

GEOCHEMICAL CHARACTERISTICS OF RARE EARTH ELEMENTS AND SELECTED TRACE ELEMENTS FROM THE MĂNĂILA ORE DEPOSIT (EASTERN CARPATHIANS)

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Abstract: The Mănăila ore deposit lies in the Tulgheș Group a Cambrian-Ordovician island arc complex metamorphosed under greenschist facies conditions. Mănăila ore deposits together with other deposits located in the Tg3 formation of the Tulgheș Group is considered by several authors to be of Kuroko type. The main purpose of the paper is to present the geochemical interpretation for REE, and selected trace from the Mănăila ore, Romania. The study was conducted on a series of ten samples collected from the Mănăila ore deposit separated in rock samples and mineralized samples. The data obtained from the analyses of REE was normalized to the chondritic value and plotted in spider diagrams. For the rock samples the range of REE contents is : 5.1 - 58.6 $\mu\text{g}\cdot\text{g}^{-1}$ for La, 9.8 – 110 $\mu\text{g}\cdot\text{g}^{-1}$ for Ce, 1.1 – 12.2 $\mu\text{g}\cdot\text{g}^{-1}$ for Pr, 4.8 – 40.9 $\mu\text{g}\cdot\text{g}^{-1}$ for Nd, 1.1 – 7.3 $\mu\text{g}\cdot\text{g}^{-1}$ for Sm, 0.21 – 1.54 $\mu\text{g}\cdot\text{g}^{-1}$ for Eu, 1.71 – 7.3 for Gd, 0.28 – 1.05 $\mu\text{g}\cdot\text{g}^{-1}$ for Tb, 1.5 – 6 $\mu\text{g}\cdot\text{g}^{-1}$ for Dy, 0.33 -1.3 $\mu\text{g}\cdot\text{g}^{-1}$ for Ho, 0.99 - 4.2 $\mu\text{g}\cdot\text{g}^{-1}$ for Er, 0.15 – 0.63 $\mu\text{g}\cdot\text{g}^{-1}$ for Tm, 1 – 4.1 $\mu\text{g}\cdot\text{g}^{-1}$ for Yb, 0.14 -0.58 $\mu\text{g}\cdot\text{g}^{-1}$ for Lu. The range of REE contents for mineralized samples is: 3.9 – 4.2 $\mu\text{g}\cdot\text{g}^{-1}$ for La, 8.3 – 9.8 $\mu\text{g}\cdot\text{g}^{-1}$ for Ce, 0.93 – 1.1 $\mu\text{g}\cdot\text{g}^{-1}$ for Pr, 3.2 – 4.5 $\mu\text{g}\cdot\text{g}^{-1}$ for Nd, 0.74 – 1.2 $\mu\text{g}\cdot\text{g}^{-1}$ for Sm, 0.15 – 0.2 $\mu\text{g}\cdot\text{g}^{-1}$ for Eu, 1.2 -1.3 $\mu\text{g}\cdot\text{g}^{-1}$ for Gd, 0.25 – 0.27 $\mu\text{g}\cdot\text{g}^{-1}$ for Tb, 1.4-1.7 $\mu\text{g}\cdot\text{g}^{-1}$ for Dy, 0.34-0.42 $\mu\text{g}\cdot\text{g}^{-1}$ for Ho, 0.94-1.1 $\mu\text{g}\cdot\text{g}^{-1}$ for Er, 0.16-0.16 $\mu\text{g}\cdot\text{g}^{-1}$ for Tm, 0.95-1.1 $\mu\text{g}\cdot\text{g}^{-1}$ for Yb, 0.14-0.18 $\mu\text{g}\cdot\text{g}^{-1}$ for Lu. An Eu anomaly is visible for the both group of samples, which in case of the mineralized samples is slightly more pronounced.

Keyword: ICP-MS, Mănăila ore deposit, trace and rare earth elements, Tulgheș Group, Kuroko.

