-Bardawil were 25.05°C (range: 22.50°C - 26.70°C), 24.80°C (range: 23.30°C - 25.50°C) and 22.06°C (range: 20.70°C - 22.70°C); respectively. The slight difference in the water temperature between the lakes is mainly due to the difference in the sampling time and very closely related to those of the air temperature. The lowest electrical conductivity value (3.30 mm-cm<sup>-1</sup>) and salinity (1.87 g-l<sup>-1</sup>) were observed at El-Manzala Lake, while the highest values were measured at El-Bardawil lake (78.8 mm-cm<sup>-1</sup> and 61.15 g-l<sup>-1</sup>; respectively). The salinity ranged from 2.23 - 19.72, 1.87 - 11.19, 40.68 - 61.15 g-l<sup>-1</sup> in the three lakes; respectively, revealing an increasing tendencies in the order: El-Bardawil Lake > El-Burullus Lake > El-Manzala Lake. The salinity level varies regionally within a wide range, from Mediterranean shelf water in El-Bardawil Lake waters to brackish water in El-Burullus Lake and El-Manzala Lake which is mainly attributed to the huge amounts of agricultural and sewage wastewaters effluents.

The pH values fluctuated between 7.56 at El-Manzala Lake to 9.44 at El-Burullus Lake. On the meantime, the relatively low pH value in El-Manzala Lake waters was most probably due to the oxidation (decomposition) of organic matter, exhausted DO and liberation of H<sub>2</sub>S. Unlike salinity, the spatial variations in pH were not obvious.

**Table 2.** Water quality parameters of El-Burullus, El-Manzala, and El-Bardawil Egyptian Northern Lakes during 2019.

Water quality Parameters	El-Burullus Lake		El-Manzala Lake		El-Bardawil Lake	
	range	average $\pm$ SD	range	average $\pm$ SD	range	average $\pm$ SD
Water temp. ( $^{\circ}$ C)	22.50 - 26.70	25.05 $\pm$ 7.88	23.30 - 25.50	24.80 $\pm$ 5.79	20.70 - 22.70	22.06 $\pm$ 2.16
pH (SU)	8.56 - 9.44	-	7.56 - 8.96	-	7.99 - 8.18	-
Salinity (g.l <sup>-1</sup> )	2.23 - 19.72	6.39 $\pm$ 10.48	1.87 - 11.19	3.61 $\pm$ 2.43	40.68 - 61.15	48.66 $\pm$ 8.10
SpCond (mS/cm)	3.89 - 29.52	10.65 $\pm$ 10.48	3.30 - 17.50	7.82 $\pm$ 2.43	55.30 - 78.80	64.69 $\pm$ 8.10
DO (mg.l <sup>-1</sup> )	3.90 - 15.28	8.88 $\pm$ 3.10	4.1 - 11.38	7.16 $\pm$ 3.20	6.75 - 8.37	7.60 $\pm$ 1.27
H <sub>2</sub> S (ml.l <sup>-1</sup> )	ND	ND	ND	ND	ND	ND
BOD (mg.l <sup>-1</sup> )	9.27 - 46.32	16.19 $\pm$ 9.62	59.02 - 126.83	79.70 $\pm$ 4.17	1.46 - 6.58	1.44 $\pm$ 2.47
COD (mg.l <sup>-1</sup> )	34.00 - 69.36	53.62 $\pm$ 27.88	270.00 - 705.00	490.91 $\pm$ 25.1	9.41 - 20.06	13.77 $\pm$ 3.13

ND: Not detected.

The concentration of DO, on the other hand, ranged from 3.90 to 15.28 mg.l<sup>-1</sup> at El-Burullus Lake. It was noticeable that the waters of the three northern lakes were well aerated *i.e.* they were in oxic condition (Table 2). It is worth to mention that the observed DO mean concentration (>7 mg.l<sup>-1</sup>) in the three northern lakes was more than that optimum DO concentration level (exceed 3 mg.l<sup>-1</sup>) for good growth and the suitability of water for aquaculture purposes [41]. The range of DO concentrations was comparable to that in other Egyptian lakes. The BOD values ranged between 1.46 mg O<sub>2</sub>/l at El-Bardawil Lake and 126.83 mg O<sub>2</sub>/l at El-Manzala Lake in which the order of abundance of BOD values was: El-Manzala Lake > El-Burullus Lake > El-Bardawil Lake. The COD levels varied between 9.41 mg O<sub>2</sub>/l at El-Bardawil Lake and 705 mg O<sub>2</sub>/l at El-Manzala Lake. The measured high COD concentrations at El-Manzala Lake were mainly due to the autochthonous source from endogenous phytoplankton and algae [42]. The increased industrialization, urbanization and man's greed to over exploit nature has led high differences in the northern lakes water chemistry which is reflected in differences in hydrology, amount of water discharges into these lakes, rate of water exchange between these lakes and the Mediterranean Sea etc. [6] [43]. These factors were mainly responsible for the lower water salinity and higher concentrations of BOD, and COD in El-Manzala lake and El-Burullus Lake compared to El-Bardawil Lake [44] [45] [46] [47].

