Scheme for high efficiency determination of critical materials elements

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Contents

I. Current Research Status

- ◆Method research is relatively laggard
- ◆Overuse chemical reagents
- ◆Single element analysis instruments
- ◆Lengthy procedure, Low efficiency

I. Current Research Status

Silver diethyl dithiocarbamate photometric :As

FAAS:Bi

Polarograph:Sn

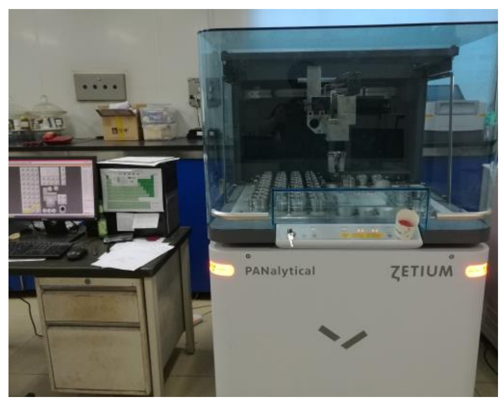
GB/T 14352-2010 tungsten,molybdenum ores

I. Current Research Status

- International popular
- High sensitivity
- Good precision
- Simultaneous
- multi-element analysis

ICP-MS

ICP-AES



XRF

Microwave digestion

II. Scheme for determination of critical materials elements

No.	Method Name	Sample digestion	Solved problem	Determination elements
1	Rare earth ores， ICP-AES, ICP-MS	mixed acid (in open vessels, with micro-wave device, or in pressurized bomb)	Determining major elements, and rare, scattered and rare earth elements. By specific resin separating, the MS interferences between REE can be eliminated effectively	Al, Fe, Ca, Mg, K, Na, Ti, Mn, P, REEs, Li, Be, Sc, Cr, Co, Ni, Cu, Zn, Ga, Rb, Nb, Mo, In, Cs, Ta, W, Tl, Pb, Bi, Th, U, Y
2	Beryllium tantalum and niobium ores etc，ICP-AES, ICP-MS	pressurized bomb or micro-wave device, preparing as HNO_3 or HF solution	HF media can prevent the hydrolyzation of elements like W, Nb, Ta in W and Nb-Ta ores. Can determine W ore samples with W content above 50%.	Al, Fe, Ca, Mg, K, Na, Ti, Mn, P, Li, Be, Sc, Cr, Co, Ni, Cu, Zn, Ga, Rb, Nb, Mo, In, Cs, Ta, W, Tl, Pb, Bi, Th, U, Y, REEs
3	Lithium metaborate Fusion Sample Pretreatment， ICP-AES, ICP-MS	$LiBO_2$ fusion digesting, extracting with 5% aqua regia and preparing as solution	Determining petrogenetic elements and rare, scattered and rare earth elements simultaneously	Si, Al, Fe, Ca, Mg, K, Na, Ti, Mn, P, REEs, Co, Sr, In, Ba, Th, Nb, Ta, Zr, Hf, Ti
4	Mixed acid digestion for Sulfide ore，ICP-AES, ICP-MS	HNO_3-HF in the pressurized bomb; extracting with HNO_3 and preparing as solution;Or HNO_3-HCl-HF-$HClO_4$; extracting with aqua regia and preparing as solution	Determining major and trace elements in the sulfide ores	Al, Fe, Ca, Mg, K, Na, Li, Be, Sc, Ti, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Sb, Te, Rb, Mo, Ag, Cd, In, Sn, Cs, REEs, W, Tl, Pb, Bi, Th, U
5	aqua regia digestion for Sulfide ore， ICP-MS	aqua regia and preparing as solution	Suitable for determining various sulfide ore minerals	As, Ag, Cd, Hg, In, Bi
6	Sodium carbonate alkali fusion for Celestine，ICP-AES , ICP-MS	Digesting $SrSO_4$ and $BaSO_4$ through the displacement reaction. After filtrating, dissolve the precipitate with HCl into solution	Replacing the gravimetric method to determining Sr and Ba in the celestite	Sr, Ba , REEs, Nb, Ta, Zr, Hf et. al.
7	Alkali fusion for bauxite， ICP-MS	Na_2O_2; extracting with HCl and preparing as solution	Determining trace elements in the bauxite	Ga, Ge, Th, U, REEs et. al.
8	Mixed acid digestion for ore samples， AAS, AFS	mixed acid, preparing as HCl solution	AAS method can eliminate the interferences from metal ions can form the hydride; suitable for various rock and ore; Separating by the microchromatography column and determining AFS with can eliminate interference elements, suitable for various samples such as Cu concentrate	Se