1. INTRODUCTION

The Tulgheș metamorphic unit is the major Mn producer in Romania and its represents a notable percentage from the output of Pb, Zn, Cu and pyrite as well as of barite (Balintoni et al., 2010). In the Eastern Carpathians, the Tulgheș Group consists of the following lithostratigraphic components (Kräutner, 1988) from the bottom up:

- Tg1 quartzitic formation,
- Tg2 graphitic formation with metacherts and associated stratiform manganese carbonate ore deposits,
- Tg3 rhyolitic volcano-sedimentary formation with metamorphosed, stratiform Kuroko-type Cu-Pb-Zn volcanogenic massive sulfide deposits,
- Tg4 quartzite-phyllite formation,
- Tg5 graphite-bearing greenschists with some thin limestone layers.

The mineralization from the Mănăila ore deposit

is located in the Tg3 formation of the Tulgheș Group which contain stratiform volcano-sedimentary base metal of Kuroko type (e.g. Burloaia, Fundu Moldovei, Mănăila, Leșu Ursului, Bălan), (Munteanu, 2010).

This paper discusses the data for the REE, and other trace elements: Sr, Y, Rb, Zr, Ba, etc., present in ten samples collected from Mănăila ore deposit, located in the NE area of Eastern Carpathians (Romania), in the Tulgheș terrane (Balintoni et al., 2009) from the Putna Nappe, represented by Tulgheș Group. The main objective of this study is to characterize the mineralization using the data obtained by Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

2. GEOLOGICAL SETTING

The Eastern Carpathians comprise metamorphic terrains with sedimentary Mesozoic cover, known as the Crystalline-Mesozoic zone, framed by Cretaceous flysch formations to the east

and a Neogene volcanic arc to the west (Munteanu et al., 2004). The rock cropping out in the Mănăila ore deposit belongs to the Tulgheș metamorphic unit, Leșu Ursului sub-unit (Balintoni et al., 2009), a Cambrian - Ordovician island arc complex (Munteanu & Tatu, 2003).

Several authors have considered the metal sulphides ore deposits from the Tulgheș Group (as Mănăila ore deposit) to be of Kuroko type (Kräutner, 1965, 1989; Balintoni, 1997; Mureșan 2002 a, b, Balintoni et al., 2009). The Mănăila ore deposit is located 6 km NE of the Valea Stânei village, on the southern slope of the Mănăila Peak from Obcina Mestecănișului (Eastern Carpathians) and is comprised exclusively of members Moroșan and Fundu Moldovei from the Tg3 formation. The study area is situated between the following coordinates: N 47°35' and E 25°13', with an elevation of 1190 m, and have the next limits (Fig. 1):

- Pârâul Tâtarca at NW
- Vârful Mănăila at NE
- Pârâul Păltiniș at W, SW.