### 3.2. Geochemical Characteristics of the Three Egyptian Northern Lakes

The grain size is one of the main factors that govern heavy metal contamination in the particulate fraction. In general, small particles are more likely to bear heavy metals because of the rise in particular surface area and because of the presence of clay minerals, organic matter, and Fe/Mn/Al oxides associated forming fine-sized aggregates [48].

The results of the total mean levels of different geochemical parameters in surface sediments of northern lakes (Table 3) revealed that the sandy size showed the highest value in El-Bardawil Lake 88.25%, while the lowest value was observed in El-Burullus Lake (11.72%). The sandy size in the lake sediments can be arranged as follows: El-Bardawil Lake (67.47%) > El-Manzala Lake (39.15%) > El-Burullus Lake (29.80%) depending on the availability of different sizes in source sediments, and the transport operations affected by sediments. The mean of silt (%) in sediments of the northern lakes cleared out that the highest value was observed at El-Burullus Lake (85.95%); while the lowest ones (14.22%) was measured in the sediments of El-Bardawil Lake (Table 3). The mean of silt (%) of the lake sediments can be arranged as follows: El-Burullus Lake (69.14%) > El-Manzala Lake (59.47%) > El-Bardawil Lake (31.09%).

The organic matter is made of low density materials. It is widely regarded as a vital component of a healthy soil. It is a major part of physical, chemical and biological fertility of the soil. The deposition of the organic matter is closely related to the precipitation of the fine volumes as they follow the same behavior. Both of them are deposited in the calm sediment media, and the organic matter affects the ecosystem significantly [49]. By studying the organic carbon, it was found that Al-Manzala Lake contains the highest value (4.90), while El-Bardawil Lake attained the lowest value (0.40%). The organic carbon in the lake sediments can be arranged as follows: El-Manzala Lake (3.92%) > Al-Burullus Lake (2.16%) > Al-Bardawil Lake (1.34%).

Comparing the mean sulfide concentrations in sediments of the three northern Lakes revealed the highest value of 419 mg.g<sup>-1</sup> at El-Burullus Lake, while the lowest value of 0.89 mg.g<sup>-1</sup> was measured in sediments of El-Bardawil Lake. The descending order was as follows: El Burullus Lake (168.7 mg.g<sup>-1</sup>) > El Bardawil Lake (86.3 mg.g<sup>-1</sup>) > El Manzala Lake (58.7 mg.g<sup>-1</sup>) for Lakes sediments (Table 3).

**Table 3.** Geochemical characteristics of El-Burullus, El-Manzala, and El-Bardawil Egyptian Northern Lakes during 2019.

Geochemical Parameters	El-Burullus Lake		El-Manzala Lake		El-Bardawil Lake	
	range	average ± SD	range	average ± SD	range	average ± SD
Sand (%)	11.72 - 49.32	29.80 ± 18.68	14.88 - 63.83	39.15 ± 24.28	34.16 - 88.25	67.47 ± 35.76
Silt (%)	50.96 - 85.95	69.14 ± 18.68	36.18 - 83.19	59.47 ± 24.28	14.22 - 61.42	31.09 ± 35.76
Organic carbon (%)	1.34 - 3.31	2.16 ± 0.87	2.71 - 4.90	3.92 ± 1.42	0.40 - 2.53	1.34 ± 1.30
Water content (%)	38 - 65	54 ± 11.93	56 - 74	64 ± 11.58	22 - 61	37 ± 18.18
H <sub>2</sub> S (mg.g <sup>-1</sup> )	25 - 419	168.7 ± 134.85	6 - 249	58.7 ± 75.17	0.89 - 238	86.3 ± 110.25
Inorganic Phosphorus (µg.g <sup>-1</sup> )	574 - 1098	780 ± 232.61	245 - 893	361 ± 151.93	122 - 326	203 ± 130.90
Organic Phosphorus (µg.g <sup>-1</sup> )	160 - 335	165 ± 159.10	168 - 286	210 ± 62.14	47 - 174	88 ± 63.47
Total Phosphorus (µg.g <sup>-1</sup> )	761 - 1244	972 ± 192.71	511 - 1232	833 ± 156.88	193 - 413	274 ± 166.45
Total Nitrogen (%)	0.88 - 1.92	1.23 ± 0.08	1.84 - 2.39	2.08 ± 0.09	0.84 - 1.06	0.95 ± 0.05

The water contents of sediments is one of the most important factors affecting chemical, physical and biological processes that affect sediments in the ecosystem [50]. The water contents results of the surface sediments in the three northern lakes revealed the lowest value of 22% in the sediments of El-Bardawil Lake, while the highest value was measured at El-Manzala Lake (74%). The water contents in the lake sediments can be arranged as follows: El-Manzala Lake (64%) > El-Burullus Lake (54%) > El-Bardawil Lake (37%).