III. Examples of typical ore sample analysis methods

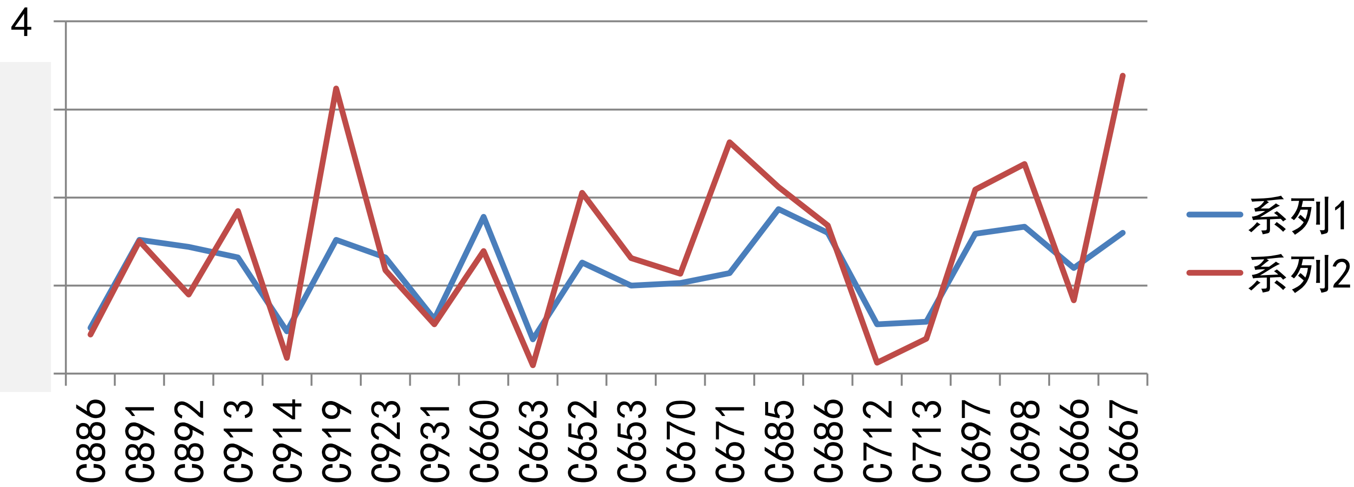
(1) Rapid qualitative and quantitative analysis for ion adsorption type REE ores in the field.

Primary purpose

Whether it is? Qualitative	How about the raw ore grade? Quantitative	Rapid screening in the field	Technical support to governmental management

Comparison between field and indoor accurate analysis

- **Easy to carry,<2kg**
- **Simple operation ,<20min**
- **Economical and practical， <200 thousand**



