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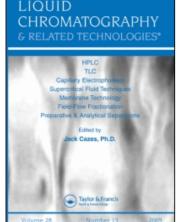
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Quantitative Determination of Iron and Aluminium in Some Alloys and Silicate Rocks After a Cation Exchange Separation on Zirconium(IV) Phospho and Silico Arsenates

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QUANTITATIVE DETERMINATION OF IRON AND ALUMINIUM IN SOME ALLOYS AND SILICATE ROCKS AFTER A CATION EXCHANGE SEPARATION ON ZIRCONIUM(IV)

PHOSPHO AND SILICO ARSENATES

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ABSTRAC T

A rapid and quantitative method has been developed for the analysis of some iron and aluminium based alloys and silicate rocks using zirconium(IV) based arsenophosphate and arsenosilicate cation exchangers. The method is simple, reproducible and precise with a standard deviation < 3%, for the direct determination of iron and aluminium in rocks and alloys. The low standard deviation values suggest that the method should be useful for the standardization purposes.

INTRODUCTION

Analysis of alloys and rocks is important in chemical technology as the presence of various constituents play a vital role in their applications. Although several papers have earlier been published in this field using well known analytical techniques (1-5), the ion-exchange technique is more useful as it gives fast separation of the ionic species present. However such studies have been made mostly on organic

resins (6,7) probably because of their excellent reproducibility and stability.

Inorganic ion-exchangers are well known for their high selectivity for metal ions and stability at elevated temperatures (8). Zirconium(IV) arsenophosphate (ZAP) and Zr(IV) arsenosilicate (ZAS) prepared in these laboratories (9,10) possess exceptionally good chemical stability and reproducibility in ion-exchange behaviour which improve further for ZAP on heating. A possibility of using these materials for the quantitative separation of metal ions from their synthetic mixtures and from some real samples has already been explored earlier in these laboratories (10). The present article summarizes our efforts for a quantitative separation of aluminium and iron from some alloys and rocks on their columns.

EXPERI MENTAL

Chemicals and Reagents

Zirconyl chloride used in these studies was of J.T.Baker Chemical Co.Philipsburg (USA), while trisodium orthophosphate was of BDH, Poole (London). Di-sodium arsenate and sodium silicate were E-Merck (Dermstadt) products and all other reagents and chemicals were of analak grade.

Apparatus

A pye unichem model SP-2900 atomic absorption spectrophotometer was used for the quantitative determination of various elements present in rocks and alloys.

Synthesis and Ion-Exchange Capacity of ZAP and ZAS

These materials were synthesized by the methods reported earlier (9,10) and were thermally treated by putting them at various temperatures for one hour each in a muffle furnace. They were washed thoroughly with dil.HNO₃ and then with demineralized water (DMW) till the effluents were free of any metallic or non metallic impurities as tested by atomic absorption spectrophotometry. The Na⁺-ion-exchange capacities of various samples thus obtained are reported in Table 1. On this basis a heated phase of ZAP upto 200°C (<-ZAP) and normal ZAS were used for further studies.

Distribution Studies

The distribution coefficients (Kd) for various metal ions were determined as usual by the batch process on < -ZAP and ZAS (10). Table 2 shows a comparative statement of these values in DMW and HNO_2 .

TABLE 1

Ion-Exchange Capacity of ZAP and ZAS After Thermal
Treatment

Heating temperature (°C)	Ion-excha capacity (meq./dry		% Retention ion-exchance apacity	
	ZAP	ZAS	ZAP	ZAS
45	0.94	1.30	100.0	100.0
100	0.95	1.30	101.1	100.0
200	1.03	1.25	109.6	96.2
400	0.94	0.46	100.0	35.4
600	0.84	0.20	89.4	15.4