By studying different phosphorus forms, it was also found that El Burullus Lake had the highest values of total and inorganic phosphorus (972 and 780  $\mu\text{g}\cdot\text{g}^{-1}$ ), while the organic phosphorus exhibited the highest value of 210  $\mu\text{g}\cdot\text{g}^{-1}$  in El-Manzala Lake. On contrast, El-Bardawil Lake showed the lowest values (274, 203 and 88  $\mu\text{g}\cdot\text{g}^{-1}$ ). Lakes sediments can be arranged according to the ratio of inorganic phosphorus and total phosphorus as follows: El Burullus Lake > El Manzala Lake > El Bardawil Lake. While the descending order according to the contents of organic phosphorus and total nitrogen was as follows: El Manzala Lake > El Burullus Lake > El Bardawil Lake for the Lakes sediments. El-Manzala Lake showed the highest percentage of organic phosphorus (210  $\mu\text{g}\cdot\text{g}^{-1}$ ) and total nitrogen (2.08%), while El-Bardawil Lake attained the lowest values (88  $\mu\text{g}\cdot\text{g}^{-1}$  and 0.95%, respectively) which is mostly related to the high contents of organic carbon (Table 3).

### 3.3. Distributions of Different Metals of the Three Egyptian Northern Lakes

Concentrations of nine rare earth elements in sediments from three lakes along the Mediterranean Sea are given in Table 4 with the corresponding values for Upper Continental Crust (UCC) [51], North America Shale Composite (NASC) [52], Average world suspended sediment (AWSS) [53], as well as average Nile sediment (ANS) as previously reported by Arafa *et al.* [54]. The present study indicated that lakes sediments generally have relatively low contents of most elements.

**Table 4.** Concentrations ( $\text{mg}\cdot\text{kg}^{-1}$ ) of investigated elements in sediments of El-Burullus, El-Manzala, and El-Bardawil Egyptian Northern Lakes during 2019.

Element	El-Burullus Lake		El-Manzala Lake		El-Bardawil Lake		UCC	NASC	AWSS	ANS
	range	average $\pm$ SD	range	average $\pm$ SD	range	average $\pm$ SD				
Germanium	0.76 - 3.10	2.05 $\pm$ 0.27	1.14 - 23.84	3.75 $\pm$ 6.69	0.17 - 1.98	0.77 $\pm$ 0.52	1.4	ND	ND	ND
Zirconium	25.52 - 144.95	92.59 $\pm$ 36.54	4.11 - 164.02	61.12 $\pm$ 45.24	ND - 0.55	0.22 $\pm$ 0.16	162	200	160	331
Molybdenum	ND - 1.83	0.65 $\pm$ 0.60	0.24 - 6.54	1.89 $\pm$ 1.69	0.04 - 2.71	1.04 $\pm$ 0.88	1.6	ND	2.98	ND
Tin	ND - 12.23	2.63 $\pm$ 3.58	ND - 5.9	1.07 $\pm$ 2.26	0.26 - 0.70	0.43 $\pm$ 0.16	2.1	ND	ND	ND
Antimony	2.36 - 15.76	8.28 $\pm$ 3.62	ND - 12.77	4.08 $\pm$ 3.78	0.04 - 0.92	0.35 $\pm$ 0.26	0.2	7.3	2.19	0.18
Hafnium	0.47 - 3.91	2.28 $\pm$ 1.0	0.10 - 4.59	1.69 $\pm$ 1.19	0.01 - 1.25	0.53 $\pm$ 0.37	4	6.3	1.27	6.86
Tantalium	0.01 - 0.61	0.32 $\pm$ 0.19	0.02 - 1.04	0.36 $\pm$ 0.33	ND - 0.47	0.17 $\pm$ 0.17	1.5	0.41	12.1	0.81
Tungsten	0.03 - 0.64	0.40 $\pm$ 0.18	0.16 - 0.76	0.35 $\pm$ 0.18	ND - 0.76	0.14 $\pm$ 0.20	1.9	ND	ND	ND
Titanium	2380 - 9090	5720 $\pm$ 2190	ND - 8430	3770 $\pm$ 2280	ND - 8.08	2.18 $\pm$ 3.31	3300	4196	4400	7370

ND: Not detected.