Applied National invention patents

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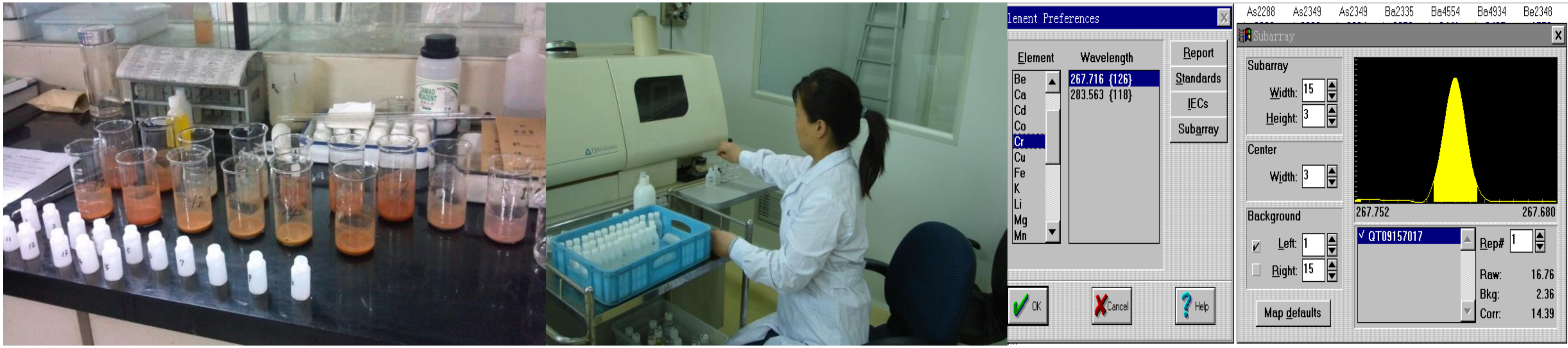
(2) Individual REE quantitative analysis method study and standard reference material development for the ion absorption REE samples

Primary purpose：
Quantitative evaluation for each REES



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Immersion extraction experiment study of individual REE for the ion absorption REE samples

01 Leaching experiment conditions

4.0 g sample, 2.5 % $(NH4)_2SO_4$ as the leachate, solid-to-liquid ratio of 1:8

02 Experiment procedure

Weight 4.0 g sample, add 32 mL 2.5% $(NH4)_2SO_4$ solution, shake and then stand for 24 h. Take 1 mL supernatant, add 9 mL 5% HNO_3, and determine the REE content in the solution. The amount of blank samples in a batch is no less than 2.

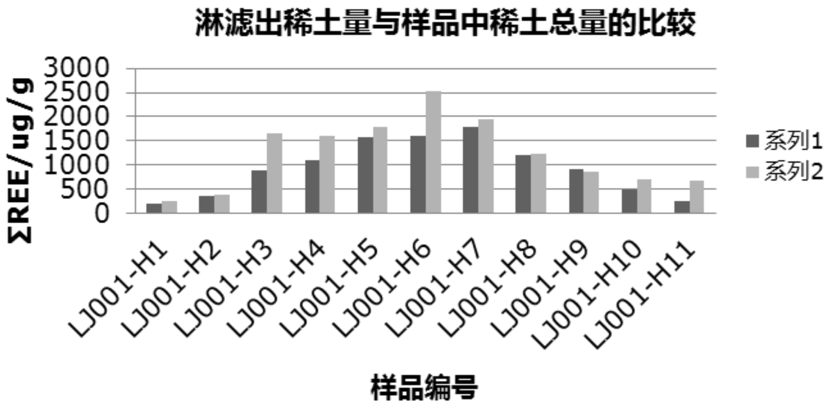

Fig. Comparison between REE content leached out and total REE content
Series 1: REE content leached out; Series 2: total REE content in the sample

