Electronic Supplementary Material (ESI) for Journal of Analytical Atomic Spectrometry. This journal is © The Royal Society of Chemistry 2022

Supplementary Information

Rapid Recognition and Quantitative Technique of Niobium Minerals by Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy

Jiangyan Yuan^{1*}, Xiaochun Li³, Yi Chen^{1, 2*}, Zhengguang Zhang⁴, Xiaoguang Li¹, Sridhar Komarneni⁵

¹ State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, P. R. China

² College of Earth and Planetary Sciences, University of Chinese Academy of Sciences, Beijing 100049, P. R. China

³ Key Laboratory of Mineral Resources, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, P. R. China

⁴General Prospecting Institute of China National Administration of Coal Geology, Beijing 100039, P. R. China

⁵Department of Ecosystem Science and Management and Materials Research Institute, 204 Energy and the Environment Laboratory, The Pennsylvania State University, University Park, PA 16802, USA

Corresponding Author Email: yuanjy@mail.iggcas.ac.cn & chenyi@mail.iggcas.ac.cn

Resolution	Single filed magnification	Min grain size	Step pixels	Image dwell time(us)	Second image dwell (us)	Fast EDS acquisition time (s)	Total time(min)
4096	□60	5	96	5	0	0.10	113
4096	$\Box 60$	5	96	5	0	0.07	96
4096	$\Box 60$	5	96	5	0	0.05	94
4096	$\Box 60$	5	96	5	0	0.03	88
4096	$\Box 60$	5	96	5	0	0.01	84
1024	×60	5	6	5	0	0.10	73
2048	×60	5	24	5	0	0.10	58
8192	×60	5	384	5	0	0.10	262
1024	×120	5	24	5	0	0.10	87
2048	×120	5	96	5	0	0.10	100
2048	×60	5	24	5	0	0.03	41
2048	×60	5	24	3	0	0.03	37
2048	×60	5	24	1	0	0.03	33
2048	×60	5	24	1	2	0.03	34
2048	×60	5	24	1	3	0.03	35
2048	×60	5	24	1	5	0.03	37
4096	×60	3	34	1	3	0.03	99
2048	×120	3	34	1	3	0.03	101
2048	×60	7	45	1	3	0.03	24
1024	×60	7	11	1	3	0.03	25
4096	×60	7	180	1	3	0.03	46
4096	×60	10	368	1	3	0.03	35
2048	×60	10	92	1	3	0.03	16
1024	×60	10	23	1	3	0.03	15
1024	×60	15	52	1	3	0.03	8
2048	×60	15	52	1	3	0.03	13

Table S1 Optimum conditions of rapid recognition experiments based on EDS

Table S2 EDS results and reference values for standards					
□Standards	Oxides	EDS results	Reference	SD	
	SrO	13.01	13.15	0.07	
$\Box Sr_{0.5}Ba_{0.5}Nb_2O_6$	BaO	19.55	19.47	0.09	
	Nb_2O_5	67.57	67.94	0.26	
□KNbO ₃	K ₂ O	25.74	25.89	0.11	
	Nb ₂ O ₅	75.23	74.10	0.43	

Table S3 EDS results and EPMA values for niobium minerals at the same locations of the slice sample

		F	Na ₂ O	CaO	TiO ₂	Nb_2O_5	Ce ₂ O ₃	Nd_2O_3	FeO	SrO	BaO	SiO ₂	Eu_2O_3	Y_2O_3
Pyrochlore	EDS	4.36	6.89	12.30	4.56	63.91	4.04	2.29	1.02	0.16	0.47	-	-	-
	EPMA	4.67	7.21	12.14	4.59	63.91	3.75	2.88	0.48	0.30	0.07	-	-	-
Aeschynite	EDS	-	-	7.00	16.56	39.06	2.35	5.64	4.09	-	-	4.97	0.70	2.84
	EMPA	-	-	6.84	16.67	39.11	2.02	5.62	3.73	-	-	4.97	0.89	2.48

 Table S4 Quantitative analysis error requirement of SEM-EDS (GBT 25189-2010)

W/~i~h+0/	Accepted RDEV/%	
weight70	alloys	minerals
wt%>20%	2	5
3%≤w <i>t</i> % ≤20%	5	10
$0.5\% \le wt\% \le 1\%$	15	20
$0.5\% \le wt\% \le 1\%$	20	30
0.1%≤ <i>wt</i> % ≤0.5%	30	50

PC	Eigenvalue	% variance		
1	163.945	76.1090		
2	27.5879	12.8070		
3	10.1021	4.68980		
4	4.52476	2.10060		
5	3.41765	1.58660		
6	1.69288	0.78590		
7	0.94468	0.43856		
8	0.70903	0.32916		
9	0.65381	0.30352		
10	0.39697	0.18429		
11	0.34289	0.15918		
12	0.25426	0.11804		
13	0.22593	0.10489		
14	0.20398	0.09470		
15	0.12457	0.05783		
16	0.10470	0.04861		
17	0.06159	0.02859		
18	0.04492	0.02086		
19	0.03882	0.01802		
20	0.02987	0.01387		
21	0.00079	0.00037		

Table S5 The eigenvalue and variance using PCA method based on the composition data of niobium minerals



Figure S1 The EDS spectrum of Nb-mineral obtained at the time of 0.03s