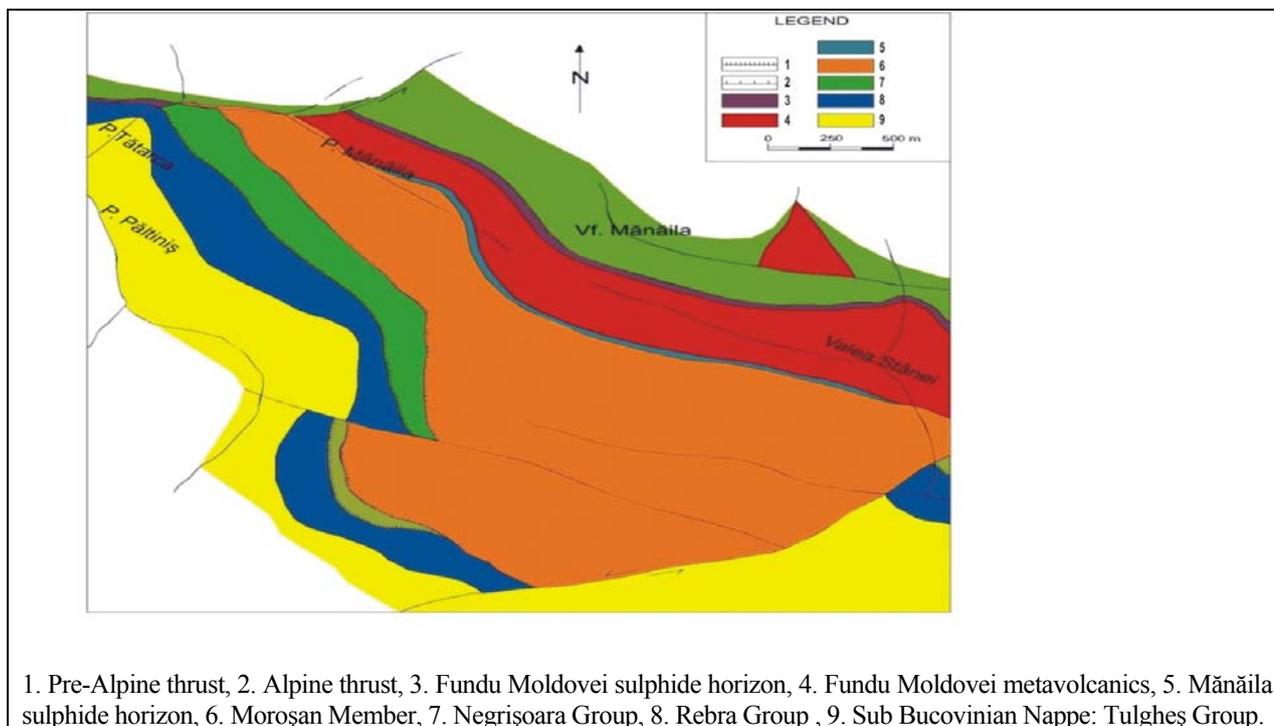
The mineralization have the form of lens layer, concordant with the surrounding crystalline schist with which are intercalated through certain schists with sulphide impregnation (Dimitrescu, 1983).

The mineralization from the Mănăila ore deposits is represented by disseminated pyrite, chalcopyrite, sphalerite and galena in the host rocks, with contents without significant economic value. The mineralogical composition in the Mănăila ore deposits is relatively simple: pyrite, chalcopyrite, sphalerite,

galena and tetrahedrite, respectively in paragenesis with quartz, chlorite and muscovite, as pointed out in a previous paper (Moldoveanu et al., 2010). Mureșan (2005) stated that regionally metamorphosed mineralizations (RMM) of Kuroko type sulphides are predominantly composed of pyrite (10-85% of ore), and subordinate chalcopyrite (3-10%), while sphalerite and galena are often sporadic and occur in small amounts (usually 0-1%). In his opinion the composition of syngenetic ore of Kuroko type have not changed substantially by regional metamorphism because sulfur and most of metals from sulphide (Cu, Pb, Zn, etc.) cannot be incorporated into the metamorphic silicate structure. Most RMM are syngenetic (concordant) and they were formed in the Early Ordovician (Mureșan, 2005).

3. MATERIALS AND METHODS

For the purpose of the present study 10 samples (representing quartzitic schists, quartzitic schist with sulphide impregnation, quartzitic sericite chlorite schists, table 1, and mineralized samples: pyrite with chalcopyrite, table 2) were analyzed for 42 elements, included REE using ICP-MS at the Institute of Geology and Mineralogy, Cologne University, Germany. Samples were crushed and subsequently reduced to a very fine powder by grinding in an agate ball mill. This powder was dried in the oven at 105°C, for four hours in order to remove the moisture. Later the same powder was heated overnight at 1000°C to determine the loss on ignition (LOI %).



1. Pre-Alpine thrust, 2. Alpine thrust, 3. Fundu Moldovei sulphide horizon, 4. Fundu Moldovei metavolcanics, 5. Mănăila sulphide horizon, 6. Moroșan Member, 7. Negrișoara Group, 8. Rebra Group, 9. Sub Bucovinian Nappe: Tulgheș Group.

