Analysis of river and stream sediments from the Kumtor gold mine area, Kyrgyz Republic

Final Report

Ljubljana, 16 December 2012

Content of Report

Note: the report was prepared by Peter Stegnar, Ph.D., Professor, Scientific Advisor of the Jožef Stefan Institute and Head of projects at Technomedica, Ljubljana, Slovenia

Term of reference
Term of reference was analysis of river and stream sediments from the Kumtor gold mine area in Kyrgyzstan. The analysis included the determination of total concentrations of 31 chemical elements by an accredited analytical method of k_0-Instrumental Neutron Activation Analysis (INAA), in 10 representatively selected sampling points from the Kumtor industrial site.

Background information
At the end of September 2012, an enquiry was made whether the Jožef Stefan Institute (JSI), Ljubljana Slovenia, can get involved into activities of the Working group of the governmental State Commission. The Working group was dealing with environmental and mining-technical expertise related to environmental pollution situation at the Kumtor operating company site and wanted to include two independent and accredited international institutions into its activities. Peter Stegnar, a scientific advisor of the Jožef Stefan Institute, responded positively to the enquiry of the Working group and the arrangements have been made to perform the requested task. This should be done in the Department of Environmental Sciences of JSI that provided the requested accreditation certificate LP-090 awarded by the Slovenian Accreditation Agency (Annex 1).

It was agreed that Technomedica, d.o.o., Ljubljana, an enterprise specialized in managing environmental impact assessment projects, will act as a contracting party with the Working group represented by JSC Kyrgyzaltin, Bishkek. The relevant contract was signed by O.Artykbaev, Chairman of the Working group and D. Zhaparov, Chairman of Kyrgyzaltin from the Kyrgyz site, and by P.Stegnar, the authorized person on behalf of Technomedica. He was, as Head of projects at Technomedica, also given full responsibility for all scientific, technical and administrative matters of this assignment.

Work done
The sampling campaign to the Kumtor industrial site was carried out on 12 and 13 October 2012, by Peter Stegnar and Thomas Nordmann from Cologne, Germany, a consultant to the Institute of Hygiene of the Ruhr area, Gelsenkirchen, Germany. The Institute of Hygiene was another accredited institution, which participated in the project with an independent programme. The following persons joined the sampling campaign (field work) mission:, Erkingul Imankozhoeva (member of Parliament of the Kyrgyz Republic), Isakbek Torgoev, Kalya Moldogazieva, Rasul Artykbaev, Sherulan Darmanov (national experts and/or members of the Working group), Gulnura Toktosunova, secretary of the Working group and Nazira Kasenova, interpreter.

The sampling was done jointly with the Kumtor "environmental" team at the same sampling points (locations) and at the same time. P. Stegnar collected river and stream sediments from 9 selected locations in the Kumtor area and 1 sample of tailings material from the Kumtor tailings pond. T. Nordmann collected the sediment samples at the same locations together with water samples and additionally two soil samples: SK14 and SK 15 (Fig. 13), and measured basic chemical parameters in water.

The sediment samples were transferred to JSI simultaneously with P. Stegnar's travel to Slovenia and delivered to the Environmental Sciences Department on 15 October 2012. Preparation of samples and analyses took place from 16 October to 30 November 2012, when the analytical work was completed and analytical report submitted to Kyrgyzaltin by email. Based on the analytical report, the final report was prepared and being submitted electronically to Kyrgyzaltin on 17 December 2012 and in a paper version, sent by regular mail on 18 December 2012.

Sampling of river and stream sediments

The river and stream (creek) sediments were sampled at 10 sampling points (locations) of the Kumtor mining area (Fig. 1, Table 1). The selected sampling points include 2 locations outside a potential environmental impact area from the Kumtor mining and processing operations: SK1 – headwaters of Kumtor river and SK12 – headwaters of Arabel river, and 8 locations from the area, which could be affected by the Kumtor operation activities.

The sediments were sampled with a small plastic shovel into 1 litre polyethylene boxes together with water: about 500 grams of sediments and about 300 ml of water. The exact sampling locations (coordinates) were determined by GPS (Garmin, model GPSmap76CS) and the coordinates were promptly written on the sampling bottles (Fig. 2 and Fig. 6). The tailings material from the Kumtor tailings pond was collected into a 250 ml glass flask (Fig. 4 – right photo).

Fig. 1: Sediment sampling points at the Kumtor industrial area

Table 1: Coordinates of sediment sampling at Kumtor and the names of sampling points

Sampling point ID	Sampling point name	Latitude	Longitude
SK1	Headwaters of Kumtor river	N 41^0 54' 58.5"	E 78^0 12' 05.0"
SK3	Lysyi Creek – Waste Rock seepage (WR's)	N 41^0 53' 10.0"	E 78^0 12' 00.2"
SK4	Kumtor river Flume	N 41^0 53' 29.1"	E 78^0 10' 13.0"
SK5	Tailings pond	N 41^0 53' 48.0"	E 78^0 08' 01.8"
SK7	Chon Sarytor river below camp area	N 41^0 52' 47.4"	E 78^0 08' 53.5"
SK8	Sarytor stream - below waste dumps and SW Open Pit	N 41^0 50' 50.5"	E 78^0 09' 32.0"
SK9	End mixing zone (EMZ) of Kumtor river	N 41^0 50' 15.5"	E 78^0 06' 04.6"
SK10	Chon Sarytor river - below waste dumps and Central Open Pit	N 41^0 52' 14.3"	E 78^0 09' 25.8"
SK12	Headwaters of Arabel river	N 41^0 55' 57.4"	E 78^0 08' 29.3"
SK13	Seepage waters from tailings pond	N 41^0 53' 32.2"	E 78^0 08' 24.4"

Analysis

The concentrations of 36 elements were determined in each of the 10 samples of river and stream sediments by the k_0 method of Instrumental Neutron Activation Analysis (INAA) [Jacimovic et al., 2003] at the Jožef Stefan Institute, Ljubljana, Slovenia. The results (element concentrations) are presented in mg/kg dry weight of the air dried sediment samples that were homogenized after sieving through 1 mm plastic sieve. The final particle size of the samples was less than 0.22 mm. It should be noted that the results obtained are related only to the k_0-INAA method, which gives total concentrations of the determined elements. A comparison with other analytical methods, i.e. those involving chemical treatment (decomposition) of the samples prior to final analysis, could lead to incomparable analytical results of certain elements [Gaudino et al., 2007]. The detailed Analytical Report is presented in Annex 2.

Results and discussion

The total element concentrations in the sediment samples are presented in Table 2. The lowest concentrations of the majority of elements were determined in the sediments from head waters of the Kumtor river (SK1), which should not be affected by the Kumtor mining and ore processing activities. The values were several times to an order of magnitude lower in comparison to the concentrations of some selected elements from other sampling points (Figs. 7-12). The elevated element contents in the sediments from the sampling points SK3, SK4, SK7, SK8, SK9, SK10 and SK13 indicated a moderate influence from technologically enhanced sources (waste dumps and rock deposits, mill facilities) on the nearby environment. This could be a consequence of higher availability of the elements to the environment due to leaching and wash-out processes from technologically enhanced sources in the Kumtor affected area over an extended period of time. However, from the environmental pollution point of view these levels were still low and generally comparable to element concentrations in sediments from the sampling point SK12 (upper Arabel river). This location is considered as another location (besides SK1) that should not be affected by the Kumtor operating activities. It is worth to mention that uranium and thorium concentrations (Fig. 12) were within the limits of "clean geochemical background" [Kayukov et al., 2012]. They followed similar concentration pattern as the other elements and showed quite a constant ratio (U:Th = 1:3 – 1:4) between these two naturally occurring radioactive elements, which is typical for a non-polluted environment

A comparison of concentrations between the sampling points closer to Kumtor mill facilities at the central open pit and waste dumps at the southwest pit (Fig. 13), shows slightly higher concentrations of several elements in the sediments of Chon-Sarytor river (SK7 and SK10, Fig. 6) and Sarytor creek (SK8) as compared to the EMZ of the Kumtor river – SK9 (Fig. 5). This might be connected with the vicinity of Chon Sarytor river and Sarytor creek to operational mining facilities and their long term influence on its nearby environment.