Germanium is an important element used in electronics devices, flat-panel display screens, light-emitting diodes, night vision devices, optical fiber, optical lens systems, and solar power arrays. Concentrations of Ge, in sediments of the three lakes were ranged between 0.76 - 3.10; 1.14 - 23.84 mg·kg<sup>-1</sup>; and 0.17 - 1.98 mg·kg<sup>-1</sup>; for El-Burullus lake, El-Manzala Lake, and El-Bardawil Lake. The descending order was as follows: El Manzala Lake (3.75 mg·kg<sup>-1</sup>) > El Burullus Lake (2.05 mg·kg<sup>-1</sup>) > El Bardawil Lake (0.77 mg·kg<sup>-1</sup>). These reported concentrations in El Manzala Lake and El-Burullus Lake are higher than abundance of Ge in UCC. Ge is sorbed by clay minerals, co-precipitates with Fe-oxides and has a strong tendency to bind to organic matter [55] [56] [57].

Zirconium and Hafnium are likely not essential for human health and generally are considered elements of low toxicity to humans [58]. The results of the present study (**Table 4**) indicated that concentrations of Zr and Hf in sediments of El-Burullus Lake ranged between 25.52 and 144.95 mg·kg<sup>-1</sup> and between 0.47 and 3.91 mg·kg<sup>-1</sup>; respectively. El-Manzala Lake exhibited concentrations ranging from 4.11 to 164.02 mg·kg<sup>-1</sup> for Zr and from 0.10 to 4.59 mg·kg<sup>-1</sup> for Hf. In case of El-Bardawil Lake concentrations varied between ND and 0.55 mg·kg<sup>-1</sup> for Zr and between 0.01 and 1.25 mg·kg<sup>-1</sup> for Hf. It is clear that the average concentrations of Zr and Hf are less than values of NASC, AWSS, UCC and ANS except lakes El-Manzala and El-Burullus which are slightly higher than AWSS for Hf. Because of the low solubility of zirconium and hafnium and their resistant to weathering, ecological health concerns in the aquatic environment are minimal. The lethal concentration for 50 percent (LC) in controlled laboratory tests was estimated to be greater than 1000 µg·kg<sup>-1</sup> for both zirconium and hafnium [59]. Average concentration of Zr was close to that reported in Nylandssjon Lake located in the Nordingrå peninsula (northern Sweden) (85 mg·kg<sup>-1</sup>) [60], but it was lower than those recorded in sediments from the Egyptian central Nile Valley (316 mg·kg<sup>-1</sup>) [61]. The descending order for Zr in the northern Egyptian lakes was as follows: El Burullus Lake (92.59 mg·kg<sup>-1</sup>) > El Manzala Lake (61.12 mg·kg<sup>-1</sup>) > El Bardawil Lake (0.22 mg·kg<sup>-1</sup>), while it was: El Burullus Lake (2.28 mg·kg<sup>-1</sup>) > El Manzala Lake (1.69 mg·kg<sup>-1</sup>) > El Bardawil Lake (0.53 mg·kg<sup>-1</sup>) in the case of Hf for the Lakes sediments.

The mean Molybdenum concentration in the sediments of the three Egyptian lakes was in the order: El-Manzala Lake (0.24 - 6.54; av.1.89 mg·kg<sup>-1</sup>) > El-Bardawil Lake (0.04 - 2.71, av.1.04 mg·kg<sup>-1</sup>) > El-Burullus Lake (ND-1.83; av. 0.65 mg·kg<sup>-1</sup>). The stream sediments of Poland contain 0.17 - 1.94 mg·kg<sup>-1</sup> of molybdenum and the median is 0.42 mg·kg<sup>-1</sup>; while in lake sediments, the median is 1.1 mg·kg<sup>-1</sup> [62]. The molybdenum content in stream and river sediments of central Upper Silesia (Southern Poland) varied in the range of <0.5 - 204.8 mg·kg<sup>-1</sup>. The average content and geometric mean were 1.9 mg·kg<sup>-1</sup> and 0.7 mg·kg<sup>-1</sup>, respectively [63]. The concentration of molybdenum found in the northern lakes of Egypt is much lower than that found in Qaroun Lake (6.77 mg·kg<sup>-1</sup>) and its drains (6.27 mg·kg<sup>-1</sup>) sediments [64], but it is within the range that recorded in sediments

from the Egyptian central Nile Valley (0.2 - 6.8 mg·kg<sup>-1</sup>) [61]. **Table 4** shows that average concentration of Mo is lower than UCC.