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TABLE 2

Kd values of Various Metal Ions on & -ZAP and ZAS in DMW and HNO3 Media

Ba(II) 1900 110 83 105 65 91 Cd(II) 5400 8030 1750 186 507 130 Pb(II) 5400 8030 1750 186 507 130 Pb(II) 5400 8030 1750 186 507 130 Pb(II) 5650 5430 360 1560 1560 268 1460 Ng(II) 3150 218 0 35 0 5 Sr(II) 326 52 13 45 9 Ca(II) 3530 100 28 57 8 36 Cu(II) 5150 8000 68 206 20 190 Cu(II) 5150 8000 68 206 5 409 Cu(II) 5150 600 5 700 5 409 Cu(II) 5250 850 700 5 0 7 Sn(II) 3900 3850 3900 293 630 200 Fe (II) </th <th>Metal ions</th> <th>ia .</th> <th>DMW</th> <th>0.01M HNO₃</th> <th>HNO₃</th> <th>0.1M HN03</th> <th>HNO₃</th> <th>1M HNO₃</th> <th>NO₃</th>	Metal ions	ia .	DMW	0.01M HNO ₃	HNO ₃	0.1M HN03	HNO ₃	1M HNO ₃	NO ₃
(II) 1900 110 83 105 65 (II) 5400 8030 1750 186 507 (II) 5650 5430 360 1580 268 1 (II) 5450 218 0 35 0 0 35 0 0 (II) 526 52 30 58 20 1 465 0 0 35 0 0 35 0 0 35 0 0 35 0 0 35 12 465 12 12 20 12 20 12 20 12		≪-ZAP	ZAS	d-ZAP	ZAS	α-ZAP	ZAS	≺-ZAP	ZAS
(11) 5400 8030 1750 186 507 (11) 5650 5430 360 1580 268 (11) 4400 367 20 77 3 (11) 3150 218 0 35 0 (11) 326 52 13 45 (11) 3280 1000 28 57 8 (11) 5150 600 5 700 5 (11) 5250 85 126 20 (11) 3250 85 126 20 (11) 3250 3850 3900 293 630 (11) 3260 3850 3900 293 630	Ba (II)	1900	110	83	105	65	91	0	0
(11) 5650 5430 360 1580 268 1 (11) 4400 367 20 77 3 (11) 326 52 30 58 20 (11) 326 52 13 45 (11) 3700 314 0 81 0 (11) 3280 1000 28 57 8 (11) 5150 600 68 206 20 (11) 3250 85 126 2 3 (11) 3250 3850 3900 293 630 (11) 3900 3850 3900 293 630	cd(II)	5400	8030	1750	186	507	130	0	0
(11) 4400 367 20 77 3 (11) 3150 218 0 35 0 (11) 326 52 13 45 (11) 3700 314 0 81 0 (11) 3280 1000 28 57 8 (11) 5150 600 68 206 20 (11) 3250 85 126 20 (11) 3250 385 3900 293 630 (11) 3900 3850 3900 293 630 (11) 3260 3850 3900 3850 3900	Pb (II)	5650	5430	360	1580	268	1460	0	0
(II) 3150 218 0 35 0 (II) 326 52 30 58 20 (II) 3700 314 0 81 0 (II) 3280 1000 28 57 8 (II) 5150 8000 68 206 20 (II) 5450 600 5 700 5 (II) 3250 85 126 2 3 (II) 3900 3850 3900 293 630 (II) 3600 3850 3900 3650	Mg (II)	0044	367	02	77	ĸ	63	0	0
(II) 326 52 30 58 20 (II) 3530 69 52 13 45 (II) 3700 314 0 81 0 (II) 3280 1000 28 57 8 (II) 5450 600 68 206 20 (II) 3250 60 5 700 5 (II) 3250 65 126 2 3 (II) 3900 3850 3900 293 630 (II) 3600 3850 3600 3650	Sr(II)	3150	218	0	رج ح	0	5	0	0
(II) 3550 69 52 13 45 (II) 3700 314 0 81 0 (II) 3280 1000 28 57 8 (II) 5450 8000 68 206 20 (II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 650 (II) 3650 3650 3650 3650		326	25	30	28	50	41	0	0
(II) 3700 314 0 81 0 (II) 3280 1000 28 57 8 (II) 5450 8000 68 206 20 (II) 5450 600 5 700 5 (II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630 (II) 3600 3650 3650 3650		3530	69	52	13	45	0	0	0
(II) 3280 1000 28 57 8 (II) 5450 8000 68 206 20 (II) 5450 600 5 700 5 (II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630 (II) 3650 3650 3650 3650		3700	314	0	81	0	36	0	0
(II) 5150 8000 68 206 20 (II) 5150 600 5 700 5 (II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630 (II) 3650 3650 3650 3650 3650		3280	1000	58	57	œ	36	0	0
(II) 5150 600 5 700 5 (II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630 (II) 3650 3650 3650 3650		5150	8000	89	206	20	198	0	0
(II) 3250 85 126 2 3 (II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630		5150	009	īν	700	ľΩ	409	0	0
(II) 1800 61 2 1 0 (II) 3900 3850 3900 293 630		3250	85	126	01	8	0	0	0
(II) 3900 3850 3900 293 630		1800	61	0 3	₩.	0	0	0	0
000 000 000 000 Table		3900	3850	3900	293	630	210	0	0
020 0000 0000 0000 0000 1	A1 (II)	3900	4350	3900	3880	320	320	0	0

