RELIABILITY AND REPRODUCIBILITY OF LEACHING PROCEDURES TO ESTIMATE THE MODES OF OCCURRENCE OF TRACE ELEMENTS IN COAL

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INTRODUCTION

The 1990 amendments to the Clean Air Act have renewed interest in chemical forms or modes of occurrence of trace elements for potentially toxic trace elements in coal. Selective solvent leaching has been useful in estimating these modes of occurrence [1,2,3,4,5]. This technique is much faster than previous attempts to determine the modes of occurrence of trace elements in coal, such as scanning electron microscopy[6], analysis of density separates of coal[7,8] and low-temperature ash[9]. However, relatively little has been published about the reproducibility and reliability of the solvent leaching procedure. For this paper, three previously analyzed coal samples were leached individually and (or) sequentially with 1*N* CH_3COONH_4, 2*N* HCl, 48% HF, and 2*N* HNO_3 using procedures described previously[4,5]. The concentrations of 45 elements were determined by analyzing the leachate by inductively coupled argon plasma-atomic emission spectroscopy (ICAP-AES) and (or) the residue by instrumental neutron activation analysis (INAA).

RESULTS

The percent determined by both INAA and ICAP-AES of each element sequentially leached by each solvent in the Illinois #6 coal is given in Table 1. The method of determining the percent leached is different for each technique. The percent leached by ICAP-AES is calculated by:

$$(C_S V_S / W_S) / C_{WC} \times 100 \qquad (1)$$

where C_S is the concentration of the element in solution V_S is the volume of the solution, W_S is the weight of the fraction prior to leaching (starting weight) ; and C_{WC} is the concentration of the element in the original whole coal sample given in Table 2. The percent leached in each fraction determined by INAA was calculated by

$$P_L - (C_{WC}W_S - C_F W_F / C_{WC}W_S) \times 100 \qquad (2)$$

where P_L is the total percent leached by all fractions previously leached (P_L is zero for the CH_3COONH_4 fraction); C_F is the concentration of the element in the residue after leaching and W_F is the weight of the fraction after leaching (final weight). The errors calculated are based on counting statistics[10] and are propagated through the calculations for the elements determined by INAA. Although the errors of concentrations determined by INAA are generally relatively low (1-10%), errors in the final calculation are much greater due to the subtractions and division in equation 2. Errors on individual elemental concentretions were not available for ICAP-AES. The data indicates that errors in these concentrations are generally higher than INAA errors, because more elements are near the detection limits of the ICAP-AES technique[11]. Because equation 1 does not have subtraction terms, the overall errors may be comparable. In cases where the error was greater than the percent leached value, this value was converted to an upper limit and denoted by a * in the tables. In a few cases, equation 2 lead to a negative number. If that number was less than the calculated error, then the value was reported as an upper limit with a * as above; but when the value exceeded the error value, no value could be calculated and a ? was reported in a manner similar to Finkelman et al[4] in 1990.

Considering the errors, there is generally good agreement between INAA and ICAP-AES results. The INAA results can also be evaluated against the data reported in the 1990 study included in Table 1. The comparison to previous data is generally good, but there are some exceptions. The 1:7 HNO_3 solution used in this study was more effective than
1:9 HNO_3 . The differences in nitric acid leaching between this study and the 1990 study have been attributed to oxidation of the coal matrix by the stronger solution[5].

Results for the Buelah-Zap lignite sample and the Lower Bakerstown coal sample (Tables 3-6) are similar to the results found for the Illinois #6 coal sample. The leaching study on the Lower Bakerstown coal was done in duplicate to further assist in evaluate reproducibility. Results from individual leaching experiments some in duplicate or triplicate can be found on Table 7. Many of these experiments were done in duplicate or triplicate. Hot (80° C) and room temperature (RT) HCl and HNO_3 acid were used to determine if differences in temperature were important. Generally, differences in temperature were not as significant as were differences in concentration.