The mean value of Tin in the northern Egyptian lakes was 2.63 mg·kg<sup>-1</sup> (ND -12.23) for El-Burullus Lake; while it was 1.07 mg·kg<sup>-1</sup> (ND-5.9) for El-Manzala Lake. There was a tendency to much lower Sn concentrations in El-Bardawil Lake (0.26 - 0.70; av. 0.43 mg·kg<sup>-1</sup>) (**Table 4**). The concentration of Sn found in the northern lakes of Egypt is lower than that found in Qaroun Lake (3.27 mg·kg<sup>-1</sup>) and drains (3.07 mg·kg<sup>-1</sup>) sediments [65]. Moreover, the median value of Sn was found to be 2.52 mg·kg<sup>-1</sup> (0.76 - 3.37) in surface sediments of Lake Qaroun, Egypt [64]. Lower concentrations were reported in sediments of the far south-east coast of New South Wales, Australia where Sn concentrations were found to be <0.01 - 2 mg·kg<sup>-1</sup> [66]. On contrast, much higher concentrations of Sn (9.2 - 106.59, mean 56.35 mg·kg<sup>-1</sup>) were measured in surface sediments from the Bernam River, Malaysia [67]. Generally, the average Sn concentration is closed to the UCC (2.10 mg·kg<sup>-1</sup>).

The present study revealed a clear geographical distribution of Antimony in lakes sediments showing high values in the sediments of El-Burullus Lake (2.3 - 615.76, mean 8.28 mg·kg<sup>-1</sup>) followed by El-Manzala Lake (ND-12.77, mean 4.08 mg·kg<sup>-1</sup>), and El-Bardawil Lake (0.04 - 0.92, mean 0.35 mg·kg<sup>-1</sup>). **Table 4** shows that average Sb concentration was higher than UCC and ANS. The geochemical behavior of Sb in aquatic sediments is directly affected by pH and redox conditions which, in turn, determine the presence of other compounds that can exert a strong control on the mobility of Sb [68]. Wu *et al.* reported lower concentrations of Sb (0.65 - 2.7 mg·kg<sup>-1</sup>) in sediments from Dianshan Lake, China [69]. Moreover, He *et al.* reported that the Sb concentration in sediments throughout the Yangtze River watershed ranged from 0.50 to 2.7 mg·kg<sup>-1</sup> dw [70]. Sediments in Dianshan Lake, China contained a geometric mean Sb concentration of 1.12 mg·kg<sup>-1</sup>, lower than the majority of lakes in Southern China, but higher than the Songhua River in Northern China which had Sb concentrations between 0.18 and 0.57 mg·kg<sup>-1</sup>.

Tantalum concentrations ranged between 0.01 and 0.61 with an average of 0.32 mg·kg<sup>-1</sup> for El-Burullus Lake, between 0.02 and 1.04 with an average of 0.36 mg·kg<sup>-1</sup> for El-Manzala Lake and between ND and 0.47 with an average of 0.17 mg·kg<sup>-1</sup> for El-Bardawil Lakes. Consequently, the descending order for Ta was as follows: El Manzala Lake (0.36 mg·kg<sup>-1</sup>) > El Burullus Lake (0.32 mg·kg<sup>-1</sup>) > El Bardawil Lake (0.17 mg·kg<sup>-1</sup>). The average Ta contents in stream sediment is 1.01 mg·kg<sup>-1</sup>, and the range varies from 0.05 to 58.4 mg·kg<sup>-1</sup> [71]. A notable feature of the Ta distribution in the three lakes sediments is its very low concentrations in El-Bardawil Lake compared to the other two northern Egyptian lakes. Generally, average Ta concentration is lower than UCC, NASC, WASS and ANS and that observed in sediments from the Egyptian central Nile Valley (0.9 mg·kg<sup>-1</sup>) [61]. Tantalum is absorbed only minimally by the human body; and nearly all the tantalum that enters the human body is eliminated within a relatively short

period of time.

Tungsten which is widely used resource in industrial and manufacturing applications due to its high density and tensile strength showed varied concentrations in sediments of the three Egyptian Lakes. It is largely used in high-tech and military industries and has been identified as an emerging contaminant by the Environmental Protection Agency (EPA) [72] [73] [74] [75]. It potentially represents a significant threat to both human and environmental health, which merits further research over the coming years in order to fill the present knowledge gaps [76]. The concentrations of W in lake sediments were in the ranges of (0.03 - 0.64 mg·kg<sup>-1</sup>), (0.16 - 0.76 mg·kg<sup>-1</sup>), and (ND-0.76 mg·kg<sup>-1</sup>) for El-Burullus lake, El-Manzala lake and El-Bardawil Lake; respectively, revealing an increase tendency in the order: El-Burullus Lake (0.40 mg·kg<sup>-1</sup>) > El-Manzala Lake (0.35 mg·kg<sup>-1</sup>) > El-Bardawil Lake (0.14 mg·kg<sup>-1</sup>). The W concentrations recorded in Lakes sediments are lower than UCC (1.9 mg·kg<sup>-1</sup>). Increasing use of tungsten (W)-based products opened new pathways for W into environmental systems and may be introduced into the food chain [77].