Analysis of the Samples for Iron and Aluminium

A. Preparation of the Standard Solutions:

The standard solutions were prepared as follows:

Synthetic Alloy Samples

Various metallic solutions were mixed in certain ratios so that they correspond to the actual metallic proportions in the standard alloys.

Standard Alloy Samples

An accurately weighed amount of the alloy was dissolved in a minimum amount of aquaregia followed by the dilution to a desired volume with DMW.

Rock Samples

Twenty milliliters of 15% NaOH were heated in a Ni-crucible until melted and then fused with 100 mg. of the sample for 5-6 minutes at dull red heat (~600°C). The melt was cooled and 100 ml of DMW was added. After keeping overnight the liquid was transferred to a 1 liter volumetric flask containing 40 ml of 1:1 HCl and the volume made upto the mark with DMW.

B. Separation and Determination of Fe(III) and Al(III)

It was done as follows:

Two grams of the 60-100 mesh sized particles of the ion exchanger in H⁺-form were packed in a glass tube having an internal diameter ~ 0.6 cm and fitted with glass wool at the bottom. The sample solution (1-5 ml) was evaporated to almost dryness to remove the excess acid and the residue was dissolved in a small amount (1-3 ml) of DMW which was then loaded on the column.

TABLE 3

quantitative Separation of Iron/Aluminium from Some Synthetic Alloys Using A-ZAP and ZAS Columns