The highest total concentrations of the majority of the analyzed elements were measured in the tailings material from the Kumtor tailings pond (SK5). The concentrations were not significantly higher in comparison to sediment samples from environmental sampling locations, but indicated a tendency of elements concentrating during milling of the ore for further gold extraction processes. For example, Au concentration was 0.6 mg/kg dry weight of the sample, which indicates that several 10s of tonnes of gold might be present in the tailing materials assuming the total volume of tailings to be around 80 million tonnes and the ratio of wet to dry mass of the tailing materials 1:1. Following the W concentration of 200 mg/kg, the total load of this element in the tailings could be several thousand tonnes considering a homogeneous distribution of tungsten and other elements in the tailing materials.

From the comparison of the element levels between SK5 (tailings pond) and SK13 (seepage waters from the tailings pond) sampling points, no influence (i.e. leaching) from the tailings pond site can be observed at the time of this field mission. The concentrations at SK13 were lower than those at SK5 and they were comparable with the levels at other environmental sampling points in the Kumtor industrial area.

Table 2: Total element concentrations in the Kumtor sediment samples

Sample	Sediment									
Sample code	SK1	SK3	SK4	SK5*	SK7	SK8	SK9	SK10	SK12	SK13
Element	mg/kg dry weight									
Ag (Silver)	< 0.4	< 0.7	< 0.6	< 0.9	< 0.7	< 0.6	< 0.6	< 0.4	< 0.7	< 0.6
As (Arsenic)	4.09	36.4	14.6	14.8	21.8	24.7	16.5	23.9	15.3	20.9
Au (Gold)	0.0017	0.117	0.218	0.61	0.065	0.056	0.243	0.060	< 0.0020	0.043
Ba (Barium)	1109	1294	1256	1860	877	781	993	1053	480	1275
Br (Bromine)	0.27	0.62	0.43	< 0.64	0.98	< 1.11	< 0.96	1.53	2.06	3.01
Ca (Calcium)	16275	18069	22826	47692	49722	20705	37693	18837	31088	37905
Cd (Cadmium)	< 0.92	< 1.98	< 1.41	< 2.42	< 3.19	< 3.41	< 4.48	< 5.36	< 1.87	< 5.35
Ce (Cerium)	34.5	98.5	71.8	110	120	96.7	87.3	116	96.9	85.8
Co (Cobalt)	3.32	17.7	9.04	19.8	17.8	20.7	14.2	17.3	14.4	14.2
Cr (Chromium)	13.7	67.9	35.7	78.4	74.3	66.1	58.1	67.6	71.3	64.4
Cs (Caesium)	1.66	2.74	2.45	2.31	5.55	2.73	4.16	3.22	8.32	4.47
Eu (Europium)	0.66	1.51	1.40	1.82	1.69	1.58	1.46	1.77	1.36	1.41
Fe (Iron)	12950	37471	34441	54732	39410	36724	32724	41117	36744	39178
Ga (Gallium)	< 25.0	40.2	31.5	40.6	47.1	42.4	34.3	50.8	27.6	52.3
Hf (Hafnium)	3.51	5.65	8.82	3.83	7.81	6.88	5.70	5.79	6.83	5.20
Hg (Mercury)	< 0.2	< 0.4	< 0.2	< 0.3	< 0.3	< 0.2	< 0.3	< 0.4	< 0.4	< 0.3
K (Potassium)	19240	31160	23290	34970	23340	29710	23420	27380	26850	25530
La (Lanthanum)	23.9	50.5	36.1	59.7	62.0	50.9	44.3	61.0	47.5	43.3
Mo (Molybdenum)	0.49	5.58	2.33	8.79	2.83	6.75	3.00	3.40	< 0.9	3.50
Na (Sodium)	35230	11270	24570	14170	10160	12280	17540	13490	15620	14110
Nd (Neodymium)	12.4	47.0	33.1	50.4	54.3	44.5	40.4	51.5	43.6	37.0
Rb (Rubidium)	67.9	130	88.1	147	115	121	104	116	148	121
Sb (Antimony)	0.68	2.24	1.68	6.17	2.08	1.88	1.93	2.13	1.06	2.49
Sc (Scandium)	3.99	12.4	7.86	15.3	13.0	12.8	11.0	13.6	13.9	11.7
Se (Selenium)	< 0.6	0.97	< 0.6	< 0.6	< 0.9	1.30	< 0.9	< 1.0	< 1.1	< 0.7
Sm (Samarium)	2.03	7.89	5.79	8.13	9.19	8.15	6.83	8.99	7.54	6.74
Sr (Strontium)	778	115	525	1547	368	120	349	166	203	232
Ta (Tantalum)	0.41	1.15	1.11	0.94	1.10	1.10	1.01	1.09	1.38	0.91
Tb (Terbium)	0.27	0.99	0.72	1.00	1.12	1.11	0.87	1.08	1.00	0.84
Te (Tellurium)	< 0.8	< 1.4	< 0.9	5.11	< 1.5	< 1.4	< 1.7	< 1.8	< 1.5	< 1.7
Th (Thorium)	4.29	13.1	8.87	16.6	17.1	13.3	12.1	14.8	14.7	12.6
U (Uranium)	1.31	4.81	3.03	4.06	4.23	5.00	3.44	3.61	3.35	3.86
W (Tungsten)	0.94	8.14	6.75	200	10.2	8.72	6.11	9.07	2.50	12.2
Yb (Ytterbium)	0.96	2.93	2.29	2.78	3.22	3.60	2.65	3.01	3.19	2.66
Zn (Zinc)	26.5	58.9	47.8	217	75.1	44.6	60.4	66.3	90.3	64.5
Zr (Zirconium)	124	235	358	138	260	241	231	218	270	196
< - limit of detection (LD)										
*tailings – sludge										

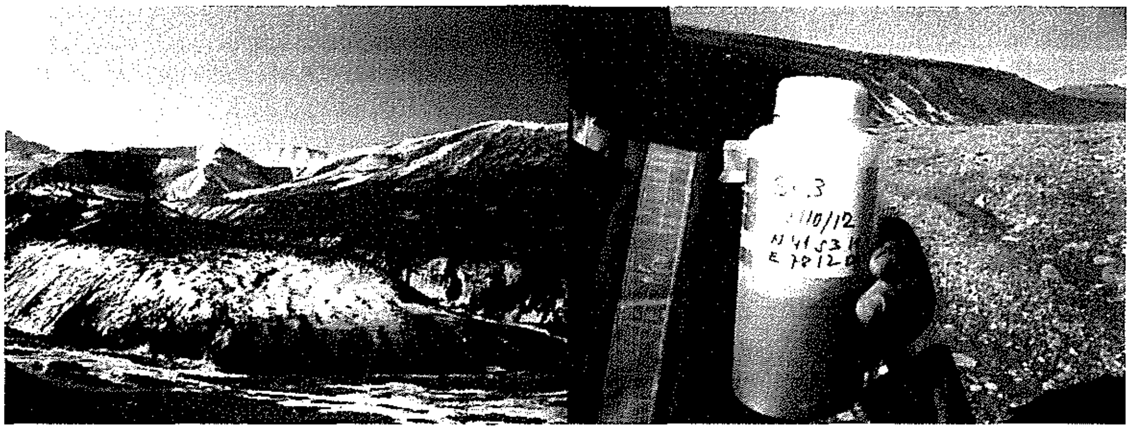

Fig. 2: SK 3 - Sampling of the sediments in the Lysyi Creek – Waste rock seepage - below the Lysyi glacier, with GPS recording (N 41^{0} 53' 10.0", E 78^{0} 12' 00.2")