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Table 1. Percent of selected elements sequentially leached from the Illinois #6 coal by different solvents. The concentrations of elements used to calculate the percent leached were determined by instrumental neutron activation analysis (INAA) on residue of the leached coal. The concentrations of the elements used to calculate the percent leached were determined directly by inductively coupled argon plasma atomic emission spectrometry (ICAP-AES) on the leached solutions. *=number converted to an upper limit due to high errors in calculations; ND= not determined, ? No value could be determined from data, 1990 refers to data previously reported by Finkelman et al.[4]

	CH_3COONH_4		HCl		HF		HNO_3	
Element-method	Run1	1990	Run1	1990	Run1	1990	Run1	1990
B-ICAP-AES	1	ND	32	ND	45	ND	<18	ND
Na-INAA	73±4	65	10±5	0	15±6	0	6±0.4	15
Na-ICAP-AES	70	ND	12	ND	<2	ND	<3	ND
Mg-ICAP-AES	8	ND	7	ND	57	ND	142	ND
Al-ICAP-AES	17	ND	36	ND	42	ND	47	ND
Si-ICAP-AES	0	ND	1	ND	ND	ND	2	ND
P-ICAP-AES	<44	ND	<50	ND	<58	ND	<100	ND
K-INAA	9±7	13	<12*	0	<91	78	<10*	>7
K-ICAP-AES	<21	ND	<24	ND	81	ND	<57	ND
Ca-ICAP-AES	71	ND	21	ND	2	ND	3	ND
Sc-INAA	<10*	0	<23*	15	64±14	32	20±16	4
Ti-ICAP-AES	<2	ND	3	ND	81	ND	9	ND
V-ICAP-AES	<20	ND	30	ND	219	ND	124	ND
Cr-INAA	<1*	0	?	22	53±4	9	33±8	24
Cr-ICAP-AES	<3	ND	22	ND	71	ND	52	ND
Mn-ICAP-AES	71	ND	21	ND	8	ND	<8	ND
Fe-INAA	9±1	0	9±1	0	4±1	0	75±6	48
Fe-ICAP-AES	0.5	ND	4	ND	10	ND	122	ND
Co-INAA	10±1	19	47±2	39	15±3	0	23±4	7
Co-ICAP-AES	<24	ND	43	ND	<31	ND	<64	ND
Ni-INAA	27±16	35	<30*	32	39±20	0	41±23	0
Ni-ICAP-AES	17	ND	36	ND	42	ND	47	ND
Cu-ICAP-AES	<10	ND	<11	ND	<13	ND	78	ND
Zn-INAA	8±6	ND	21±9	ND	58±9	ND	12±10	ND
Zn-ICAP-AES	<1	ND	26	ND	58	ND	18	ND
As-INAA	10±7	0	19±10	17	<18*	0	60±11	0
Se-INAA	21±4	0	<7*	0	<7*	0	65±5	0
Br-INAA	40±9	53	?	?	?	?	?	?
Rb-INAA	<26*	ND	<27*	ND	67±21	ND	<38*	ND
Sr-INAA	<49*	30	<100*	0	<40*	5	<20*	ND
Sr-ICAP-AES	36	ND	10	ND	19	ND	22	ND
Y-ICAP-AES	<25	ND	<29	ND	<33	ND	<69	ND
Zr-ICAP-AES	<5	ND	<5	ND	46	ND	<12	ND
Ag-ICAP-AES	<100	ND	<100	ND	<100	ND	<100	ND
Sb-INAA	<16*	0	<16*	13	22±11	0	60±13	6
Cs-INAA	<2*	0	15±7	0	71±8	0	20±8	88
Ba-INAA	28±13	11	22±19	25	31±20	9	20±15	8
Ba-ICAP-AES	32	ND	18	ND	35	ND	28	ND
La-INAA	<3*	0	38±3	37	11±2	4	43±1	29
Ce-INAA	<10*	8	45±15	38	<25*	0	40±17	24
Ce-ICAP-AES	<17	ND	34	ND	<22	ND	<46	ND
Nd-INAA	20±12	ND	<100*	ND	?	ND	58±20	ND
Sm-INAA	13±10	0	40±15	44	<20*	0	37±17	13
Eu-INAA	17±10	0	29±14	40	<20*	0	38±16	7
Tb-INAA	21±16	0	<40*	28	<40*	0	39±26	12
Yb-INAA	<15*	0	<22*	0	25±16	18	53±16	0
Lu-INAA	<18*	ND	<38*	ND	<40*	ND	55±28	ND
Hf-INAA	?	0	<2*	8	62±11	23	<12	9
Ta-INAA	<16*	0	<18*	0	48±17	0	36±19	0
W-INAA	?	0	?	0	?	0	?	0
Pb-ICAP-AES	<24	ND	30	ND	12	ND	50	ND
Th-INAA	<2*	0	24±14	30	<28*	0	51±16	14
U-INAA	<19*	0	<22*	16	24±15	0	40±16	0

Table 2. Concentrations of trace elements in the original Illinois #6 coal sample in parts per million except as noted.