Mg 945) Mg 870) Mg 870) Mg 870) Mg 870 Mg 945) Mg 870 M	Synthetic alloy and its ml of the solution	d 1		ositic	composition (µg) per	per	Alur obte eff]	Aluminium/iron obtained in ti effluent (wg)	Aluminium/iron obtained in the effluent (ug)	v		% Brror	rror		D 34	% Standard devlation	dard	
Mg 945) Mg 946,1 Mg 947 Mg 947 Mg 947 Mg 948,2							8-Z-	4.		S.	4-2	'AP	Z.	Ŋ	Z-≻	AP	ZAS	S
Mg 945) Mg 945 Mg 945) Mg 945 Mg 945 Mg 945 Mg 945 Mg 945 Mg 945 Mg 946 Mg 970 Mg 9						'	TV V	Fe	A1	Fe	A1	Fe	A1	Fe	A1	Fe	¥1	E.
Mail 120, Mail 10, Mg 870 118.3 - 122.4 1.42 - +2.00 - 0.80 - 1.40 - 1.42 - 1.42 - 1.40 - 0.95 - 1.40 0.95 - 1.40 0.95 - 1.40 0.95 - 1.40 0.95 -	How metal	3	40 Ma	1 5 Mg	945)		41.2	1	40.3	•	+3.00	ı	+0.75	ı	0.50	ı	%.	ı
50) 745 - 7430.670.95 - 1.400.95 - 1.400.95 - 1.160.95 - 2.100.95 - 1.2141.160.95 - 2.100.50 - 41.50 - 0.460.50 - 42.50 - 42.50 - 0.75 - 0.75 - 0.75 - 0.28 - 1.80 - 0.75 - 0.28 - 1.80 - 0.80 - 0.65 - 0.75 - 0.80 - 0.65 - 0.75 - 0.80 - 0.65 - 0.70 - 0.80 - 0.65 - 0.70 - 0.80 -	-0p-	3	120, Mn	10, M	870)	,	118.3	ı	122.4	1	-1.42	ı	+5.00	ı	0.80	•	0.95	ı
995 101.5 -0.95 2.10 - 122.4 123.0 +2.50 -41.50 -0.75 - 122.4 123.0 +2.00 -42.50 -0.75 - 9843.6 847.6 -40.75 -0.28 1.80 - 995.9 904.2 -1.07 -0.28 1.80 - 995.4 918.9 -40.80 -0.65 -0.65 - 995.4 944.9 -40.80 -0.65 -0.65 -0.65 - 995.4 -91.8 -0.29 -40.93 -40.59 -1.03 - 995.1 -0.29 -40.65 -0.15 -0.65 -0.15 -0.66 -0	Aluminium-	3	1	1	_	•	74.5		747	,	-0.67	ŧ	-0.93	1	1.40	,	2.30	,
0) 843.6 847.6 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 122.4 123.0 123.0 123.0 138.2 138	Zine binary allo	53	2 / JU , Z	200		- •	930	1	9.5		-1.16	ı	-0.95	ł	2.10	ı	1.60	ł
5, Sn 5) 122.4	Aluminium bronze	3	100	200		•	96°F	١	101.5	•	-6.50	ı	+1.50	ı	94.0	ı	1.20	,
943.6 - 847.6 - 40.750.28 - 1.80 - 904.2 - 1.01 - 40.47 - 1.30 - 904.2 - 1.01 - 40.47 - 1.30 - 904.2 - 1.01 - 40.47 - 1.30 - 904.2 - 904.20.66 - 0.65 - 0.65 - 0.70 - 946.10.29 - 40.65 - 0.70 - 946.1 - 0.59 - 40.65 - 0.70 - 946.1 - 959.1 - 0.59 - 40.59 - 1.03 - 941.6 - 853.60.87 - 40.59 - 1.03 - 921.82.350.87 - 40.59 - 1.20 - 949.91.050.831.05 -	-do-		120,021	875	Sn 5)	•	122.4	,	123.0	١	+2.00	1	+5.50	١	0.75	ı	1.20	1
0) 890.9 - 904.2 - 1.01 - +0.47 - 1.50 - 1.50 - 918.9 - +0.800.66 - 0.65 - 0.65 - 0.65 - 0.65 - 0.700.56 - 0.70 - 0.70 - 0.57 - 0.55 - 0.56 - 0.57 - 0.57 - 0.55 - 0.56 - 0.57 - 0.57 - 0.55 - 0.55 - 0.57 -	Magnal film	3	850. 36	20	()	•	343.6	١	847.6	ı	+0.75	ı	-0.28	t	1.80	,	2.80	,
(i) 932.4 - 918.9 - +0.800.66 - 0.65 - 0.70 - 946.10.29 - +0.65 - 0.70 - 946.10.29 - +0.65 - 0.70 - 946.10.29 - +0.65 - 0.70 - 944.9 - 959.1 - 0.54 - +0.93 - +0.95 - 1.03 - 921.8 - 921.82.350.87 - +0.59 - 0.66 - 0.66 - 0.65 - 0.66 - 0.66 - 0.68 - 0.66 - 0.68	-00-	3	900	100		•	900.6	ı	904.2	•	-1.01	ı	40.47	1	1.30	í	0.30	ı
(i) Min 10, Cu 50) 937.3 — 946.1 — -0.29 — +0.65 — 0.170 — Mig 6, Cu 40) 944.9 — 959.1 — -0.54 — +0.95 — 0.15 — 1.05 — 0.15 — 0.54 — 1.05 — 0.15 — 0.15 — 0.15 — 0.15 — 0.15 — 0.15 — 0.15 — 0.15 — 0.10 — 0.	1 000	3	925, Mg	75)			932.4	•	918.9	1	10 .80	ı	99.0-	1	0.65	ı	0.34	ı
Mg 6,cu 40) 944.9 - 959.10.54 - +0.95 - 0.12 - 0.05 -	Y-alloy	3	940,Nt	, S	n 10,Cu	_	937.3	•	946.1	ŧ	-0.29	•	0.65	1	0.70	1	94.0	ı
00) 841.6 857.5 40.95 40.59 60.90 921.82.350.87 40.59 60 921.82.350.87 60 921.81.051.05 60 949.91.05 60 949.01.05 60 949.01.05 60 949.01.05 60 9	Duralumin	₹	950.Mn	5. K	9°C u 4C		6.446	ı	959.1	ŧ	-0.54			,		, ,	0.42	, 8
00)	Manganese steel	Fe	879.Mm	120)				887.2	•	884.2	•	40.93	1	+0.59	•	1.03	ı	\$ i
, in 18)	100-	E.	849. M	150			1	841.6	ı	853.6	•	-0.87	•	+0.54	ŧ	1.20	ı	0.28
, (cr 15)	Nickel steel	<u>ج</u>	944 N.	76.M	•			921.8	•	•	1	-2.35	ı	•	ı	0.00	,	ı
0, Mn 19.5) - 949.91.05 -	Chrome ateal	٤	965 X	19	r 15)		ı	957.0	ı	ı	,	-0.83	ı	:	ı	0.85	ı	ı
0, Mn 5(0 19.5) - 609.8 1.65		9	960 Mm	10,0	r 20)		1	6.646	1	2	ı	-1.05	ı	•	,	0.30	,	ı
less steel (Fe 754,N1 80,Mn 5,Gr 180) - 730.50.480.670.67	Inver	, E	620.Nt	360	Mn 19.5		ı	8.609	•	1	•	-1.65	•	ı	ı	1.40	1	ı
10.01 = -0.67 = -0.67 = -0.67	Stainless steel	9	734 NI		n 5.Cr	(80)	1	730.5	,	ı	ŧ	-0.48	•	1	1	1.98	ı	•
(Fe 120,NI /20,MI 20,CI 110) - 113.2	Nichrome	E e	120,Nt		Mn 20,0	110)	ı	119.2	١	ı	1	-0.67	i	i	ı	0.21	1	ı

