DETERMINATION OF TRACE ELEMENTS IN FLY COAL ASH (ENO, EOP, ECH REFERENCE MATERIALS)

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Abstract: Concentration of 32 elements in three reference materials is determined

INTRODUCTION

This work presents the work regarding an intercomparison organised by the Institute of Radioecology and Applied Nuclear Techniques from Košice - Czechoslovakia. By using the instrumental neutron activation analysis method the materials of fly-ashes character from coal-fired power plants was analysed. A number of 34 laboratories from 11 countries have participated at this intercomparison.

EXPERIMENTAL

The concentration of Ba, Ca, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Lu, Nd, Rb, Sb, Sc, Sm, Sr, Ta, Tb, Th, U, Yb, Zn, Zr was determined after a long irradiation (50 hours) of the samples in a thermal neutron flux of $1.1 \times 10^{11} n/cm^2.s$. The samples (~ 100 mg in weight) and Soil-5, SL-1, GSP-1 as standards, were measured 2-5 hours after a decay times of 10-30 days. Concentration of Al, As, Eu, K, Mn, Na, Sm, Ti, V, Dy was obtained after a short irradiation of 1 min. in a $10^{12} n/cm^2.s$. flux. W-1 and arsenic oxyde were used as standards. After 6 min.- 24 hours cooling time the samples and standards were measured for 100 - 1800 s., by using a Ge(Li) detector with 2 keV resolution.

RESULTS AND DISCUSSION

The content of elements determined in this way are presented in table 1, 2, 3, for ENO, EOP and ECH respectively. A, B class of results denotes the certified values with satisfactory or acceptable degree of confidence respectively. Non recommended values are denoted by C. Our values are obtained as arithmetic average from four independent determinations. The standard deviation is given. Our results are in a good enough agreement with the certified values.

Only for Zn in ENO material our value was rejected from the overall mean calculated. As there can be observed, a large content of arsenium is present in the ENO material.

Our values for U concentration are systematically lower than the certified values. We have to check our method in analysing this element.

For Eu, Lu, Na, Sc in the EOP material our standard deviation is very small i.e. the four values obtained for each element being very close. Also as one can see our value for the Na concentration in the EOP material is higher than the certified value. We would suspect a small contamination in our measurements for this material.

TABLE 1 - ENO

Element	Concentration (ppm)	Confidence limits	Our results	Class of results
0	1	2	3	4
Al(%)	10.9	10.5 - 11.3	11.0 ± 0.7	A
As	1790	1680 - 1900	1643 ± 37	B
Ba	674	630 - 717	690 ± 78	A
Ca(%)	3.34	2.96 - 3.72	2.50 ± 0.05	A
Ce	98.7	93.2 - 104	92.5 ± 1.5	B
Co	26.1	24.9 - 27.4	27.7 ± 0.6	B
Cr	96.1	88.0 - 104	92.3 ± 0.8	A
Cs	118	110 - 126	122 ± 0.8	A
Dy	7.02		7.52 ± 0.27	C
Eu	1.76	1.41 - 2.11	1.77 ± 0.09	A
Fe(%)	7.46	7.23 - 7.70	7.94 ± 0.04	A
Hf	4.89	4.64 - 5.14	4.46 ± 0.28	B

0	1	2	3	4
K(%)	1.73	1.67 - 1.78	1.84 ± 0.06	A
La	42.9	39.7 - 46.1	49.1 ± 0.8	B
Lu	0.54	0.50 - 0.59	0.57 ± 0.02	B
Mn	634	607 - 661	628 ± 11	A
Na(%)	0.54	0.52 - 0.57	0.54 ± 0.01	A
Nd	56.1		40.5 ± 7.6	C
Rb	149	141 - 157	163 ± 15	A
Sb	5.72	3.68 - 7.76	4.49 ± 0.35	B
Sc	20.7	18.7 - 22.8	21.3 ± 0.1	A
Sm	9.45	8.33 - 10.6	8.85 ± 0.18	B
Sr	283	262 - 304	345 ± 17	A
Ta	1.22	1.17 - 1.27	1.20 ± 0.02	B
Tb	1.25		1.47 ± 0.19	C
Th	15.3	13.7 - 16.9	15.7 ± 0.2	B
Ti(%)	0.46	0.42 - 0.49	0.51 ± 0.08	A
U	7.29	6.13 - 8.45	3.96 ± 1.14	B
V	191	179 - 203	193 ± 7	A
Yb	3.49	3.14 - 3.84	2.85 ± 0.10	B
Zn	149	141 - 157	203 ± 13	A
Zr	222	170 - 274	246 ± 33	B

TABLE 2 - NOP

Element	Concentration (ppm)	Confidence limits	Our results	Class of results
0	1	2	3	4
Al(%)	15.8	14.5 - 17.0	16.6 ± 0.3	A
As	79.1	72.4 - 85.7	77.0 ± 3.7	A
Ba	1100	1050 - 1160	1202 ± 104	A
Ca(%)	1.68	1.43 - 1.93	1.83 ± 0.13	B
Ce	322	301 - 343	306 ± 1	A
Co	53.2	51.5 - 55.0	56.7 ± 0.5	A
Cr	183	172 - 195	188 ± 2	A
Cs	20.1	18.4 - 21.7	22.5 ± 0.3	A
Dy	10.8		9.8 ± 0.1	C
Eu	4.99	4.22 - 5.77	5.20 ± 0.00	A
Fe(%)	5.16	4.96 - 5.36	5.55 ± 0.04	A
Hf	17.7	16.5 - 18.9	16.3 ± 0.4	B
K(%)	0.64	0.62 - 0.67	0.60 ± 0.01	A
La	164	155 - 173	170 ± 2	A
Lu	0.51	0.47 - 0.55	0.52 ± 0.00	B
Mn	440	409 - 470	451 ± 16	A
Na(%)	0.37	0.32 - 0.41	0.55 ± 0.00	B
Nd	141		146 ± 15	C
Rb	69.0	59.3 - 78.6	94.7 ± 3.3	A
Sb	1.94	1.81 - 2.07	2.15 ± 0.36	B

