

SUPPLEMENTARY MATERIAL TO
**Investigation of different extraction procedures for the
determination of major and trace elements in coal by
ICP-AES and ion chromatography**

ALEKSANDRA M. TASIĆ¹, IVANA D. SREDOVIĆ IGNJATOVIĆ^{2*}, LJUBIŠA M.
IGNJATOVIĆ¹, IVAN B. ANĐELKOVIĆ³, MALIŠA P. ANTIĆ²
and LJUBINKA V. RAJAKOVIĆ⁴

¹University of Belgrade, Faculty of Physical Chemistry, Studentski trg 12–16, 11000
Belgrade, Serbia, ²University of Belgrade, Faculty of Agriculture, Nemanjina 6, 11080
Belgrade, Serbia, ³University of Belgrade, Innovation Center of the Faculty of Chemistry,
Studentski trg 12–16, 11000 Belgrade, Serbia and ⁴University of Belgrade, Faculty of
Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

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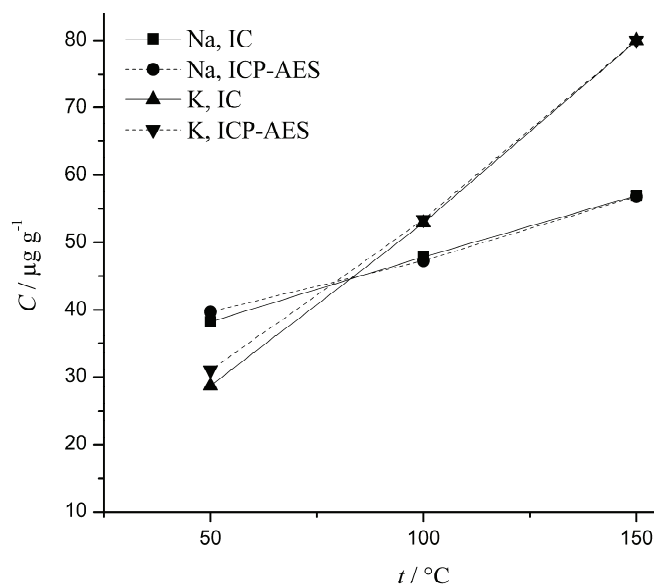


Fig. S-1. The contents of: Na and K obtained by microwave assisted extraction at different temperatures, measured by IC and ICP-AES.

* Corresponding author. E-mail: isredovic@agrif.bg.ac.rs

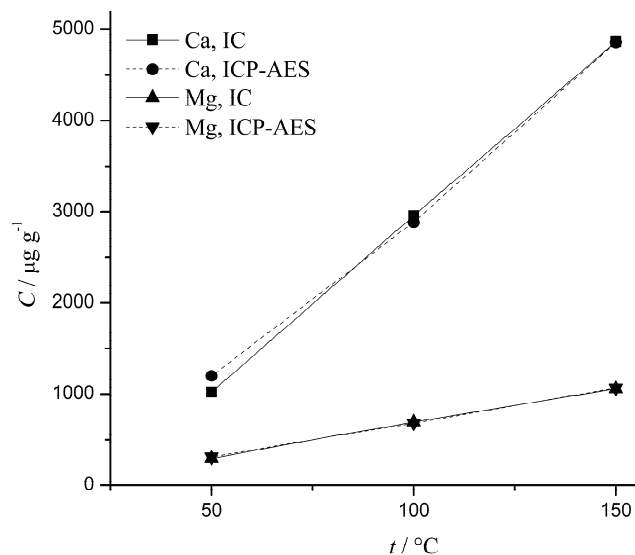


Fig. S-2. The contents of Ca and Mg obtained by microwave assisted extraction at different temperatures, measured by IC and ICP-AES.