Figure 1. Geological map of the Mănăila ore deposits (modified after Podașcă, 2004)

Table 1. Trace element contents of the rocks of Mănăila ore deposit ($\mu\text{g}\cdot\text{g}^{-1}$)

Sample	07M	010M	015M	011M	012M	013M	014 M	018M
La	5.1	17.5	12.7	7.5	6.7	58.6	8.4	47.7
Ce	9.8	36.9	54.1	15.6	15.5	110	18.7	104
Pr	1.2	4.7	3.2	1.9	2.01	12.2	2.2	10.9
Nd	4.8	19.6	12.9	7.04	7.6	40.9	8.6	38.6
Sm	1.1	4.8	3.08	1.7	2.3	6.7	2.3	7.3
Eu	0.37	0.53	0.56	0.21	0.4	1.48	0.35	1.54
Gd	1.8	5.7	4.6	1.71	2.91	5.8	2.6	7.3
Tb	0.3	0.95	0.85	0.28	0.53	0.83	0.51	1.05
Dy	1.9	6.04	6.02	1.5	3.2	3.9	3.3	5.4
Ho	0.34	1.2	1.3	0.33	0.71	0.74	0.8	1.1
Er	1	3.9	4.2	0.99	2.1	1.9	2.4	2.9
Tm	0.15	0.61	0.63	0.19	0.37	0.29	0.43	0.45
Yb	1	4.09	4.1	1.2	2.5	1.7	2.8	2.8
Lu	0.14	0.58	0.52	0.23	0.46	0.26	0.49	0.46
P	212	37.1	721	123	99.2	962	45.7	778
K	2128	24889	27622	16744	10650	41658	21991	27998
Sc	1.3	5.5	5.9	2.9	4.2	1.2	5.03	14.4
Ti	321	471	2165	201	211	5458	250	5278
V	44.5	24.7	93.3	14.6	25.6	188	26.9	163
Mn	2136	111	2078	40.6	513.8	125.1	23.5	1422
Co	1.7	0.41	6.6	6.71	0.21	1.97	2.7	20.6
Ni	6.9	3.5	12.6	2.6	2.9	9.3	9.3	45.6
Cu	18.04	41.6	105	60.2	18.3	537	105	72.2
Zn	50.4	142	72.5	188	57.4	106	49.4	155
Ga	2.3	20.4	18.7	11.1	11.7	22.2	13.9	28.4
As	5.1	6.8	5.6	75.6	0.72	19.4	17.6	18.07
Rb	5.1	113	163	76.1	50.2	206	1 07	122
Sr	62.7	68.9	56.9	16.5	92.4	104	19.5	96.1
Y	9.6	32.9	38.7	7.2	18.1	14.1	22.5	26.8
Zr	11.7	102	103	56.6	52.6	159	64.5	133
Nb	1.7	7.8	11.9	3.7	4.3	13.8	5.6	14.6
Mo	0.29	1.5	0.04	1.8	0.41	7.1	20.4	2.1
Ag	0.16	0.51	0.52	0.65	0.23	0.55	1.21	0.57
Cd	0.32	0.08	0.43	0.27	0.15	0.23	0.08	0.07
Sb	0.27	0.56	0.45	0.8	0.26	1.82	0.86	0.54
Ba	276	1348	735	494	1187	639	1028	1343
Hf	0.37	5.1	3.6	5.2	2.6	4.7	3.3	4.6
Ta	28.9	0.89	1.1	0.44	0.13	0.82	0.56	1.26
Pb	50.01	156	23.2	33.8	22.8	1449	41.2	28.4
Bi	0.61	0.72	0.26	11.4	0.23	2.98	14.1	0.82
Th	1.5	13.3	12.9	7.7	7.8	5.4	6.4	16.2
U	0.44	4.07	3.4	2.1	1.6	5.02	2.7	3.1

07M = quartzitic schist, 010M = quartzitic schist, 015M= sericite-quartzite schist, 011M = quartzitic sericite chlorite schist, 012M = quartzitic schist, 013M = quartzitic schist, 014M= quartzitic schist with sulphide impregnation, 018M = quartzitic schist (M = Mănăila ore).