The average concentrations of Titanium in sediments were 5.72 g·kg<sup>-1</sup>, 3.77 g·kg<sup>-1</sup> and 2.18 mg·kg<sup>-1</sup> in El-Burullus, El-Manzala and El-Bardawil respectively (**Table 4**) revealing an increase tendency in the order: El-Burullus Lake > El-Manzala Lake > El-Bardawil Lake. This indicated that the geochemistry of underlying surficial deposits was the most important factor for Ti in lakes. The average concentrations of Ti were less than the value of the elemental abundance in upper continental crust (0.66%) [78]. Moreover, the Ti content is lower in sediment in comparison with the values reported for ANS, even with respect to the Nile sediment and soil data previously reported by Badawy *et al.* (9.672 g·kg<sup>-1</sup>) [79]. El-Bardawil Lake was recorded the lowest content of Ti which is lower than the values of UCC and NASC Concentrations in El-Bardawil Lake were comparable with that reported by Chirinos *et al.* (0.4 mg·kg<sup>-1</sup>) in sediments of Laguna Chica de San Pedro lake in Chile [80] and in Nylandssjon Lake located in the Nordingr  peninsula (northern Sweden) (2.3 mg·kg<sup>-1</sup>) [60]. Data are in line with previous studies on soils which concluded that Ti in the humic layer was due to mixing from underlying mineral soils. This is also in line with the sediment survey, which concluded that Ti in sediment was dominated by geological sources [81].

### 3.4. Correlation Analysis

In a complex lacustrine system, variations of any environmental factors is not independent. But it is interdependent with other environmental parameters, which can be analyzed by correlation method. Correlation analysis can estimate the strength and direction of linear relationships between pairs of continuous variables. The correlation between water quality parameters and the geochemical parameters in the three lakes showed limited relationships (**Table 5**). For example, sand was negatively correlated with  $Wc_s$  (-0.76) in El-Burullus Lake. While,

**Table 5.** Correlation coefficients of water quality parameters and sediments of geochemical characteristics of El-Burullus, El-Manzala, and El-Bardawil Lakes during 2019.

lake	DO(w)	BOD(w)	COD(w)	pH(w)	S%(w)	Sand(S)	Silt(S)	H <sub>2</sub> S(S)	OC(S)	WC(S)	
El-Burullus	DO(w)	1.000									
	BOD(w)	0.471	1.000								
	COD(w)	0.518	0.393	1.000							
	pH(w)	0.083	0.011	0.385	1.000						
	S%(w)	-0.138	0.027	0.173	-0.347	1.000					
	Sand(S)	0.071	-0.309	0.135	-0.138	0.590	1.000				
	Silt(S)	-0.071	0.309	-0.135	0.138	-0.590	-1.000	1.000			
	H <sub>2</sub> S(S)	0.344	0.071	0.124	-0.673	0.451	0.448	-0.448	1.000		
	OC(S)	0.045	0.166	0.176	0.494	-0.478	-0.417	0.417	-0.458	1.000	
	WC(S)	0.093	0.080	0.075	0.478	<u>-0.758</u>	-0.745	0.745	-0.520	0.819	1.000
	El-Manzala	DO(w)	1.000								
BOD(w)		-0.174	1.000								
COD(w)		-0.652	-0.016	1.000							
pH(w)		<u>0.794</u>	-0.376	-0.398	1.000						
S%(w)		0.291	-0.310	0.455	0.262	1.000					
Sand(S)		0.464	-0.194	-0.619	0.489	-0.249	1.000				
Silt(S)		-0.464	0.194	0.619	-0.489	0.249	-1.000	1.000			
H <sub>2</sub> S(S)		-0.601	0.361	0.525	-0.779	0.213	-0.582	0.582	1.000		
OC(S)		0.079	-0.224	0.025	0.283	0.028	-0.283	0.283	-0.091	1.000	
WC(S)		-0.002	-0.118	-0.002	-0.037	0.081	-0.584	0.584	0.197	0.787	1.000
El-Bardawil		DO(w)	1.000								
	BOD(w)	<u>0.861</u>	1.000								
	COD(w)	0.161	0.414	1.000							
	pH(w)	0.613	0.549	0.242	1.000						
	S%(w)	-0.403	-0.056	0.491	-0.175	1.000					
	Sand(S)	-0.051	-0.194	-0.187	0.194	-0.352	1.000				
	Silt(S)	0.051	0.194	0.187	-0.194	0.352	-1.000	1.000			
	H <sub>2</sub> S(S)	0.628	<u>0.880</u>	0.424	0.203	0.152	-0.528	0.528	1.000		
	OC(S)	0.243	0.360	0.422	0.162	0.464	-0.810	<u>0.810</u>	0.560	1.000	
	WC(S)	0.076	0.266	0.424	-0.047	0.525	-0.900	<u>0.900</u>	0.569	0.953	1.000