0	1	2	3	4
Sc	36.7	33.0 - 40.3	40.8 ± 0.00	B
Sm	21.9	21.0 - 22.9	22.1 ± 0.1	B
Sr	574	535 - 614	475 ± 40	B
Ta	13.0	11.4 - 14.6	11.1 ± 0.2	B
Tb	1.93	1.34 - 2.52	2.62 ± 0.29	B
Th	23.9	21.3 - 26.5	25.6 ± 0.2	A
Ti(%)	3.68	3.32 - 4.04	3.69 ± 0.08	A
U	9.44	7.79 - 11.10	4.03 ± 0.40	B
V	553	525 - 582	568 ± 11	A
Yb	4.41	3.62 - 5.21	3.23 ± 0.21	B
Zn	219	191 - 248	337 ± 2	A
Zr	822	701 - 944	841 ± 89	B

TABLE 3 - NCH

Element	Concentration (ppm)	Confidence limits	Our results	Class of results
Al(%)	14.6	14.1 - 15.1	13.7 ± 0.5	A
As	56.9	52.6 - 61.2	53.3 ± 1.3	A
Ba	711	636 - 786	813 ± 11	A
Ca(%)	1.86	1.68 - 2.04	1.57 ± 0.15	B
Ce	183	174 - 193	169 ± 6	A
Co	49.8	47.1 - 52.5	52.4 ± 0.9	A
Cr	183	174 - 193	183 ± 2	A
Cs	23.0	21.2 - 24.7	25.2 ± 0.4	B
Dy	8.80		9.40 ± 0.16	C
Eu	2.95	2.85 - 3.04	3.18 ± 0.08	B
Fe(%)	5.57	5.40 - 5.74	6.00 ± 0.06	A
Hf	8.22	7.89 - 8.54	8.13 ± 0.40	B
K(%)	1.32	1.25 - 1.38	1.57 ± 0.16	A
La	84.4	79.9 - 88.9	96.0 ± 1.6	A
Lu	0.61	0.57 - 0.66	0.62 ± 0.01	B
Mn	381	358 - 404	394 ± 3	A
Na(%)	0.29	0.28 - 0.30	0.29 ± 0.01	B
Nd	85.7		82.0 ± 7.8	C
Rb	141	128 - 155	162 ± 7	A
Sb	3.73	3.55 - 3.91	3.92 ± 0.03	B
Sc	29.2	26.8 - 31.6	31.0 ± 0.2	A
Sm	13.6	13.0 - 14.1	12.8 ± 0.0	B
Sr	401	366 - 436	386 ± 13	A
Ta	4.37	4.09 - 4.65	4.04 ± 0.12	B
Tb	1.41	0.86 - 1.96	2.43 ± 0.26	B
Th	22.1	19.6 - 24.6	23.6 ± 0.4	A
Ti(%)	1.37	1.23 - 1.50	1.08 ± 0.06	A
U	7.36	6.18 - 8.54	3.71 ± 0.18	B
V	375	352 - 398	415 ± 6	A
Yb	3.62	2.93 - 4.32	3.01 ± 0.27	B

0	1	2	3	4
Zn	251	233 - 269	299 ± 36	B
Zr	361	291 - 430	497 + 77	A

URANIUM CONTENT MEASUREMENTS ON U-PHOSPHATE ORES

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Abstract: Concentration of uranium in S-17, S-18 and S-19 reference materials has been determined using the neutron activation analysis

INTRODUCTION

The analytical Quality Control Service of IAEA has organised this intercomparison on three Brasilian uranium phosphate ores containing a low, a medium and a large concentration of uranium (S-17, S-18 and S-19 respectively) in order to certify these materials as reference materials and also to provide an opportunity to the participating laboratories to compare their analytical methods and results with the others. A number of 24 laboratories from 19 countries have participated in this intercomparison. 19 % from the results used the neutron activation analysis method.

EXPERIMENTAL

Using the thermal neutron activation analysis four independent determinations for each of the three types of samples have been performed by our laboratory.

The samples with ~ 150 mg in weight and uranium acetate (aqueous solution) as a standard, have been irradiated 10 hours in a 1.1×10^{11} n/cm^2.s. flux and measured for 1-2 hours after 6-7 days decay time by using a Ge(Li) detector having 2 keV resolution.

RESULTS AND DISCUSSION

In the table the concentration of uranium in the IAEA/S-17, S-18 and S-19 phasphate uranium ores is presented and can be recommended with a satisfactory degree of confidence.

Material	Concentration (ppm)	Confidence intervals	Our results
S-17	370	360 - 390	384 ± 4
S-18	770	750 - 790	791 ± 6
S-19	2280	2210 - 2390	2388 ± 17

Our results are within of the confidence limits given by IAEA after the evaluation of all the received results.