TABLE S-I. The results of the determinations of the contents ($\mu\text{g g}^{-1}$) of Al, As, Cd, Co, Cr, Fe, Mn, Ni and Pb obtained by rotary (RE), ultrasonic (UE) and microwave (ME) extraction under different time and temperature regimes

Extraction condition	Al	As	Cd	Co	Cr	Fe	Mn	Ni	Pb
RE									
30 min	0.32	0.19	–	0.002	0.005	1.71	2.03	0.02	0.04
60 min	0.20	0.21	–	–	0.008	0.87	1.99	0.03	0.09
90 min	0.14	0.21	–	0.004	0.005	1.11	2.06	0.04	0.07
120 min	0.33	0.20	–	–	0.007	1.68	2.19	0.02	0.04
UE									
10 min	0.14	0.18	–	0.001	0.002	0.91	1.52	0.05	0.07
20 min	0.08	0.20	–	0.003	0.013	0.40	2.13	0.06	0.08
30 min	0.15	0.20	–	0.004	0.012	0.46	2.26	0.04	0.05
40 min	0.20	0.21	–	0.003	0.012	0.45	2.26	0.04	0.08
50 min	0.08	0.23	–	0.003	0.010	0.41	2.06	0.03	0.05
ME									
50 °C	3.36	0.46	0.01	–	0.03	6.80	4.30	0.10	0.10
100 °C	14.38	0.89	0.03	0.16	0.08	33.2	21.77	0.35	0.18
150 °C	39.40	2.20	0.04	0.52	0.08	528	61.00	1.41	0.28

TABLE S-II. Concentrations of the trace elements As, Be, Cd, Co, Cr, Ni and Pb ($\mu\text{g g}^{-1}$), in the different fractions obtained by sequential microwave extraction at 50, 100 and 150 °C

Fraction	<i>t</i> / °C	As	Be	Cd	Co	Cr	Ni	Pb
I	50	1.12	–	0.06	0.39	0.04	0.66	0.13
II		2.09	0.15	0.28	1.74	0.08	4.09	2.66
III		4.81	0.17	0.23	0.64	3.10	5.60	4.70
IV		7.49	0.20	0.43	1.35	15.48	4.91	12.63
V		0.25	–	–	0.12	0.21	0.16	0.81
Sum		15.76	0.52	1.00	4.27	18.91	15.42	20.93
I	100	1.09	0.01	0.02	0.85	0.07	1.40	0.11
II		4.15	0.28	0.36	1.86	0.13	6.20	1.06
III		3.66	0.12	0.15	0.35	4.83	4.49	4.77
IV		5.87	0.13	0.33	1.03	11.57	3.99	6.91
V		–	0.01	0.03	0.21	1.57	0.53	1.46
Sum		14.77	0.55	0.89	4.30	18.17	16.61	14.31
I	150	2.84	0.20	0.10	1.14	0.11	2.45	0.57
II		5.49	0.26	0.40	1.74	0.28	7.25	3.20
III		1.33	0.09	0.15	0.42	10.46	3.33	7.61
IV		1.23	0.05	0.24	0.95	5.55	4.17	8.39
V		0.42	0.07	0.17	0.53	5.55	1.44	2.80
Sum		11.31	0.67	1.06	4.78	21.95	18.64	22.57
ASTM D6349-13		53.60	n.d.	n.d.	14.10	51.50	35.40	42.56

TABLE S-III. Determination of the elements ($\mu\text{g g}^{-1}$) in a spiked coal sample by RE, UE and MAE with ICP-AES determination