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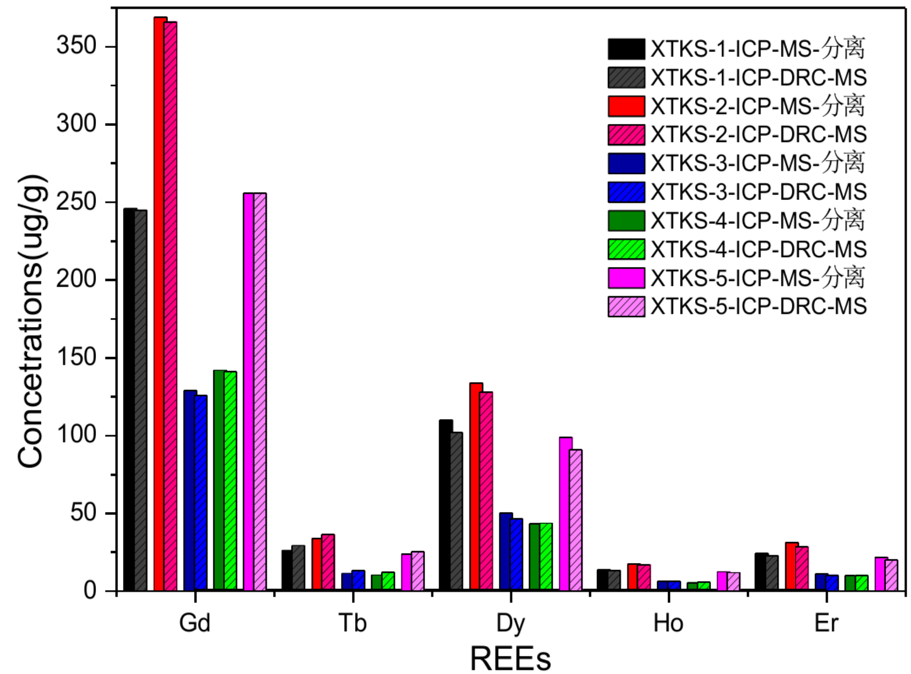
(3) Accurate analysis of REE contents in REE ores, tailings, concentrates and products

content difference between LREE and HREE is huge, LREE(Ce, Pr, Nd and Sm) oxide interference to Gd, Tb, Dy and Er will be very severe

样品	Gd				Tb			
	ICP-MS	ICP-MS -扣干扰	ICP-DRC -MS	ICP-MS -分离	ICP-MS	ICP-MS -扣干扰	ICP-DRC -MS	ICP-MS -分离
XTKS-1	1316	279	245	246	58.4	21.4	29.4	26.2
XTKS-2	1423	367	366	369	66.7	24.9	36.5	34.1
XTKS-3	481	86.3	126	129	24.2	10.4	13.2	11.4
XTKS-4	527	92.4	141	142	31.3	9.24	12.3	10.4
XTKS-5	1084	356	256	256	49.6	18.4	25.6	23.7

样品	Dy				Ho		
	ICP-MS	ICP-MS -扣干扰	ICP-DRC -MS	ICP-MS -分离	ICP-MS	ICP-DRC -MS	ICP-MS -分离
XTKS-1	150	95.9	102	110	13.6	13.4	13.8
XTKS-2	182	121	128	134	16.5	17.1	17.6
XTKS-3	66.7	46.6	46.7	50.3	6.10	6.36	6.33
XTKS-4	74.3	42.0	43.8	43.3	5.83	5.89	5.51
XTKS-5	133	87.1	90.9	99.0	11.5	12.0	12.5

样品	Er				Tm		
	ICP-MS	ICP-MS -扣干扰	ICP-DRC -MS	ICP-MS -分离	ICP-MS	ICP-DRC -MS	ICP-MS -分离
XTKS-1	67.2	20.8	22.8	24.3	1.83	2.06	2.05
XTKS-2	77.4	24.9	28.6	31.5	2.28	2.63	2.68
XTKS-3	27.8	10.4	10.3	11.1	0.76	0.88	0.89
XTKS-4	39.8	12.0	10.3	10.1	0.87	1.02	0.92
XTKS-5	60.9	21.6	20.0	21.9	1.56	1.86	1.94



1) Separation with specific resin – Measure with ICP-MS

2) Measure with ICP-MS in DRC Mode

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(4) Simultaneous analysis of critical materials elements in Nb-Ta and W ores

GB/T17415 "Chemical analysis method of Nb and Ta ores", including determination methods of 2 elements