Accurately weighed 200 mg of sample powder were transferred into PTFE digestion vessels and wetted with a few drops of aqua distilled (Milli-Q 18 M Ω .cm). HF (3 ml) and HClO₄ (3 ml) were added. Samples were digested for 16 hours at 180°C under pressure. After cooling, samples were evaporated at

180°C for approximately 4 hours to near dryness, redissolved with 5 ml HCl and heated again at 160°C to incipient dryness. The moist hot residues were taken up again in 5 ml HCl and, after closing the digestion system, treated at 130°C for 2 hours. After cooling, the solutions were evaporated to incipient dryness again

and the hot sample cakes carefully redissolved in 2 ml HCl and 10 ml water. The resulting, generally clear solutions were transferred into 50 ml volumetric flasks and stored in PE bottles. Dissolution was carried out using in all stages Merck Suprapur grade reagents (HF, HClO₄, HNO₃, HCl). Measurements were made on a Sciex/Perkin Elmer ELAN 6000 ICP-MS (quadrupole mass spectrometer).

4. RESULTS AND DISCUSSIONS

The contents of the trace elements in the investigated samples from Mănăila ore deposit are listed in table 1, for the rock samples and in table 2 for the

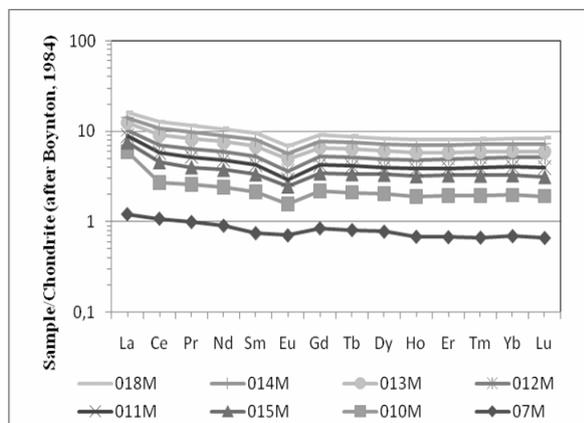
mineralized samples. REE concentrations in rocks are usually normalized to a common reference standard, which most commonly comprises the values for chondritic meteorites. Chondritic normalization therefore, has two important functions. Firstly, it eliminates the abundance variation between odd and even atomic number elements and secondly, it allows any fractionation of the REEs group relative to chondritic meteorites to be identified (Rollinson, 1993).

The set of results (Tables 1, 2) was chondrite - normalized, using the data after Boynton (1984) and plotted in two spider diagrams (Fig. 2 a, b).

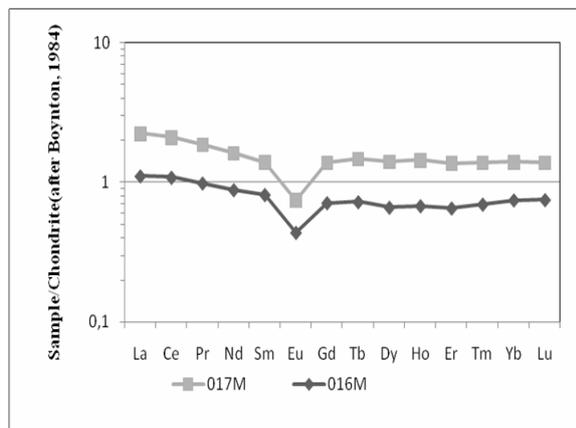
Table 2. Trace element contents of the mineralized rocks of Mănăila ore deposit ($\mu\text{g}\cdot\text{g}^{-1}$)