B	Na	Mg	Al (%)	Si(%)	P	K (%)	Ca (%)	Sc	Ti	V	Cr	Mn
160	1020	750	1.25	3.0	47	0.2	0.93	2.3	700	34.5	35.8	76

Fe (%)	Co	Ni	Cu	Zn	As	Se	Br	Rb	Sr	Y	Zr	Ag
2.7	4.4	30	10.1	201	4.7	4.2	5.72	16	32.2	4.1	22.8	0.62

Sb	Cs	Ba	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Hf	Ta
0.84	0.94	86	5.99	10.2	6.5	1.03	0.226	0.134	0.47	0.06	0.435	0.176

W	Pb	Th	U
1.51	8.6	1.65	4.27

Table 3. Percent of selected elements sequentially leached from the Buelah-Zap lignite by different solvents. The concentrations of elements used to calculate the percent leached were datermined by instrumental neutron activation analysis (INAA) on residue of the leached coal. The concentrations of the elements used to calculate the percent leached were determined directly by inductively coupled argon plasma atomic emission spectrometry (ICAP-AES) on the leached solutions. *=number converted to an upper limit due to high errors in calculations; ND= not determined, ? No value could be determined from data, 1990 refers to data previously reported by Finkelman et al.[4]

	CH_3COONH_4		HCl		HF		HNO_3	
Element-method	Run1	1990	Run1	1990	Run1	1990	Run1	1990
B-ICAP-AES	<17	ND	40	ND	<26	ND	<40	ND
Na-INAA	54±4	95	26±5	0	18±5	0	19±1	0
Na-ICAP-AES	<100	ND	<100	ND	<100	ND	<100	ND
Mg-ICAP-AES	101	ND	11	ND	17	ND	42	ND
Al-ICAP-AES	1	ND	28	ND	66	ND	1	ND
Si-ICAP-AES	<1	ND	10	ND	ND	ND	3	ND
P-ICAP-AES	<22	ND	100	ND	<34	ND	<66	ND
K-INAA	41±21	?	<32*	0	<100	>85	<100	ND
K-ICAP-AES	<100	ND	<100	ND	<100	ND	<100	ND
Ca-ICAP-AES	82	ND	23	ND	1	ND	0	ND
Sc-INAA	?	0	50±6	15	45±11	32	<13*	4
Ti-ICAP-AES	<7	ND	23	ND	66	ND	<21	ND
V-ICAP-AES	167	ND	<86	ND	<100	ND	<100	ND
Cr-INAA	?	14	?	70	?	0	22±6	0
Cr-ICAP-AES	371	ND	<66	ND	203	ND	<100	ND
Mn-ICAP-AES	46	ND	42	ND	<5	ND	<10	ND
Fe-INAA	<10*	15	37±14	0	<26*	0	50±22	0
Fe-ICAP-AES	<1	ND	34	ND	9	ND	30	ND
Co-INAA	?	5	75±5	58	13±7	0	<12*	0
Co-ICAP-AES	<100	ND	<100	ND	<100	ND	<100	ND
Ni-INAA	<17*	0	32±14	0	<44*	0	<30*	0
Ni-ICAP-AES	<27	ND	69	ND	<100	ND	<100	ND
Cu-ICAP-AES	76	ND	<32	ND	<41	ND	<79	ND
Zn-INAA	3±1	0	<73*	76	<23*	16	<1*	0
Zn-ICAP-AES	<27	ND	82	ND	67	ND	<79	ND
As-INAA	<6*	16	22±8	4	19±1	0	44±1	11
Se-INAA	<17*	0	9±6	0	?	0	58±12	0
Br-INAA	1±0.1	0	?	?	?	?	?	?
Rb-INAA	16±8	ND	<23*	ND	>23	ND	>34[a]	ND
Sr-INAA	64±1	45	>32*	54	?	0	?	0
Sr-ICAP-AES	76	ND	26	ND	1	ND	<1*	ND
Y-ICAP-AES	<67	ND	<100	ND	<100	ND	<100	ND
Zr-ICAP-AES	<11	ND	<13	ND	61	ND	<33	ND
Ag-ICAP-AES	<100	ND	<100	ND	<100	ND	<100	ND
Sb-INAA	<13*	0	<19*	0	68±30	14	<40*	30
Cs-INAA	<10*	0	<46*	0	>68	82	?	?
Ba-INAA	<13*	23	71±13	73	29±15	?	<1	?
Ba-ICAP-AES	18	ND	87	ND	16	ND	1	ND
La-INAA	?	0	75±9	83	13±10	0	<18*	7
Ce-INAA	?	14	69±13	70	18±13	0	14±8	7
Ce-ICAP-AES	<61	ND	92	ND	<91	ND	<100	ND
Nd-INAA	33±25	ND	>47	ND	0	ND	>16	ND
Sm-INAA	<5*	0	73±8	81	18±14	0	<21*	7
Eu-INAA	<19*	0	71±30	79	>50	0	<40*	7
Tb-INAA	<11*	0	70±15	84	17±15	0	<20*	3
Yb-INAA	<20*	0	67±31	72	<40*	0	<45*	7
Lu-INAA	<22*	ND	64±18	ND	<38*	ND	<28*	ND
Hf-INAA	?	0	<10*	0	81±10	55	<10*	0
Ta-INAA	?	0	<13*	0	94±13	37	14±11	0
W-INAA	?	0	?	0	?	0	?	0
Pb-ICAP-AES	<100	ND	172	ND	<100	ND	<100	ND
Th-INAA	?	0	39±8	60	60±8	0	21±9	29
U-INAA	<18*	0	54±27	60	34±31	27	<40*	0