in El-Manzala Lake, DO positively correlated with pH<sub>w</sub> (0.74), pH negatively correlated with H<sub>2</sub>S<sub>s</sub> (-0.78). Similar with other lakes, DO was positively correlated with BOD<sub>w</sub> (0.86). Moreover, BOD<sub>w</sub> positively correlated with H<sub>2</sub>S<sub>s</sub> (0.88), silt positively correlated with Wc<sub>s</sub> (0.90), and OC<sub>s</sub> (0.81) in El-Bardawil Lake. The examined metals seemed to be less influenced by many water environmental parameters, such as T, pH, DO etc., and the geochemical parameters, such as

Sand (%), Silt (%), organic carbon, water content, H<sub>2</sub>S, etc., as inferred from the relatively very few correlations between metals and either with water environmental parameters and/or geochemical parameters. Except for El-Burullus Lake in which Mo negatively correlated with H<sub>2</sub>S<sub>s</sub> (−0.60), and positively correlated with IP<sub>w</sub> (0.76). and Sn negatively correlated with OC<sub>s</sub> (−0.61), WC<sub>s</sub> (−0.79), and Silt<sub>s</sub> (−0.65), no other relationships were found in any of the three Egyptian lakes between the studied metals and either with water environmental parameters and/or geochemical parameters (**Table 6**) suggesting that the above environmental factors and the geochemical parameters may probably not regulate the distribution behaviors of metals in the sediments of the three lakes. On the other hand, examination of the correlations between the distribution of these metals with other water quality parameters and/or the geochemical parameters in surface sediments cleared out that the above environmental factors may probably do not regulate the distribution behaviors of these metals in such a complex ecosystems of the northern Egyptian lakes subjected to severe anthropogenic activities resulting from the excessive discharges of different agricultural, industrial, and sewage effluents. Therefore, the above two parameters (*i.e.*, water quality parameters and the geochemical parameters) did not present strong significant correlations between each other and/or with most of the sedimentary metals. It was an interesting finding that the distribution of most different examined metals showed generally strong significant positive correlations with each other in the three Egyptian Lakes (**Table 5**). Ge distributions in El-Burullus Lake sediments was strongly correlated with Zr (0.94), Hf (0.90), Ta (0.89), Ti (0.79) and W (0.81). On the meantime, Zr distribution was positively correlated with Hf (0.97), Ta (0.92), Ti (0.81), Sb (0.97), and W (0.73). On the other hand, Hf positively correlated with Ta (0.95), Ti (0.84), Sb (0.75), and W (0.92). Ta positively correlated with Ti (0.86). The situation was different in El-Manzala Lake where Ge was positively correlated with Sn (0.72). On the meantime, a strong positive correlation was observed between Zr and several metals such as Hf (0.94), Ta (0.95), W (0.92), Ti (0.97), and Sb (0.80). Similar with Zr to some extent, Ta showed a very strong positive correlation with Ti (0.90), W (0.91), and Sb (0.76). Ti positively correlated with W (0.94). Sb positively correlated with Ti (0.78), and W (0.79). In El-Bardawil Lake Ge showed a positive correlation with Zr (0.81), Hf (0.76), and Ta (0.69), Zr positively correlated with Hf (0.97), Ta (0.96), Sn (0.85). Hf positively correlated with Ta (0.97), Sn (0.86). Ta positively correlated with Sn (0.92). On contrast to all other metals, Sn was negatively correlated with Ti (−0.63) (**Table 5**). This suggests that the studied metals are generally not affected by the complex situation of these lakes and their spatial distribution still not affected by the different anthropogenic inputs such as municipal wastewater, manufacturing industries, and agricultural activities discharged inside the three lakes. Moreover, the high correlations among these metals are a further evidence of the absence of any anthropogenic source of contamination by these metals in the three lakes.