Sample	016M	017M	Sample	016M	017M
La	3.9	4.2	Ni	3.7	6.3
Ce	9.8	8.3	Cu	1208	2676
Pr	1.1	0.93	Zn	1170	249
Nd	4.5	3.2	Ga	9.09	0.38
Sm	1.2	0.74	As	233	30.8
Eu	0.2	0.15	Rb	7.7	0.44
Gd	1.3	1.2	Sr	2	0
Tb	0.25	0.27	Y	9.7	15.04
Dy	1.4	1.7	Zr	29.01	3.8
Ho	0.34	0.42	Nb	2.3	1.1
Er	0.94	1.1	Mo	17.6	41.6
Tm	0.16	0.16	Ag	4.3	1
Yb	1.1	0.95	Cd	2.2	0.51
Lu	0.18	0.14	Sb	11.7	9.9
P	53.7	57.2	Ba	233	238
K	1663	157	Hf	1.5	0.21
Sc	1.41	0	Ta	0.28	0.08
Ti	129	58.6	Pb	247	61
V	18.6	25.9	Bi	67.1	40
Mn	181	20.3	Th	4.1	0.46
Co	44.2	38.2	U	1.6	0.38

016M= pyrite and chalcopyrite, 017 = pyrite with chalcopyrite, (M= Mănăila ore)



(a)



(b)

Figure 2 a, b. Chondrite - normalized REE pattern of metamorphic rocks (a) and mineralized rocks (b) from the Mănăila area.

Table 3. Correlation matrix for the rock samples

	Cu	Zn	Pb	Bi	Sr	Zr	Mo	Ag	Cd	Ba	Ti	Y	Co	As	Rb
Cu	1														
Zn	0.01	1													
Pb	0.97	-0.01	1												
Bi	0.07	0.67	0.04	1											
Sr	0.44	-0.35	0.48	-0.69	1										
Zr	0.69	0.34	0.62	-0.12	0.52	1									
Mo	0.93	0.27	0.94	0.21	0.43	0.7	1								
Ag	0.34	0.82	0.23	0.53	-0.24	0.67	0.43	1							
Cd	0.08	-0.41	-0.01	0.13	-0.47	-0.28	-0.19	-0.09	1						
Ba	-0.21	0.23	-0.19	-0.41	0.48	0.42	-0.08	0.16	-0.78	1					
Ti	0.69	0.17	0.61	-0.17	0.62	0.85	0.7	0.44	-0.18	0.17	1				
Y	-0.15	-0.06	-0.22	-0.57	0.19	0.43	-0.27	0.23	-0.04	0.6	0.16	1			
Co	-0.11	0.47	-0.24	0.05	0.08	0.36	-0.01	0.46	-0.23	0.29	0.56	0.21	1		
As	0.04	0.75	0	0.98	-0.67	-0.05	0.19	0.61	0.08	-0.34	-0.08	-0.51	0.22	1	
Rb	0.75	0.23	0.65	-0.04	0.33	0.93	0.66	0.68	0.03	0.19	0.73	0.45	0.18	0	1

Table 4. Correlation factor between rock samples and mineralized samples

	Cu	Zn	Pb	Bi	Sr	Zr	Mo	Ag	Cd	Ba	Ti	Y	Co	As	Rb
Cu	1														
Zn	0.4	1													
Pb	0.04	0	1												
Bi	0.73	0.9	-0.05	1											
Sr	-0.63	-0.59	0.37	-0.78	1										
Zr	-0.46	-0.32	0.56	-0.53	0.71	1									
Mo	0.99	0.38	0.01	0.72	-0.64	-0.47	1								
Ag	0.41	0.99	0.02	0.9	-0.58	-0.29	0.39	1							
Cd	0.41	0.96	0	0.89	-0.61	-0.4	0.38	0.97	1						
Ba	-0.56	-0.42	-0.1	-0.61	0.7	0.61	-0.53	-0.43	-0.55	1					
Ti	-0.22	-0.25	0.59	-0.38	0.66	0.85	-0.23	-0.22	-0.3	0.35	1				
Y	-0.28	-0.34	-0.19	-0.43	0.37	0.51	-0.29	-0.3	-0.34	0.65	0.27	1			
Co	0.78	0.77	-0.16	0.9	-0.68	-0.41	0.78	0.77	0.73	-0.46	-0.15	-0.25	1		
As	0.32	0.97	0	0.87	-0.63	-0.32	0.3	0.96	0.93	-0.48	-0.27	-0.45	0.7	1	
Rb	-0.44	-0.41	0.57	-0.56	0.63	0.95	-0.46	-0.37	-0.43	0.47	0.77	0.54	-0.5	-0.38	1