[a]Includes amount leached by HF

Table 4. Concentrations of trace elements in the original Buelah-Zap lignite sample in parts per million except as noted.

B	Na	Mg	Al	Si	P	K	Ca (%)	Sc	Ti	V	Cr	Mn
79	4960	3740	4000	6700	120	250	1.48	0.76	190	3.7	2.4	81

Fe	Co	Ni	Cu	Zn	As	Se	Br	Rb	Sr	Y	Zr	Ag
4800	0.82	5	5	5.23	2.63	0.59	1	0.93	530	2	12	0.14

Sb	Cs	Ba	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Hf	Ta
0.173	0.5	610	2.67	3.92	2.3	0.4	0.081	0.52	0.26	0.036	0.293	0.92

W	Pb	Th	U
0.38	1.5	0.9	0.41

Table 5. Percent of selected elements sequentially leached from the Lower Bakerstown coal by different solvents. The concentrations of elements used to calculate the percent leached were determined by instrumental neutron activation analysis (INAA) on residue of the leached coal. The concentrations of the elements used to calculate the percent leached were determined directly by inductively coupled argon plasma atomic emission spectrometry (ICAP-AES) on the leached solutions. *=number converted to an upper limit due to high errors in calculations; ND= not determined, ? No value could be determined from data, 1990 refers to data previously reported by Finkelman et al.[4]