* Average value of four replicates.

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Quantitative Separation of Iron from Some Standard Steel Samples Using & -ZAP Columns

Steel analysed	Volume of the stock	Elements proceedings of the composition	ts presentition of	ot as the g	per the steel	e standard	Jard	Iron deter- mined	% Error	% Stan- dard devia-
	tion loaded (M1)	₽	r F	NI	n O	₩.	St	in the efflu- efflu- ent (µg)		u011
AISI-303*	1.0	222.80	56	26.69	,	4.71	1.57		+3.44	0.17
	2.0	445.60	113	53.38	ı	9.42	3.14		+1.97	46.0
	2.5	557.00		66.725	,	11.775	3.925	266.40	+1.69	0.81
AI SI -347	1.0	219.40	56.52	31.40		4.71	1.57		+2.61	96.0
	2.0	438.80	113.04	62.80	ı	9.45	3.14	446.26	+1.70	1.26
	2.2	548.50	141.30	78.50	,	11.775	3.925		+1,08	0.57
;	3.0	658.20	169.56	94.20	1	14.13	4.71		-4.83	44.0
Incone1-600	1.0	99.99	155.80	769.3	1.6	2.0	į	08.49	-2.70	0.75
Incone1-800	1.0	451.4	209.90 319.8	319.8	5.9	8.1	ŧ	450.20	-0.27	0.58

* A.I.S.I. Standard Steels.