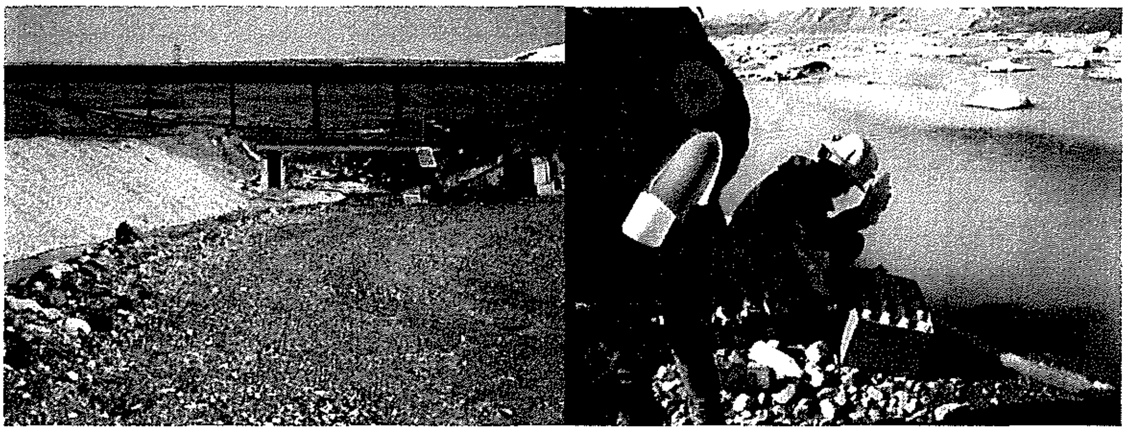

Fig. 3: SK 4 - Sampling at the Kumtor river Flume, jointly with environmental team of the Kumtor company

Fig. 4: SK 4 - Sampling at the Kumtor tailings pond

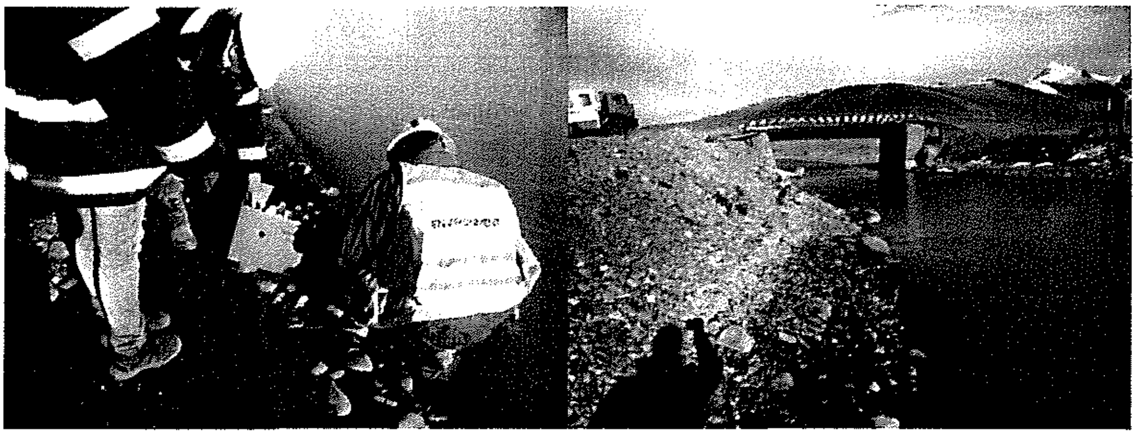

Fig. 5: SK 9 - Sampling point at the end mixing zone (EMZ) of the Kumtor river

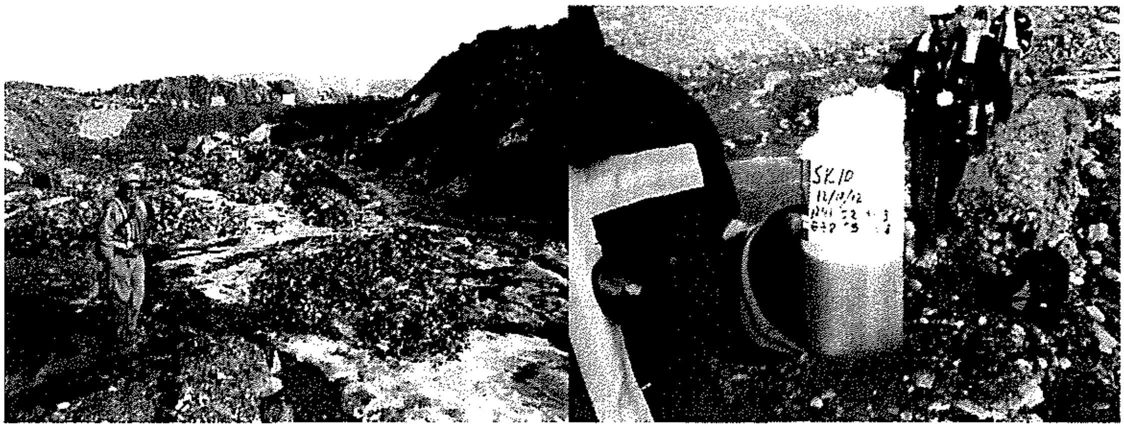

Fig.6: SK 10 - Sampling at Chon Sarytor river - below waste dumps and Central Open Pit