	CH_3COONH_4			HCl			HF			HNO_3		
Element-method	Run1	Run2	1990	Run1	Run2	1990	Run1	Run2	1990	Run1	Run 2 80°C	1990
B-ICAP-AES	<100	<100	ND	<100	<100	ND	<100	<100	ND	<100	<100	ND
Na-INAA	10±4	10±4	0	<7*	<10*	0	75±5	75±5	65	<8*	<9*	0
Na-ICAP-AES	19	13	ND	8	<9	ND	61	72	ND	<13	<16	ND
Mg-ICAP-AES	10	12	ND	6	7	ND	41	48	ND	5	5	ND
Al-ICAP-AES	59	45	ND	11	16	ND	36	24	ND	38	<16	ND
Si-ICAP-AES	<0.2	<0.3	ND	0.3	0.5	ND	ND	ND	ND	4	<1	ND
P-ICAP-AES	12	<9	ND	54	78	ND	14	17	ND	<13	19	ND
K-INAA	<13*	<13*	0	<22*	<18*	0	>89	>85	>79	>2	>1	ND
K-ICAP-AES	<58	<88	ND	<64	<88	ND	84	<100	ND	<100	<100	ND
Ca-ICAP-AES	75	66	ND	21	22	ND	18	18	ND	9	8	ND
Sc-INAA	<3*	<3*	8	14±3	16±3	63	33±3	31±3	23	<3*	6±3	0
Ti-ICAP-AES	<4	<3	ND	<3	<4	ND	49	53	ND	21	11	ND
V-ICAP-AES	<20	<30	ND	<22	<30	ND	49	52	ND	<44	<53	ND
Cr-INAA	<4*	<4*	0	?	?	0	?	?	12	?	?	11
Cr-ICAP-AES	<12	<17	ND	<12	<18	ND	104	109	ND	77	73	ND
Mn-ICAP-AES	65	60	ND	<23	<32	ND	<26	<39	ND	<48	<57	ND
Fe-INAA	<12*	<11*	15	29±7	42±7	26	29±7	23±7	8	40±8	33±8	4
Fe-ICAP-AES	17	<0.2	ND	40	61	ND	8	8	ND	35	49	ND
Co-INAA	60±3	60±3	59	11±3	12±3	8	<7*	<6*	0	8±3	8±3	0
Co-ICAP-AES	79	67	ND	<17	<23	ND	<19	<28	ND	<33	<40	ND
Ni-INAA	39±14	39±14	55	<38*	<19*	0	<19*	<19*	0	22±18	34±18	0
Ni-ICAP-AES	59	45	ND	10	15	ND	36	24	ND	39	<16	ND
Cu-ICAP-AES	46	<15	ND	21	24	ND	21	30	ND	<22	<26	ND
Zn-INAA	53±6	53±6	64	31±8	19±8	21	<9*	<13*	0	<11*	<11*	0
Zn-ICAP-AES	72	64	ND	18	43	ND	9	9	ND	<5	39	ND
As-INAA	<5*	6±4	0	57±5	46±5	41	15±6	16±6	6	30±5	30±5	6
Se-INAA	<10*	<11*	0	<18*	<23*	0	<14*	<25*	0	74±14	70±14	0
Br-INAA	<4*	<4*	0	7±4	17±4	19	9±4	<4*	15	<40*	<40*	0
Rb-INAA	<24*	<24*	ND	<22*	<23*	ND	>91	>90	ND	>7	>3	0
Sr-INAA	<25*	<25*	16	<24*	<33*	0	58±24	63±24	9	<28*	<30*	11
Sr-ICAP-AES	19	17	ND	7	9	ND	41	44	ND	8	8	ND
Y-ICAP-AES	<31	<47	ND	<34	<47	ND	<39	<57	ND	<69	<83	ND
Zr-ICAP-AES	<6	<10	ND	<7	<10	ND	30	36	ND	14	<17	ND
Ag-ICAP-AES	<100	<100	ND	<100	<100	ND	<100	<100	ND	<100	<100	ND
Sb-INAA	<6*	<6*	10	37±4	38±5	33	20±4	21±4	21	22±4	30±4	0
Cs-INAA	<17*	<17*	12	<17*	<18*	?	84±19	85±20	37	>21*	<21*	14
Ba-INAA	<17*	<17*	23	<19*	<19*	?	75±19	79±18	?	17±15	23±18	>23
Ba-ICAP-AES	22	11	ND	67	12	ND	27	19	ND	10	<8	ND
La-INAA	<4*	<4*	0	10±5	11±5	0	13±5	16±5	5	25±4	34±4	22
Ce-INAA	<4*	<4*	0	14±4	14±4	3	12±5	14±4	0	22±5	34±3	22
Ce-ICAP-AES	<20	<30	ND	<22	<30	ND	<25	<37	ND	<44	<53	ND
Nd-INAA	<19*	<19*	ND	22±17	30±17	ND	17±1	16±3	ND	26±17	32±17	ND
Sm-INAA	<3*	<3*	0	22±5	19±4	9	10±4	12±4	0	15±4	24±4	17
Eu-INAA	<5*	<5*	0	28±7	23±6	14	<14*	9±7	7	12±7	22±7	16
Tb-INAA	<7*	<7*	0	23±10	20±10	15	<16*	<10*	0	12±10	23±10	16
Yb-INAA	<12*	<12*	20	<18*	16±9	?	13±8	<15*	?	<15*	17±8	9
Lu-INAA	<8*	<8*	ND	<16*	<23*	ND	<17*	11±8	ND	11±9	15±8	ND
Hf-INAA	<10*	<10*	0	<8*	<10*	0	41±8	40±8	21	<9*	10±8	7
Ta-INAA	<9*	<9*	0	<20*	<13*	0	19±13	17±12	0	19±12	34±12	0
W-INAA	?	?	0	<25*	<23*	0	42±24	40±21	15	<30*	26±19	0
Au-INAA	>31	>28	ND	?	?	ND	?	<32*	ND	?	<33*	ND
Pb-ICAP-AES	35	<53	ND	62	101	ND	5	<7	ND	<79	<94	ND
Th-INAA	<4*	<4*	0	9±5	13±4	0	9±4	9±4	6	15±4	16±4	10
U-INAA	15±13	<20*	0	<18*	<18*	0	37±17	24±17	22	<24*	19±17	9