———1. Ta

———2. Nb

Butyl Rhodamine B spectrophotometric method for Ta content determination in Nb and Ta ores

Thiocyanate spectrophotometric method for Nb content determination in Nb and Ta ores

GB/T14352-2010 "Chemical analysis method of W and Mo ores", including determination methods of 18 elements

———1. W
———2. Mo
———3. Cu
———4. Pb
———5. Zn
———6. Cd
———7. Co
———8. Ni
———9. S
———10. As
———11. Bi
———12. Ag
———13. Sn
———14. Ga
———15. Ge
———16. Se
———17. Te
———18. Re

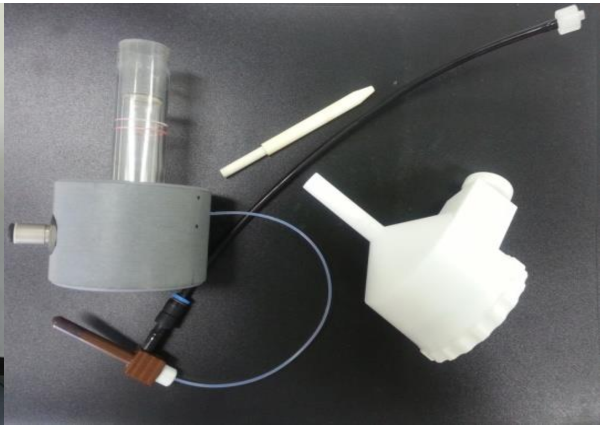
- High-temperature or high-pressure microwave digestion in acid media (HNO_3+HF)
- directly measured by instruments with HF-resistant sample introduction device

Results comparison between different acid media conditions

Acid medias	10%HF,2%HNO_3		5%HF,2%HNO_3		3%HF,2%HNO3	
Elements	Nb	Ta	Nb	Ta	Nb	Ta
GBW 07185-1	3792	8596	3733	8313	3733	8513
GBW 07185-2	3539	8978	3767	8518	3767	8518
GBW 07185-3	3659	8034	3665	8362	3665	8563
Mean	3663	8536	3722	8398	3722	8531
Reference value	3635	8353	3635	8353	3635	8353

Microwave digestion conditions for Nb-Ta ores

Tem.	Time	Power
130℃	15min	1200w
160℃	15min	1200 w
190℃	25min	1200 w



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Technical Features

Sample digestion technique – less reagent consumption

- **High-efficient micro-wave digestion**

HF-resistant sample introduction system – easy operation

- **Effectively avoid the hydrolyzation of Nb, Ta and W**

Wide dynamic range – both raw ore and concentrate can be analyzed

- **The advantage in analyzing high content samples is more prominent**

(4) Simultaneous analysis of critical materials elements in Nb-Ta and W ores

Results of 200.0 mg sample
(2.0 mL+1.0mL HNO3)

		Nb_2O_5	Ta_2O_5	W
GBW07154	Mean (n=8)	43.3	85.9	17.4
	Standard value	42.3+2.5	88.6+6.0	16.4+1.2
	RSD(%)	6.0	5.2	3.1
	RE(%)	2.3	-3.0	6.3
GBW07155	Mean (n=8)	466	684	199
	Standard value	430+30	700+60	200+20
	RSD(%)	6.1		3.2
	RE(%)	8.4	-2.3	-0.6
GBW07185	Mean (n=10)	5288	10444	24.0
	Standard value	5200+100	10200+20	21.4+1.8
	RSD(%)	3.7	1.9	3.3
	RE(%)	1.7	2.4	12.0

Results of different W ore types （%）

Sample	KSCN-$TiCl_3$ spectrophotometry %	ICP-AES %
Wolframite ore	0.13	0.13
Scheelite ore	0.44	0.44
Wolframite concentrate	48.4	48.0
GBW07241	0.22	0.22
GBW07284	3.67	3.67

Thanks for your attention !