Element	Spike 1			Spike 2			Spike 3		
	Expected	Found	R / %	Expected	Found	R / %	Expected	Found	R / %
Rotary extraction									
Al	1.33	1.26±0.07	95	2.33	2.38±0.12	102	3.33	3.10±0.18	93
Ca	1260	1222±60	97	1560	1498±80	96	1860	1916±90	103
Fe	2.68	2.76±0.12	103	3.68	3.82±0.15	104	4.68	4.31±0.21	92
K	35.2	32.7±7.1	93	45.2	47.4±8.1	105	55.2	53.5±9.2	97
Mg	425	416±18	98	525	488±30	93	625	588±35	94
Mn	3.19	3.29±0.20	103	4.19	4.06±0.20	97	5.19	4.93±0.23	95
Na	48.7	47.8±6	96	58.7	60±8	102	67.8	71±14	105
As	1.20	1.22±0.08	102	2.20	2.16±0.11	98	3.20	3.33±0.18	104
Cd	1.00	1.04±0.09	104	2.00	1.91±0.09	96	3.00	2.80±0.15	93
Co	1.01	0.93±0.06	92	2.01	1.90±0.10	94	3.01	2.90±0.16	96
Cr	1.00	0.93±0.05	93	2.00	1.90±0.14	95	3.00	3.05±0.18	102
Ni	1.02	0.98±0.06	96	2.02	2.10±0.10	104	3.02	2.95±0.16	98
Pb	1.04	1.01±0.06	97	2.04	2.08±0.12	102	3.04	2.90±0.17	95
Ultrasonic extraction									
Al	1.15	1.20±0.07	104	2.15	2.10±0.10	98	3.15	3.11±0.15	99
Ca	1340	1300±65	97	1640	1580±70	96	1940	1860±85	96
Fe	1.46	1.40±0.07	96	2.46	2.51±0.08	102	3.46	3.38±0.10	98
K	37.5	36±6	96	47.5	43±8	90	57.5	56±9	97
Mg	450	439±16	98	550	541±20	98	650	638±26	98

TABLE S-III. Continued

Element	Spike 1			Spike 2			Spike 3		
	Expected	Found	R / %	Expected	Found	R / %	Expected	Found	R / %
Ultrasonic extraction									
Mn	3.26	3.30±0.14	101	4.26	4.18±0.16	98	5.26	5.19±0.20	99
Na	50.0	47±7	94	60.0	63±9	105	70.0	67±14	96
As	1.20	1.16±0.09	97	2.20	2.16±0.12	98	3.20	3.22±0.15	101
Cd	1.00	1.06±0.10	106	2.00	1.90±0.12	95	3.00	2.91±0.15	97
Co	1.00	0.90±0.06	90	2.00	1.91±0.09	96	3.00	2.89±0.12	96
Cr	1.01	1.03±0.09	102	2.01	1.96±0.18	97	3.01	2.91±0.25	97
Ni	1.04	1.01±0.07	97	2.04	2.02±0.11	99	3.04	3.06±0.20	101
Pb	1.05	1.08±0.08	103	2.05	2.01±0.10	98	3.05	3.02±0.21	99
Microwave extraction									
Al	59.4	55±4	93	79.4	74±6	93	99.4	96±7	97
Ca	5100	4790±360	94	5400	5120±380	95	5700	5280±410	93
Fe	728	670±32	92	928	881±48	95	1128	1173±65	104
K	100	94±15	94	120	126±20	105	140	147±25	105
Mg	1280	1331±66	104	1580	1648±81	104	1880	1767±96	94
Mn	81.0	77.8±6	96	101	97±7	96	121	123±9	102
Na	76.0	80±11	105	96.0	91±15	95	116	106±21	91
As	3.20	2.94±0.12	92	4.20	4.36±0.15	104	5.20	5.30±0.17	102
Cd	1.04	1.10±0.07	106	2.04	2.10±0.09	103	3.04	2.92±0.10	96
Co	1.52	1.46±0.11	96	2.52	2.43±0.13	96	3.52	3.45±0.15	98
Cr	1.08	1.01±0.15	94	2.08	2.12±0.20	102	3.08	2.90±0.24	94
Ni	2.41	2.50±0.10	104	3.41	3.36±0.15	99	4.41	4.38±0.19	99
Pb	1.28	1.17±0.09	91	2.28	2.24±0.12	98	3.28	3.41±0.16	104