The variation in total rare earth elements is relatively large ($TREE = 26.3-23.4 \mu\text{g}\cdot\text{g}^{-1}$ for the mineralized samples, $TREE = 29-245 \mu\text{g}\cdot\text{g}^{-1}$ for the rock samples). In figure 2 a, b, the samples present negative Eu/Eu^* anomalies ranging in the limits:

- $\text{Eu}/\text{Eu}^* = 0.30 - 0.80$ for the rock samples;
- $\text{Eu}/\text{Eu}^* = 0.47 - 0.48$ for the mineralized samples.

Chondrite normalized REE patterns are moderately fractionated with La_N/Yb_N values between 1.9-22.5 for the rock samples and 2.3-2.9 for the mineralized samples which are less fractionated. The rock samples show LREE enrichment ($\text{La}_N/\text{Sm}_N = 1.80 - 4.08$) and HREE depletion ($\text{Gd}_N/\text{Yb}_N = 0.74 - 2.68$). The mineralized samples are characterized also by LREE enrichment ($\text{La}_N/\text{Sm} = 1.97-3.57$) and HREE depletion ($\text{Gd}_N/\text{Yb}_N = 0.92 - 1.04$). There are not significant differences between the LREE enrichment and HREE depletion in the rock samples and mineralized samples.

Based on the correlation matrix can be observed that Cu has strongly positive correlation with Pb, and Mo and had a less strongly positive correlation with Rb, Zr and Ti (Table 3).

A good correlation can be seen between Zn-Bi, Zn-Ag and Zn-As, Pb-Zr, Pb-Mo, Pb - Rb (Table 3). A strong positive correlation is also between Bi-As and Zr-Rb. In the mineralized samples good correlation are between Cu-Mo, Zn-Ag, Zn-Cd, Zn-As, Zr-Rb (Table 4).

Negative correlation are between: Sr-Bi, Sr-As, Y-As, Y-Bi, Cd-Ba. The correlation between different components may vary from one sample to another, in the rock samples Cu has positive correlation with Pb and no correlation in the mineralized samples. The high $1348 \mu\text{g}\cdot\text{g}^{-1}$ Ba content is not unusual since barite mineralization occur in the black cherts of the Tg2 formation (Munteanu et al., 2004), (Table 1).

5. CONCLUSIONS

For this study 10 samples were collected from the Mănăila ore deposits, and analyzed through ICP-MS for trace elements including REE. The REE data from rocks of the Mănăila deposit are provided here for the first time. The samples used for the analyses were separated in rock samples and mineralized samples, and plotted in two spider diagrams. In the REE diagrams it emphasizes for the two groups of rocks an enrichment of LREE compared to HREE and a negative Eu anomaly. The Eu anomaly is slightly more pronounced for the mineralized samples.

The study of the correlation matrix shows strong positive correlation between: Cu-Pb, Cu-Mo, Zn-Ag, Pb-Mo, Bi-As, Zr-Ti, Zr-Rb and good positive correlation for: Cu-Zr, Cu-Ti, Cu-Rb, Zn-Bi, Zn-As, Pb-Zr, Pb-Rb, Sr-Ti, Zr-Ag, Mo-Rb, Ag-As, Ag-Rb, Ti-Rb.

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