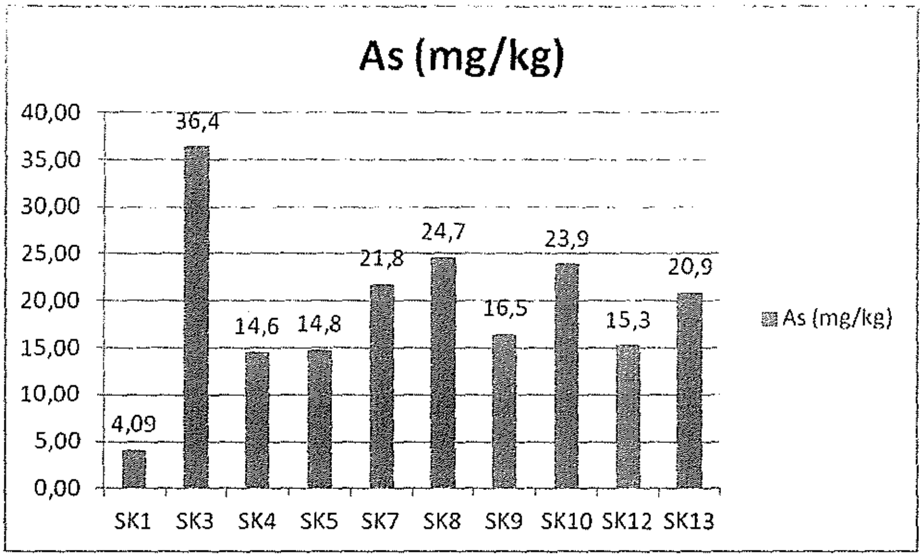

Fig. 7: Comparison of As levels in the Kumtor sediment samples

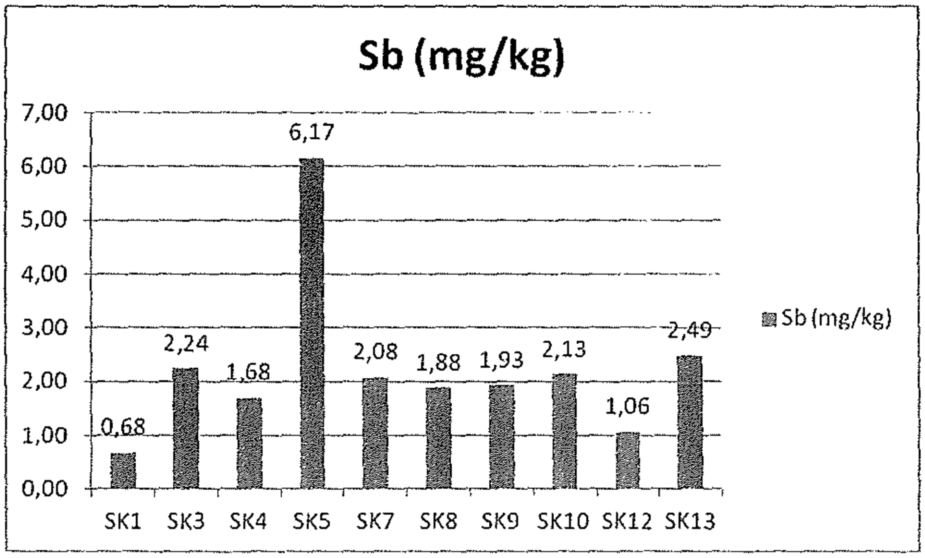

Fig. 8: Comparison of Sb levels in the Kumtor sediment samples

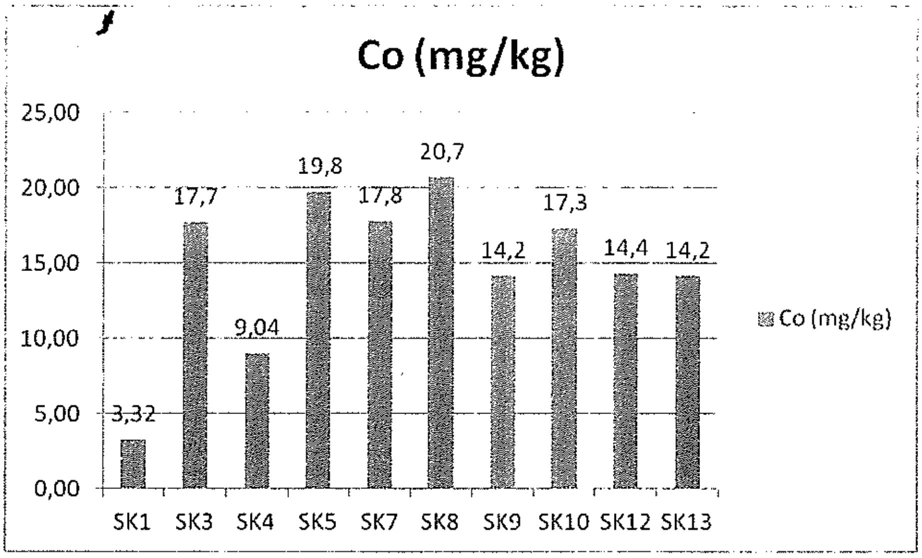

Fig. 9: Comparison of Co levels in the Kumtor sediment samples

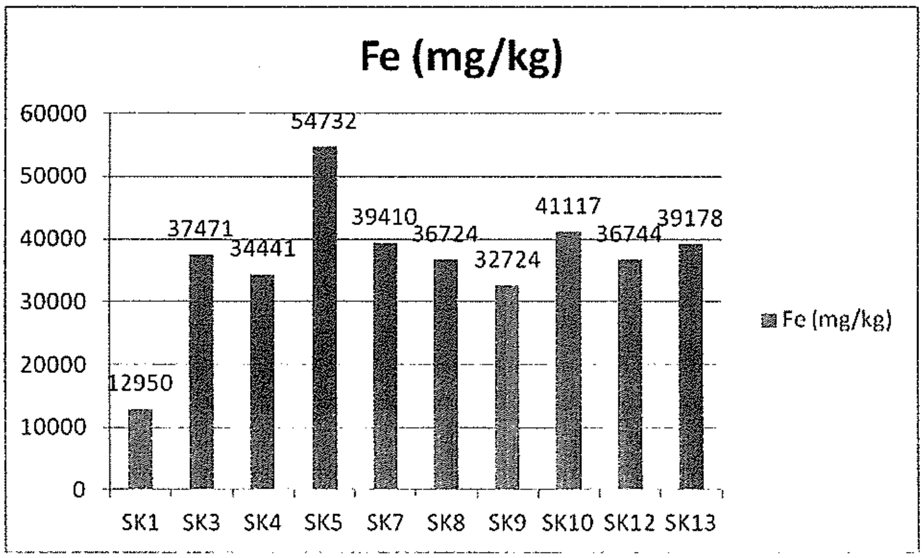

Fig. 10: Comparison of Fe levels in the Kumtor sediment samples

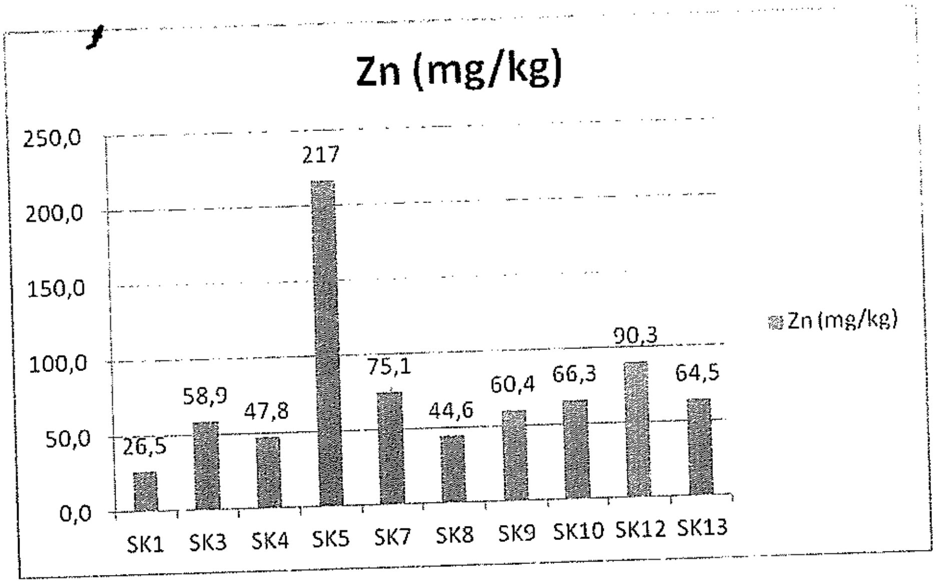

Fig. 11: Comparison of Zn levels in the Kumtor sediment samples

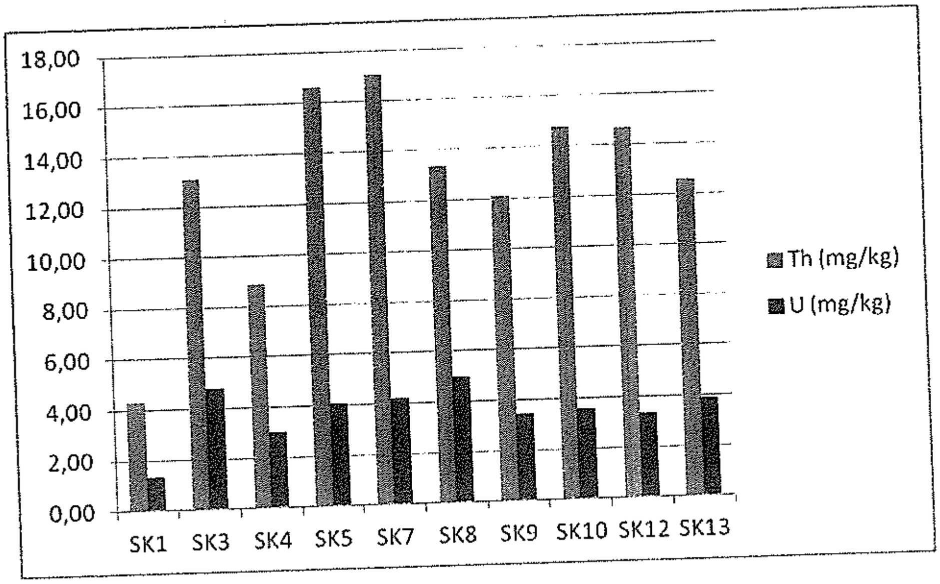

Fig. 12: Comparison of Th and U levels in the Kumtor sediment samples

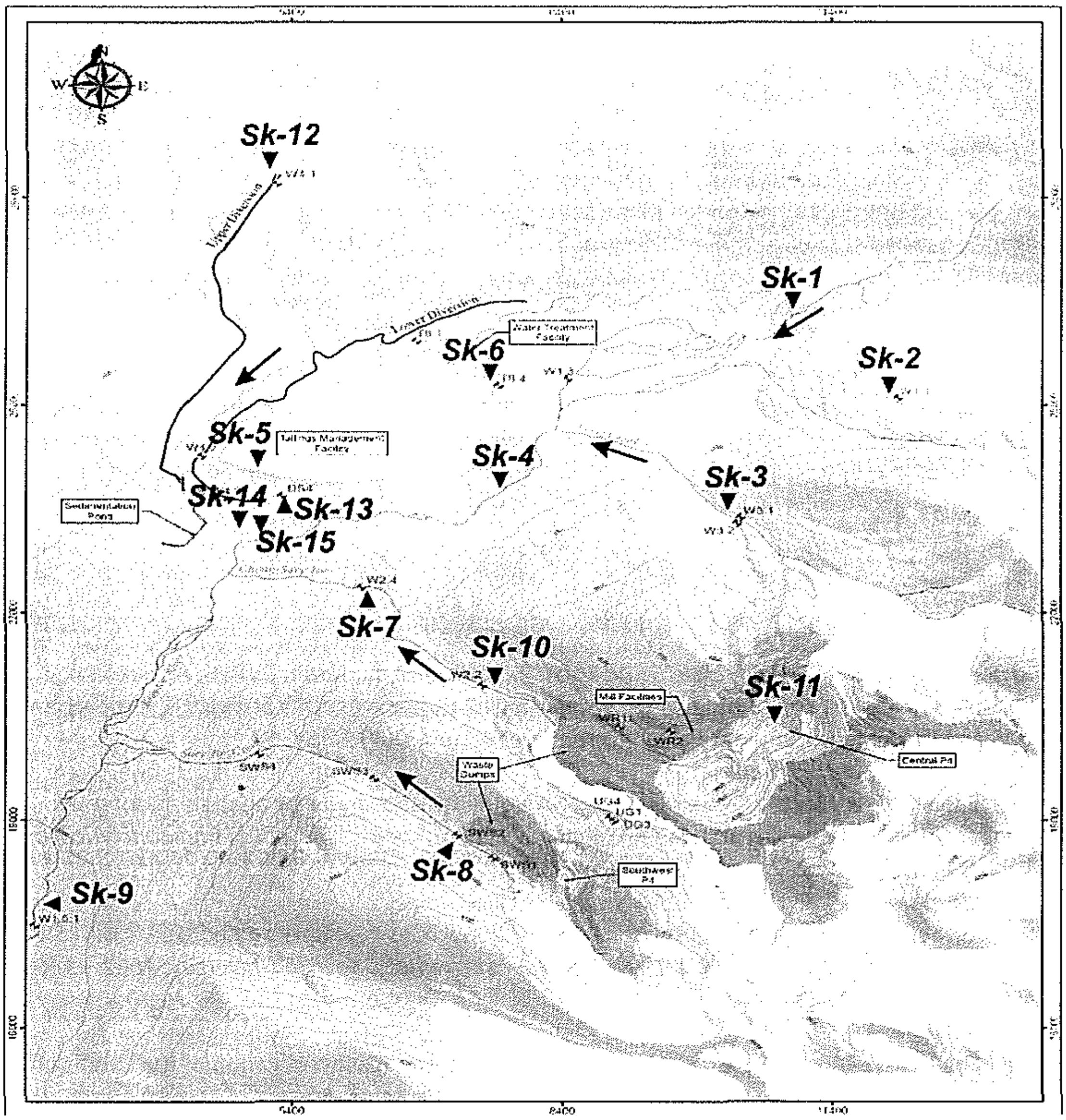

Fig. 13: Sampling points of river and stream sediments, soil (SK14 and SK15) and water samples at the Kumtor operating area

Concluding remarks

In this report, the analyses of river and stream sediments as indicators for environmental pollution from the Kumtor industrial area are provided. The total concentrations of 36 chemical elements were determined in sediments at 10 representatively sampling locations. The results obtained showed a tendency of increasing element levels at the sampling points affected by the Kumtor milling and ore processing operations over an extended period of time. It should be noted that the concentrations levels of the analyzed elements in the environment were within the limits typical for such industrial operations [Milačič et al., 2010] and were below the values, which indicate an environmental pollution or contamination situation.

The obtained results reveal the situation present only at this sampling campaign carried out under climatologically specific conditions and cannot be used for direct comparison with the data obtained at other times and under different conditions. Also, a direct comparison of analytical results obtained by different sample preparation techniques and distinct analytical methods should be avoided without considering careful evaluation of the methods used.

References

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Kayukov, P.G., Fedorov, G.V., Distribution of natural radionuclides in waters and bottom sediments in Kazakhstan, *Environmental Radioactivity in Central Asia, Almaty, Kazakh University, 2012 – 140 p., ISBN 978-601-247-673-6*

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SLOVENSKA AKREDITACIJA

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PRILOGA K AKREDITACIJSKI LISTINI
Annex to the accreditation certificate

LP-090

1 AKREDITIRANI ORGAN / *Accredited body*

Institut Jožef Stefan
Jamova cesta 39, 1000 Ljubljana
Odsek za znanosti o okolju: Skupina za radiokemijo

2 STANDARD

SIST EN ISO/IEC 17025:2005

3 OBSEG AKREDITACIJE / *Scope of accreditation*

V okviru te akreditacijske listine Slovenska akreditacija priznava akreditiranemu organu usposobljenost za opravljanje naslednjih dejavnosti: / *SA hereby acknowledges the accredited body as being competent for performing the following activities:*