**Table 6.** Correlation coefficients of sediment measurements of the three Egyptian Northern Lakes.

Lake	Ge	Zr	Hf	Ta	Mo	Sn	Sb	Ti	W	Tl	Sand	Silt	H <sub>2</sub> S	OC	WC	IP	OP	TP	TN	
El-Burullus	Ge	1.000																		
	Zr	<u>0.935</u>	1.000																	
	Hf	<u>0.898</u>	<u>0.974</u>	1.000																
	Ta	<u>0.879</u>	<u>0.920</u>	<u>0.883</u>	1.000															
	Mo	0.090	0.188	0.199	0.138	1.000														
	Sn	-0.046	-0.052	-0.009	-0.056	-0.321	1.000													
	Sb	-0.100	-0.027	0.018	0.071	-0.112	0.538	1.000												
	Ti	<u>0.789</u>	<u>0.814</u>	<u>0.790</u>	<u>0.859</u>	-0.087	0.259	0.203	1.000											
	W	<u>0.807</u>	<u>0.734</u>	<u>0.764</u>	0.652	0.285	-0.072	-0.014	0.469	1.000										
	Tl	0.125	0.020	0.014	0.120	-0.337	0.378	0.252	0.518	0.028	1.000									
	Sand	0.230	0.086	0.056	0.234	-0.196	0.647	0.400	0.333	0.217	0.396	1.000								
	Silt	-0.230	-0.086	-0.056	-0.234	0.196	-0.647	-0.400	-0.333	-0.217	-0.396	-1.000	1.000							
	H <sub>2</sub> S	-0.172	-0.241	-0.258	-0.208	<u>-0.601</u>	0.261	0.226	0.013	-0.249	0.436	0.448	-0.448	1.000						
	OC	-0.285	-0.263	-0.240	-0.249	0.500	<u>-0.610</u>	-0.286	-0.500	-0.136	-0.557	-0.417	0.417	-0.458	1.000					
	WC	-0.293	-0.218	-0.253	-0.258	0.348	<u>-0.789</u>	-0.503	-0.551	-0.228	-0.658	-0.745	0.745	-0.520	0.819	1.000				
	IP	0.189	0.248	0.259	0.212	<u>0.755</u>	-0.340	-0.147	0.027	0.077	-0.468	-0.299	0.299	-0.647	0.591	0.425	1.000			
	OP	-0.044	-0.033	0.049	-0.078	-0.571	0.489	0.314	0.371	-0.121	0.756	0.127	-0.127	0.542	-0.597	-0.627	-0.571	1.000		
	TP	0.192	0.272	0.354	0.191	0.439	-0.007	0.082	0.339	-0.007	0.060	-0.256	0.256	-0.333	0.221	-0.004	0.735	0.136	1.000	
	TN	-0.044	0.088	0.160	-0.113	-0.306	0.582	0.119	0.255	-0.238	0.210	-0.137	0.137	0.075	-0.470	-0.371	-0.133	0.652	0.378	1.000
	El-Manzala	Ge	1.000																	
Zr		-0.351	1.000																	
Hf		-0.014	<u>0.937</u>	1.000																
Ta		-0.128	<u>0.935</u>	0.950	1.000															
Mo		-0.142	0.146	0.140	0.016	1.000														
Sn		<u>0.715</u>	-0.174	0.053	0.042	-0.358	1.000													
Sb		-0.292	0.795	0.745	<u>0.758</u>	-0.127	-0.014	1.000												
Ti		-0.489	<u>0.969</u>	0.842	<u>0.897</u>	0.103	-0.286	<u>0.789</u>	1.000											
W		-0.272	<u>0.968</u>	0.921	<u>0.911</u>	0.036	-0.165	<u>0.791</u>	<u>0.943</u>	1.000										
Tl		-0.233	-0.151	-0.213	-0.183	-0.080	-0.445	0.050	-0.056	-0.144	1.000									
Sand		-0.310	0.030	-0.086	0.082	-0.297	0.129	-0.093	0.070	-0.050	0.062	1.000								
Silt		0.310	-0.030	0.086	-0.082	0.297	-0.129	0.093	-0.070	0.050	-0.062	-1.000	1.000							
H <sub>2</sub> S		0.020	0.631	0.662	0.494	0.089	-0.161	0.520	0.562	0.722	-0.116	-0.582	0.582	1.000						
OC		0.449	-0.546	-0.425	-0.580	0.391	-0.004	-0.602	-0.566	-0.477	0.185	-0.283	0.283	-0.091	1.000					
WC		0.336	-0.428	-0.335	-0.548	0.367	-0.173	-0.438	-0.457	-0.402	0.208	-0.584	0.584	0.197	0.787	1.000				
IP		0.488	-0.073	0.103	0.013	-0.152	0.123	0.173	-0.113	0.097	0.120	-0.757	0.757	0.487	0.175	0.225	1.000			
OP		0.580	-0.243	-0.062	-0.031	-0.169	0.580	-0.229	-0.299	-0.141	-0.272	0.277	-0.277	-0.346	0.340	-0.212	0.108	1.000		
TP		0.554	-0.091	0.106	0.022	-0.168	0.199	0.150	-0.138	0.089	0.080	-0.703	0.703	0.439	0.212	0.189	0.991	0.238	1.000	
TN		-0.129	0.499	0.474	0.354	-0.202	-0.144	0.675	0.455	0.617	-0.139	-0.396	0.396	0.722	-0.258	-0.040	0.394	-0.158	0.371	1.000



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