** Huntington Alloy Products, Division of International Nickel Co., U.S.A.

Average value of five replicates.

TABLE 5

quantitative Separation of Aluminium and Iron from Various U.S.G.S. Standard Rocks Using of -ZAP and ZAS Columns

Rock analysed	Standard Al ₂ O ₃ and Fe ₂ O ₃ obticomposition of the rock ed in the effluent for Al and Fe per 5 ml	Al ₂ O ₃ and * in the	Al ₂ O ₃ and Fe ₂ O ₃ obtain- ed * in the effluent (µg)		% Brror	% Standard	% Standard deviation	
	of the stock solu-	~-ZAP	ZAS	d-zap	SYZ	≪-2AP	ZAS	
	(ng)	A1203 Fe2	Al203 Fe205 Al203 Fe203 Al203 Fe203 Al203 Fe205 Al203 Fe205 Al203 Fe203	A1203 Fe203	A1203 Fe203	A1203 Fe203	A1203 Fe20	.
	A1203 Fe203							1
G-2	77.00 13.45	78.00 13.6	80 79.00 18.80	0 +1.30 +2.60	+2.59 +2.60	1.94	1.64	9
AGV-1	85.95 38.90	85.40 39.8	20 87.00 38.50	74.0+ 49.0- (+1,22 -1,02	1.38 1.97	0.94 1.10	0
BHV0-1	68.50 60.00	69.45 61.(00 71.00 59.2	5 +1.39 +1.67	+3.64 -1.25	5.99	1.89	0
BC R-1	68.60 67.05	69.45 69.6	00 69.80 68.10) +1.24 +2.91	+1.74 +1.56	1.75	2.14	0
FCC-1	3.65 41.40	3.57 40.	40 3.57 40.35 3.60 40.70 -2.19 -2.54 -1.36 -1.69) -2.19 -2.54	-1.36 -1.69	6.47	0.68	ľΩ
* Average	Average values of fi	five replicates	tes.					1

All the elements except Al and Fe were eluted out either in DMW or 0.01M HNO3 (max.volume ~ 150 ml). These metals were then leached out with 1M HNO3 (max.volume ~ 100 ml) and determined quantitatively by atomic absorption spectrophotometry. The observations are summarized in Tables 3-5.

RESULTS AND DISCUSSION

The essential feature of these studies is to use inorganic ion exchangers for the analysis of some alloys and silicate rocks. As it is clear from Table 2, the distribution behavior of ZAP is significantly affected on heating. The heated phase of this material (C-ZAP) becomes highly selective for Al (III) and Fe(III). Also, zirconium(IV) arsenosilicate preferentially holds these two ions (10). property of these two ion-exchangers has been successfully utilized for the separation and quantitative determination of Al(III) and Fe(III) in some alloys and silicate rocks. When a solution of these samples (synthetic or real) is passed through the ionexchange column with a very slow rate, only Al(III) and Fe(III) ions are retained and others are completely excluded by the column simply in DMW or 0.01M HNO. They are then eluted out in 1M HNO3. (Tables 3-5). The method is quite simple and requires much less time as compared to the classical methods. Furthermore, quite a large number of samples can be analyzed using a single column because these materials have shown the excellent reproducibility in their ion exchange behavior, and then ion exchange capacity is not affected even after several

recycling processes. Since ZAS has a high Kd value for Ni(II) ions it could not be used for the separation of iron in the nickel containing steels. For such analyses, however, \angle -ZAP is quite suitable (Tables 3,4).

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