3.1 Skrajšan opis obsega akreditacije / *A short description of the scope*

Področja preskušanja glede na vrsto preskušanja / *Testing fields with reference to the type of test:*
- kemija / *chemistry*

Področja preskušanja glede na vrsto preskušanca / *Testing fields with reference to the type of test item:*
- okolje in vzorci iz okolja / *environment and samples from the environment*
- živila / *foodstuffs*
- kmetijski proizvodi (krma) / *agricultural products (fodders)*

j bioloski vzorci / *biological samples*

3.2 Podroben opis obsega akreditacija / *Detailed scope of accreditation*

3.2.1 Odsek za znanosti o okolju, Skupina za radiokemijo, Jamova cesta 39, 1000 Ljubljana

Tabela 1 / *Table 1*

Tip obsega: **fiksni** / *Type of scope: **fixed***
Mesto izvajanja: **v laboratoriju** / *Site: **in the laboratory***
Področja preskušanja glede na vrsto preskušanja: **radiokemija, sevanje** / *Testing fields with reference to the type of test: **radiochemistry, radiation***
Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item: **environment and samples from the environment; foodstuffs; agricultural products (fodders); biological samples***

Št. *No.*	Oznaka standarda ali nestandardne preskusne metode *Reference to standard or non-standard testing method*	Naslov standarda ali nestandardne preskusne metode in morebitne navezave na druge standarde ali metode *Title of standard or non-standard testing method and eventual relations to other standards or methods*	Območje preskušanja; Negotovost rezultata preskušanja (kjer je to pomembno) *Range of testing; Uncertainty of the result of testing (where relevant)*	Materiali; proizvodi *Materials; products*
1.	SDN-O2-STC (01) interna metoda, 5. izdaja *in-house method, version 5*	Določanje stroncija z beta štetjem *Determination of strontium by beta counting*	$^{89/90}Sr$ minimalna aktivnost *minimal detectable activity* (MDA): 0,01Bq zemlja, sedimenti *soil, sediment (on dry matter basis)* 1,9 E-01 Bq/kg *(on dry matter basis)* voda *water* 1,2E-02 Bq/kg 1,2E-01 Bq/kg *(on dry mater basis)* Mleko, milk 3,6E-03 Bq/kg Hrana, krma, foodstuffs, feedstuffs 1,2E-02 Bq/kg, (on dry mater basis) Zračni filtri, aerosol filters 1,2E-01Bq/kg, (filter and air particles) ^{90}Sr Minimalna aktivnost Minimal detectable activity (MDA) 0,03 Bq Zemlja, sedimenti,Soil, sediment 7,5E-01 Bq/kg, (on dry matter basis) Voda, water 4,5 E-02 Bq/kg 4,5E-01Bq/kg, (on dry matter basis) Mleko, milk 1,5E-02 Bq/kg Hrana, krma, foodstuffs feedstuffs 4,5E-02 Bq/kg, (on dry matter basis) Zračni filtri, aerosol filters 4,5E-01 Bq/kg, (filter and air particles) ^{89}Sr Minimalna aktivnost, minimal detactable activity (MDA) 0,16 Bq Zemlja, sedimenti: Soil, sediment 2,5E+00 Bq/kg, (on dry matter basis) Voda,water 1,5E-01 Bq/kg 1,5E+00 Bq/kg (on dry matter basis) Mleko, milk 5,2E-02 Bq/kg Hrana, krma, foodstuffs, feedstuffs 1,5E-01 Bq/kg, (on dry matter basis) Zračni filtri, aerosol filters 1,5E+00 Bq/kg (filter and air particles)	vzorci iz okolja, živila in krma *samples from the environment, foodstuffs and feedstuffs*