Table 6. Concentrations of trace elements in the original Lower Bakerstown coal sample in parts per million except as noted.

B	Na	Mg	Al (%)	Si (%)	P	K	Ca (%)	Sc	Ti	V	Cr	Mn
2.86	170	330	0.74	1.07	360	700	1.4	2	430	10.4	9.1	9.7

Fe (%)	Co	Ni	Cu	Zn	As	Se	Br	Rb	Sr	Y	Zr	Ag
0.86	6.92	17.9	10.6	47.9	2.63	0.59	34.1	4.85	82	3.33	16	0.41

Sb	Cs	Ba	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu	Hf	Ta
1.5	0.29	34	6.08	10.4	3.65	1.20	0.23	0.14	0.54	0.67	0.38	0.11

W	Au	Pb	Th	U
0.65	0.001	5.84	1.32	0.53

Table 7. Percent of selected elements leached from the Lower Bakerstown coal by different solvents. Individual experiments were conducted using room temperature solvents (RT) and 80°C solvents (hot). The numbers after RT and hot indicate run numbers. The concentrations of elements used to calculate the percent leached were determined by instrumental neutron activation analysis (INAA) on residue of the leached coal. The concentrations of the elements used to calculate the percent leached were determined directly by inductively coupled argon plasma atomic emission spectrometry (ICAP-AES) on the leached solutions. *=number converted to an upper limit due to high errors in calculations; ND= not determined, ? No value could be determined from data,