Tip obsega: **fiksni** / *Type of scope:* ***fixed***
Mesto izvajanja: **v laboratoriju** / *Site:* ***in the laboratory***
Področja preskušanja glede na vrsto preskušanja: **radiokemija, sevanje** / *Testing fields with reference to the type of test:* ***radiochemistry, radiation***
Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item:* ***environment and samples from the environment; foodstuffs; agricultural products (fodders); biological samples***

Št. *No.*	**Oznaka standarda** ali nestandardne preskusne metode ***Reference** to standard or non-standard testing method*	**Naslov standarda** ali nestandardne preskusne metode in morebitne navezave na druge standarde ali metode ***Title** of standard or non-standard testing method and eventual relations to other standards or methods*	**Območje** preskušanja; **Negotovost** rezultata preskušanja (kjer je to pomembno) ***Range** of testing; **Uncertainty** of the result of testing (where relevant)*	**Materiali; proizvodi** ***Materials; products***
2.	SDN-O2-STC (02) interna metoda, 4. izdaja *in-house method, version 4*	Določanje tritija s tekočinskim scintilacijskim štetjem *Determination of tritium activity by liquid scintillation counting*	direktna metoda, meja zaznavnosti *direct method, limit of detection:* 3,47E-03 Bq/g elektroliza, meja zaznavnosti *electrolytical enrichment,limit of detection:* 3,37E-04 Bq/g	voda, urin *water, urine* voda *water*
3.	SDN-O2-STC (03) interna metoda, 5. izdaja *in-house method, version 5*	Določanje ^{14}C v bazični raztopini *Determination of ^{14}C in alkaline solution*	meja zaznavnosti *limit of detection:* 1,73E-03 Bq /g	voda, urin *water, urine*

3.2.2 Odsek za znanosti o okolju, Skupina za radiokemijo, Brinje 40, 1231 Podgorica

Tabela 2 / *Table 2*

Tip obsega: **fiksni** / *Type of scope:* ***fixed***
Mesto izvajanja: **v laboratoriju** / *Site:* ***in the laboratory***
Področja preskušanja glede na vrsto preskušanja: **kemija** / *Testing fields with reference to the type of test:* ***chemistry***
Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item:* ***environment and samples from the environment; foodstuffs; agricultural products (fodders); biological samples***

Št. *No.*	**Oznaka standarda** ali nestandardne preskusne metode ***Reference** to standard or non-standard testing method*	**Naslov standarda** ali nestandardne preskusne metode in morebitne navezave na druge standarde ali metode ***Title** of standard or non-standard testing method and eventual relations to other standards or methods*	**Območje** preskušanja; **Negotovost** rezultata preskušanja (kjer je to pomembno) ***Range** of testing; **Uncertainty** of the result of testing (where relevant)*	**Materiali; proizvodi** ***Materials; products***
4.	SDN-O2-K0 (01) interna metoda, 5. izdaja *in-house method, version 5*	Določanje elementne sestave v vzorcih iz okolja s k_0-INAA *Determining elemental composition of environmental samples using k_0-INAA*	meja zaznavnosti *limit of detection (LD):* Sediment / Biološki vz. *Sediment / Biological s.* mg/kg / mg/kg Ag 0,30 0,04 As 0,5 0,09 Au 0,0017 0,0016 Br 0,6 0,1 Ca 437 131 Ce 0,3 0,1 Co 0,02 0,005 Cr 0,5 0,0003 Cs 0,05 0,008 Eu 0,01 0,002 Fe 29 5 Hf 0,04 0,007 Hg 0,17 0,019 K 761 24 La 0,1 0,03 Mo 0,9 0,3 Na 5 0,2	tla, sedimenti, blata čistilnih naprav, biološki vzorci *soil, sediments, sewage sludge, biological samples*

Tip obsega: **fiksni** / *Type of scope: **fixed***
Mesto izvajanja: v laboratoriju / *Site: in the laboratory*
Področja preskušanja glede na vrsto preskušanja: **kemija** / *Testing fields with reference to the type of test: **chemistry***
Področja preskušanja glede na vrsto preskušanca: **okolje in vzorci iz okolja; živila; kmetijski proizvodi (krma); biološki vzorci** / *Testing fields with reference to the type of test item: **environment and samples from the environment; foodstuffs; agricultural products (fodders); biological samples***

Št. *No.*	**Oznaka standarda** ali nestandardne preskusne metode *Reference to standard or non-standard testing method*	**Naslov standarda** ali nestandardne preskusne metode in morebitne navezave na druge standarde ali metode *Title of standard or non-standard testing method and eventual relations to other standards or methods*	**Območje preskušanja; Negotovost** rezultata preskušanja (kjer je to pomembno) *Range of testing; Uncertainty of the result of testing (where relevant)*	**Materiali; proizvodi** *Materials; products*
			Nd 0,7 0,2 Rb 1,3 0,2 Sb 0,04 0,008 Sc 0,002 0,0004 Se 0,27 0,074 Sm 0,01 0,01 Sr 23 2,9 Ta 0,01 0,003 Tb 0,02 0,003 Th 0,04 0,007 U 0,10 0,03 Yb 0,04 0,009 Zn 0,8 0,2 Zr 30 4 območje merjenih koncentracij elementov je med LD in 1 kg/kg (100 %) *for all samples maximum concentration is 1 kg/kg (100 %)*	

Opombe / *Notes*:

- V vseh točkah podrobnega obsega akreditacije, pri katerih v rubriki "**Območje** preskušanja; **Negotovost** rezultata preskušanja" ni navedenih podatkov, veljajo določila posameznih standardov oziroma nestandardnih preskusnih metod, ki se na to nanašajo.
  *In all columns of the scope of accreditation where the cells under "**Range** of measurement, testing; **Uncertainty** of the result of testing" are empty, the provisions of the relevant standards or non-standard testing methods should apply.*
- V točkah podrobnega obsega akreditacije, pri katerih v rubriki "**Oznaka** standarda" ni navedena letnica izdaje standarda, se sklic nanaša na zadnjo (veljavno) izdajo standarda, kar jamči interni sistem sledenja in prilagajanja laboratorija spremembam.
  *In those columns of the scope of accreditation where the cells under "**Reference**" do not specify the year of issue of the standard, the latest (valid) standard should apply. This is assured by internal laboratory system of follow-up and adaptation to changes.*
- V točkah podrobnega obsega akreditacije, pri katerih sta v rubriki "**Oznaka** standarda" navedeni dve ali več oznak standardov, se sklic v celoti nanaša na identične standarde.
  *In those columns of the scope of accreditation where the cells under "**Reference**" specify two or more codes of standards, the complete citation to identical standards should apply.*

Direktor / *Director*
dr. Boštjan Godec

"Jožef Stefan" Institute, Ljubljana, Slovenia

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Analytical Report

No. K0 012/12

SLOVENSKA AKREDITACIJA
SIST EN ISO/IEC 17025
LP-090
The results marked by #, relate to non-accredited activity

Customer	TECHNOMEDICA d.o.o.
Address	Miklošičeva cesta 28, 1000 Ljubljana
Contact person	Žiga Krušič, M. D.
Analysis performed	k_0-INAA

Order / Contract	Naročilnica št. 01/12 from 5.11.2012
Samples delivered by	Prof. Dr. Peter Stegnar
Date of sample receipt	15.10.2012
Samples taken by	Dr. Radojko Jaćimović
Sample description	Sediment (10 samples)

ANALYTICAL METHOD USED

Short description of the method used, No. of standard working procedure at the O-2:
SDN-O2-K0(01), Issue 05, Valid from Nov. 2011; Title: "Determining elemental composition of environmental samples using k_0-INAA"

The samples were received in polyethylene boxes containing sediment material mixed with water. The water was removed and residue was dried at 45 °C until air dry matter was attained (6 days). Then, the samples were sieved through 1 mm sieve and only particles < 1 mm were milled in an agate mill (Fritsch, Pulverisette 7, 6 min at 3600 rpm). After milling the particle size was < 0.22 mm. In order to confirm it, one sample of ten was randomly chosen and sieved through 0.22 mm sieve. The result shows that whole milled sample passed through 0.22 mm sieve.

For k_0-INAA an aliquot of milled sediment sample < 0.22 mm (varied from 0.17 to 0.18 g) was sealed into a pure polyethylene ampoule (SPRONK system, Lexmond, The Netherlands). A sample and standard Al-0.1%Au (IRMM-530R) were stacked together, fixed in the polyethylene vial in sandwich form and irradiated for 19 hours in the carousel facility (CF) of the TRIGA reactor with a thermal neutron flux of 1.1×10^{12} $cm^{-2} \cdot s^{-1}$.

After irradiation the aliquot was measured after 5 and 8-12 days cooling time on absolutely calibrated HPGe detector with 45 % relative efficiency. For peak area evaluation, the HyperLab 2002 program was used. The values f = 28.63 (thermal to epithermal flux ratio) and $\alpha = -0.0011$ (epithermal flux deviation from the ideal 1/E distribution) were used to calculate element concentrations. For elemental concentrations and effective solid angle calculations the software package Kayzero for Windows was applied.

For QA/QC purposes for k_0-INAA the BCR-320R Channel Sediment was used.

RESULTS OF ANALYSIS

The preparation step is presented in Table 1. Only the sediment samples with size particle < 1 mm were milled and used for analysis by k_0-INAA.

Table 1. Preparation of sediment samples.

Sample code	Air dry mass (g)	Mass of particles > 1 mm (g)	% of particles > 1 mm	Mass of particles < 1 mm (g)	% of particles < 1 mm
SK1	882.5	561.1	64	321.4	36
SK12	239.2	80.7	34	158.5	66
SK3	542.7	36.3	7	506.4	93
SK4	474.4	0.3	0	474.1	100
SK7	436.3	23.0	5	413.3	95
SK8	538.8	177.1	33	361.7	67
SK9	346.7	0	0	346.7	100
SK10	478.9	13.5	3	465.4	97
SK5	286.0	0	0	286.0	100
SK13	263.9	78.1	30	185.8	70

Results obtained by k_0-INAA are given in Tables 2-5 on air dry matter basis for milled samples (particle size < 0.22 mm).

Table 2. Results obtained by k_0-INAA in sediment samples.

Sample code	SK1			Sample code	SK3			Sample code	SK4		
Lab. Sample code	J9856			Lab. Sample code	J9858			Lab. Sample code	J9859		
El.	**Content* mg/kg**			El.	**Content* mg/kg**			El.	**Content* mg/kg**		
Ag	< 0.4			Ag	< 0.7			Ag	< 0.6		
As	4.09	±	0.25	As	36.4	±	1.3	As	14.6	±	0.6
Au	0.0017	±	0.0002	Au	0.117	±	0.004	Au	0.218	±	0.008
Ba#	1109	±	41	Ba#	1294	±	45	Ba#	1256	±	44
Br#	0.27	±	0.02	Br	0.62	±	0.04	Br#	0.43	±	0.05
Ca	16275	±	651	Ca	18069	±	835	Ca	22826	±	935
Cd#	< 0.92			Cd#	< 1.98			Cd#	< 1.41		
Ce	34.5	±	1.2	Ce	98.5	±	3.5	Ce	71.8	±	2.7
Co	3.32	±	0.12	Co	17.7	±	0.6	Co	9.04	±	0.32
Cr	13.7	±	0.5	Cr	67.9	±	2.4	Cr	35.7	±	1.3
Cs	1.66	±	0.06	Cs	2.74	±	0.11	Cs	2.45	±	0.09
Eu	0.66	±	0.05	Eu	1.51	±	0.07	Eu	1.40	±	0.06
Fe	12950	±	454	Fe	37471	±	1313	Fe	34441	±	1206
Ga#	< 25.0			Ga#	40.2	±	3.2	Ga#	31.5	±	3.7
Hf	3.51	±	0.12	Hf	5.65	±	0.20	Hf	8.82	±	0.31
Hg	< 0.2			Hg	< 0.4			Hg	< 0.2		
K	19240	±	877	K	31160	±	1232	K	23290	±	1023
La	23.9	±	0.8	La	50.5	±	1.8	La	36.1	±	1.3
Mo#	0.49	±	0.06	Mo	5.58	±	0.28	Mo	2.33	±	0.35
Na	35230	±	1234	Na	11270	±	395	Na	24570	±	861
Nd	12.4	±	0.6	Nd	47.0	±	1.8	Nd	33.1	±	1.2
Rb	67.9	±	2.4	Rb	130	±	5	Rb	88.1	±	3.1
Sb	0.68	±	0.04	Sb	2.24	±	0.08	Sb	1.68	±	0.06
Sc	3.99	±	0.15	Sc	12.4	±	0.4	Sc	7.86	±	0.28
Se	< 0.6			Se	0.97	±	0.15	Se	< 0.6		
Sm	2.03	±	0.07	Sm	7.89	±	0.28	Sm	5.79	±	0.20
Sr	778	±	28	Sr	115	±	10	Sr	525	±	19
Ta	0.41	±	0.02	Ta	1.15	±	0.04	Ta	1.11	±	0.04
Tb	0.27	±	0.01	Tb	0.99	±	0.04	Tb	0.72	±	0.03
Te#	< 0.8			Te#	< 1.4			Te#	< 0.9		
Th	4.29	±	0.15	Th	13.1	±	0.5	Th	8.87	±	0.31
U	1.31	±	0.05	U	4.81	±	0.18	U	3.03	±	0.11
W#	0.94	±	0.09	W#	8.14	±	0.41	W#	6.75	±	0.34
Yb	0.96	±	0.03	Yb	2.93	±	0.10	Yb	2.29	±	0.08
Zn	26.5	±	1.1	Zn	58.9	±	2.4	Zn	47.8	±	1.9
Zr	124	±	7	Zr	235	±	11	Zr	358	±	14

NOTE:
– The results relate to non-accredited activity;
* – Uncertainty is given with a coverage factor k=1;
< – Limit of detection (LD) of the method used

Table 3. Results obtained by k_0-INAA in sediment samples.