	CH_3COONH_4	HCl					HF	HNO_3				
Element- method	RT1	RT1	RT2	RT3	Hot1	Hot2	RT1	RT1	RT2	RT3	Hot1	Hot2
B-ICAP-AES	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100
Na-INAA	9±4	17± 3	15±3	19±3	31±4	30±4	86±4	35±4	25±4	19±4	18±4	30±4
Na-ICAP-AES	15	16	<59	<59	<59	<65	124	31	<59	<65	<65	<65
Mg-ICAP-AES	47	57	28	29	57	60	67	30	30	31	40	51
Al-ICAP-AES	59	57	<56	<56	68	67	70	75	66	78	81	80
Si-ICAP-AES	<0.3	1	<2	<2	16	17	ND	3	<2	17	6	7
P-ICAP-AES	<9	69	72	72	88	92	83	73	71	99	80	92
K-INAA	<13*	12±10	13±11	12±11	37±11	39±11	<99*	31±10	15±11	13±11	16±11	22±11
K-ICAP-AES	<86	<9	<100	<100	<100	<100	99	<86	<100	<100	<100	<100
Ca-ICAP-AES	62	77	100	97	121	131	41	108	106	119	117	135
Sc-INAA	<3*	17±2	18±3	19±2	29±2	28±2	47±2	35±2	21±2	20±2	20±2	29±3
Ti-ICAP-AES	<4	<43	<23	<23	<23	<26	54	<4	<23	<25	<23	<26
V-ICAP-AES	<29	<29	<100	<100	<100	<100	57	<29	<100	<100	<100	<100
Cr-INAA	<6*	<4*	<6*	<4*	<4*	9±4	23±4	15±4	<7*	<5*	?	8±6
Cr-ICAP-AES	<17	<17	<100	<100	<100	<100	42	<17	<100	<100	<100	<100
Mn-ICAP-AES	56	43	<100	<100	<100	<100	68	40	<100	<100	<100	<100
Fe-INAA	<4*	49±4	48±4	53±3	54±4	53±4	59±4	97±5	96±5	96±5	96±5	96±5
Fe-ICAP-AES	<0.2	53	53	53	58	60	56	108	104	113	103	116
Co-INAA	60±2	70±2	70±2	72±3	73±3	73±2	72±3	85±2	79±2	80±3	79±3	83±2
Co-ICAP-AES	65	65	<100	<100	<100	<100	65	73	<100	<100	<100	<100
Ni-INAA	33±13	44±13	50±13	51±13	46±13	61±13	59±13	70±13	63±13	65±13	61±13	68±13
Ni-ICAP-AES	47	57	<56	<56	66	67	70	75	66	76	81	80
Cu-ICAP-AES	<14	31	<94	<94	<94	<100	57	87	<94	<100	<100	<100
Zn-INAA	57±8	84±5	84±5	85±5	86±5	85±5	85±5	93±5	87±5	86±5	83±5	90±5
Zn-ICAP-AES	88	100	220	124	128	116	138	150	103	144	125	116
As-INAA	<5*	60±4	59±4	62±4	61±4	60±4	65±4	96±4	94±4	96±4	96±4	95±4
Se-INAA	<10*	<10*	21±9	<15*	11±10	<15*	<15*	86±10	57±10	58±10	49±9	78±10
Br-INAA	<4*	11±3	16±3	16±3	73±3	73±3	29±3	57±3	36±3	23±3	23±3	44±3
Rb-INAA	<14*	<24*	<22*	18±14	48±14	45±14	81±16	38±14	<24*	15±14	<34*	26±14
Sr-INAA	<18*	23±17	<34*	<29*	64±18	73±18	52±17	61±17	40±20	30±17	22±17	49±17
Sr-ICAP-AES	17	22	28	29	83	90	48	48	35	38	68	75
Y-ICAP-AES	<45	<45	<100	<100	<100	<100	<45	<45	<100	<100	<100	<100
Zr-ICAP-AES	<10	<9	<62	<62	<62	<70	81	<10	<62	<69	<70	<69
Ag-ICAP-AES	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	<100	ND
Sb-INAA	<5*	36±3	44±3	32±3	28±3	25±3	34±3	56±3	21±4	48±3	47±3	54±3
Cs-INAA	<14*	<17*	18±12	22±12	56±13	56±13	78±14	42±12	15±12	27±12	23±12	36±14
Ba-INAA	<22*	<20*	<17*	<15*	45±11	53±12	70±12	47±12	<15*	23±18	>25	19±14
Ba-ICAP-AES	15	12	<30	<30	64	72	141	29	<29	<33	49	54
La-INAA	<5*	13±3	17±3	20±3	58±3	58±3	13±3	52±3	22±3	19±3	19±3	43±3
Ce-INAA	<3*	15±4	21±4	24±3	59±3	59±3	9±3	55±3	25±3	24±3	23±3	47±3
Ce-ICAP-AES	<29	<29	<100	<100	<100	<100	<29	29	<100	<100	<100	<100
Nd-INAA	<13*	14±12	<25*	17±12	40±12	61±12	<16*	57±12	24±11	<15*	31±12	46±12
Sm-INAA	<5*	25±3	29±3	30±3	59±3	58±3	11±3	59±3	36±3	32±3	32±3	50±3
Eu-INAA	<5*	24±5	30±5	32±5	56±5	54±5	10±5	56±5	34±5	35±5	35±5	51±5
Tb-INAA	<7*	17±7	29±7	28±7	40±7	39±7	8±7	43±7	27±7	27±7	24±7	42±7
Yb-INAA	<7*	8±6	<12*	9±6	25±5	21±6	<6*	35±5	14±6	12±6	12±6	18±6
Lu-INAA	<6*	11±6	<13*	13±7	23±5	24±5	6±5	34±5	16±5	13±6	9±6	24±6
Hf-INAA	<6*	<9*	?	<6*	<12*	8±6	40±6	12±6	<6*	<11*	<13*	7±6
Ta-INAA	?	?	?	?	?	?	<13*	<9*	?	?	?	?
W-INAA	?	?	?	?	<14*	?	23±13	<14*	?	?	?	?
Au-INAA	<60*	<44*	33±30	?	59±33	<31*	?	<32*	<42*	<56*	?	?
Pb-ICAP-AES	<52	93	<100	<100	<100	<100	32	98	<100	<100	<100	<100
Th-INAA	<3*	9±3	10±5	13±3	29±3	29±3	8±3	32±3	14±3	17±5	15±3	25±4
U-INAA	<24*	<19*	<24*	34±12	29±11	32±11	21±12	44±11	23±12	28±12	20±12	36±12