Sample code	SK5	Sample code	SK7	Sample code	SK8
Lab. Sample code	J9860	Lab. Sample code	J9861	Lab. Sample code	J9862
El.	**Content* mg/kg**	El.	**Content* mg/kg**	El.	**Content* mg/kg**
Ag	< 0.9	Ag	< 0.7	Ag	< 0.6
As	14.8 ± 0.6	As	21.8 ± 0.8	As	24.7 ± 0.9
Au	0.61 ± 0.02	Au	0.065 ± 0.002	Au	0.056 ± 0.002
Ba#	1860 ± 65	Ba#	877 ± 31	Ba#	781 ± 28
Br	< 0.64	Br	0.98 ± 0.15	Br	< 1.11 ±
Ca	47692 ± 1831	Ca	49722 ± 1880	Ca	20705 ± 871
Cd#	< 2.42	Cd#	< 3.19	Cd#	< 3.41
Ce	110 ± 4	Ce	120 ± 4	Ce	96.7 ± 3.4
Co	19.8 ± 0.7	Co	17.8 ± 0.6	Co	20.7 ± 0.7
Cr	78.4 ± 2.8	Cr	74.3 ± 2.6	Cr	66.1 ± 2.3
Cs	2.31 ± 0.08	Cs	5.55 ± 0.20	Cs	2.73 ± 0.12
Eu	1.82 ± 0.08	Eu	1.69 ± 0.07	Eu	1.58 ± 0.07
Fe	54732 ± 1916	Fe	39410 ± 1380	Fe	36724 ± 1286
Ga#	40.6 ± 3.3	Ga#	47.1 ± 3.4	Ga#	42.4 ± 3.8
Hf	3.83 ± 0.13	Hf	7.81 ± 0.27	Hf	6.88 ± 0.24
Hg	< 0.3	Hg	< 0.3	Hg	< 0.2
K	34970 ± 1374	K	23340 ± 1002	K	29710 ± 1228
La	59.7 ± 2.1	La	62.0 ± 2.2	La	50.9 ± 1.8
Mo	8.79 ± 0.39	Mo	2.83 ± 0.31	Mo	6.75 ± 0.58
Na	14170 ± 497	Na	10160 ± 356	Na	12280 ± 431
Nd	50.4 ± 1.8	Nd	54.3 ± 2.0	Nd	44.5 ± 1.7
Rb	147 ± 5	Rb	115 ± 4	Rb	121 ± 4
Sb	6.17 ± 0.22	Sb	2.08 ± 0.08	Sb	1.88 ± 0.07
Sc	15.3 ± 0.5	Sc	13.0 ± 0.5	Sc	12.8 ± 0.5
Se	< 0.6	Se	< 0.9	Se	1.30 ± 0.05
Sm	8.13 ± 0.29	Sm	9.19 ± 0.32	Sm	8.15 ± 0.29
Sr	1547 ± 55	Sr	368 ± 15	Sr	120 ± 8
Ta	0.94 ± 0.03	Ta	1.10 ± 0.04	Ta	1.10 ± 0.04
Tb	1.00 ± 0.04	Tb	1.12 ± 0.04	Tb	1.11 ± 0.04
Te#	5.11 ± 0.25	Te#	< 1.5	Te#	< 1.4
Th	16.6 ± 0.6	Th	17.1 ± 0.6	Th	13.3 ± 0.5
U	4.06 ± 0.14	U	4.23 ± 0.15	U	5.00 ± 0.18
W#	200 ± 7	W#	10.2 ± 0.5	W#	8.72 ± 0.40
Yb	2.78 ± 0.10	Yb	3.22 ± 0.11	Yb	3.60 ± 0.13
Zn	217 ± 8	Zn	75.1 ± 3.0	Zn	44.6 ± 2.0
Zr	138 ± 7	Zr	260 ± 12	Zr	241 ± 11

NOTE:
– The results relate to non-accredited activity;
* – Uncertainty is given with a coverage factor k=1;
< – Limit of detection (LD) of the method used

Table 4. Results obtained by k_0-INAA in sediment samples.

Sample code	SK9	Sample code	SK10	Sample code	SK12
Lab. Sample code	J9863	Lab. Sample code	J9864	Lab. Sample code	J9857
El.	Conc.	El.	Conc.	El.	Conc.
Ag	< 0.6	Ag	< 0.4	Ag	< 0.7
As	16.5 ± 0.6	As	23.9 ± 0.9	As	15.3 ± 0.6
Au	0.243 ± 0.009	Au	0.060 ± 0.003	Au	< 0.0020
Ba#	993 ± 35	Ba#	1053 ± 37	Ba#	480 ± 17
Br	< 0.96	Br	1.53 ± 0.17	Br	2.06 ± 0.08
Ca	37693 ± 1560	Ca	18837 ± 1019	Ca	31088 ± 1240
Cd#	< 4.48	Cd#	< 5.36	Cd#	< 1.87
Ce	87.3 ± 3.1	Ce	116 ± 4	Ce	96.9 ± 3.4
Co	14.2 ± 0.5	Co	17.3 ± 0.6	Co	14.4 ± 0.5
Cr	58.1 ± 2.0	Cr	67.6 ± 2.4	Cr	71.3 ± 2.5
Cs	4.16 ± 0.15	Cs	3.22 ± 0.11	Cs	8.32 ± 0.30
Eu	1.46 ± 0.06	Eu	1.77 ± 0.08	Eu	1.36 ± 0.08
Fe	32724 ± 1146	Fe	41117 ± 1440	Fe	36744 ± 1288
Ga#	34.3 ± 3.6	Ga#	50.8 ± 4.3	Ga#	27.6 ± 2.9
Hf	5.70 ± 0.20	Hf	5.79 ± 0.20	Hf	6.83 ± 0.24
Hg	< 0.3	Hg	< 0.4	Hg	< 0.4
K	23420 ± 1050	K	27380 ± 1173	K	26850 ± 1090
La	44.3 ± 1.6	La	61.0 ± 2.1	La	47.5 ± 1.7
Mo	3.00 ± 0.29	Mo	3.40 ± 0.55	Mo	< 0.9
Na	17540 ± 616	Na	13490 ± 474	Na	15620 ± 548
Nd	40.4 ± 1.6	Nd	51.5 ± 2.0	Nd	43.6 ± 1.7
Rb	104 ± 4	Rb	116 ± 4	Rb	148 ± 5
Sb	1.93 ± 0.07	Sb	2.13 ± 0.08	Sb	1.06 ± 0.04
Sc	11.0 ± 0.4	Sc	13.6 ± 0.5	Sc	13.9 ± 0.5
Se	< 0.9	Se	< 1.0	Se	< 1.1
Sm	6.83 ± 0.24	Sm	8.99 ± 0.32	Sm	7.54 ± 0.26
Sr	349 ± 15	Sr	166 ± 10	Sr	203 ± 10
Ta	1.01 ± 0.04	Ta	1.09 ± 0.04	Ta	1.38 ± 0.05
Tb	0.87 ± 0.03	Tb	1.08 ± 0.04	Tb	1.00 ± 0.04
Te#	< 1.7	Te#	< 1.8	Te#	< 1.5
Th	12.1 ± 0.4	Th	14.8 ± 0.5	Th	14.7 ± 0.5
U	3.44 ± 0.14	U	3.61 ± 0.13	U	3.35 ± 0.12
W#	6.11 ± 0.41	W#	9.07 ± 0.41	W#	2.50 ± 0.30
Yb	2.65 ± 0.09	Yb	3.01 ± 0.11	Yb	3.19 ± 0.11
Zn	60.4 ± 2.7	Zn	66.3 ± 2.7	Zn	90.3 ± 3.5
Zr	231 ± 12	Zr	218 ± 11	Zr	270 ± 12

NOTE:
– The results relate to non-accredited activity;
* – Uncertainty is given with a coverage factor k=1;
< – Limit of detection (LD) of the method used

Table 5. Results obtained by k_0-INAA in sediment samples.

Sample code	SK13		
Lab. Sample code	J9865		
El.	**Content* mg/kg**		
Ag	< 0.6		
As	20.9	±	0.8
Au	0.043	±	0.002
Ba#	1275	±	45
Br	3.01	±	0.23
Ca	37905	±	1538
Cd#	< 5.35		
Ce	85.8	±	3.0
Co	14.2	±	0.5
Cr	64.4	±	2.3
Cs	4.47	±	0.16
Eu	1.41	±	0.06
Fe	39178	±	1376
Ga#	52.3	±	4.0
Hf	5.20	±	0.18
Hg	< 0.3		
K	25530	±	1109
La	43.3	±	1.5
Mo	3.50	±	0.30
Na	14110	±	495
Nd	37.0	±	1.5
Rb	121	±	4
Sb	2.49	±	0.09
Sc	11.7	±	0.4
Se	< 0.7		
Sm	6.74	±	0.24
Sr	232	±	11
Ta	0.91	±	0.03
Tb	0.84	±	0.03
Te#	< 1.7		
Th	12.6	±	0.4
U	3.86	±	0.14
W#	12.2	±	0.5
Yb	2.66	±	0.10
Zn	64.5	±	2.7
Zr	196	±	11

NOTE:
– The results relate to non-accredited activity;
* – Uncertainty is given with a coverage factor k=1;
< – Limit of detection (LD) of the method used

Analysed by:

Dr. Radojko Jaćimović

Report approved by:

Prof. Dr. Milena Horvat

Head, Department of Environmental Sciences

Date:

28.11.2012

Results reviewed by:

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