DETERMINATION OF REE IN HIGH- PURE Y_2O_3 BY NAA

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ABSTRACT

The method used is divided into two parts. One part is the determination of La, Ce, Pr and Sm by pretreatment neutron activation analysis. The other part is the determination of Ce, Eu, Tb, Tm, Yb and Lu by direct instrument neutron activation analysis. Pretreatment of samples was carried out by P_{507} extraction chromatography. The decontamination factor of Y was 2×10^4. A sensitive method for determination of REE in high- pure Y_2O_3 was established.

Keywords: REE NAA Extraction chromatography Y_2O_3

1 INTRODUCTION

In this work P_{507} extraction chromatography[1-6] was used for separating rare- earth elements (REE) from Y_2O_3. The separated REE were then collected and irradiated. Ten impurities of REE in high- pure Y_2O_3 have been determined.

2 EXPERIMENTAL

2.1 Determination of La, Ce, Pr and Sm in high- pure Y_2O_3 by pretreatment NAA

2.1.1 *Preparation of samples* About 100 mg Y_2O_3 dried at 100 °C for 4h was weighed and dissolved in diluted HCl. The solution was heated until nearly dry. The residue was dissolved in 10 ml 0.4 mol/l HCl and quantitatively transferred to a P_{507} column for adsorption of REE. Fourty ml eluate was collected from the column with 0.7 mol/l HCl and concentrated, then a little $HClO_4$ was added and heated for removing bromine. At last the residue was dissolved in diluted HCl and transferred onto Xinhua quantitative filter paper and dried at 80 °C. Then it was wrapped with a pure aluminium foil for irradiation.

2.1.2 *Preparation of standards* The standard solutions of La, Ce, Pr and Sm were added onto Xinhua quantitative filter paper and dried at 80 °C. Then they were wrapped with a pure aluminium foil for irradiation.

2.1.3 *Irradiation* The samples and standards were packed together, irradiated with a neutron flux of $3\text{-}5\times 10^{13}\ n \cdot cm^{-2} \cdot s^{-1}$ for 5- 6 h and cooled in 3- 4 d.

2.1.4 *Measurements* The activities of standards and samples were measured by a multichannel analyzer CABBERRA. The measuring time was 300- 500 s for standards and 3600 s for samples. The energies used were 1596 keV for ^{140}La, 145 keV for ^{141}Ce, 1576 keV for ^{142}Pr and 103 keV for ^{153}Sm.

2.1.5 *The analytical results of La, Ce, Pr and Sm*

Table 1

The analytical results of La, Ce, Pr and Sm in high– pure Y_2O_3 obtained from pretreatment NAA

ppm

Samples	La		Ce		Pr		Sm	
	added	determined	added	determined	added	determined	added	determined
Y_2O_3 870603	–	0.128	–	0.0860	–	–	–	0.0475
3a=	–	0.159	4.10	3.99	8.28	8.33	–	0.103
6=	–	0.202	8.20	8.00	16.6	15.9	–	0.113

The P_{507} column could be used again after it was washed successively by 6 mol/l HCl, distilled water and 0.7 mol/l HCl.

2.2 Determination of Ce, Eu, Gd, Tb, Tm, Yb and Lu

2.2.1 *The preparation of samples* About 200 mg Y_2O_3 dried at 100 °C for 4 h was weighed and wrapped in a pure aluminium foil for irradiation.

2.2.2 *Preparation of standards* The standard solutions of Ce, Eu, Gd, Tb, Tm, Yb and Lu were added to Xinhua quantitative filter paper and dried at 80 °C. They were wrapped with a pure aluminium foil for irradiation

2.2.3 *Irradiation* The standards and samples were packed together, irradiated with a neutron flux of $3-5\times10^{13}$ n · cm^{-2} · s^{-1} for 15–20 h and cooled in one month.

2.2.4 *Measurements* The samples and standards were measured directly by S–90 (CANBERRA Series 90). The measuring time was 3600–7200 s for samples and 300–500 s for standards. The energies used were 145 keV for ^{141}Ce, 1408 keV for ^{152}Eu, 103 keV for ^{153}Gd, 879 keV for ^{160}Tb, 84 keV for ^{170}Tm, 177 keV for ^{169}Yb and 208 keV for ^{177}Yb.

2.2.5 *The analytical results of Ce, Eu, Gd, Tb, Tm, Yb and Lu in high– pure Y_2O_3*

Table 2

The analytical results of Ce, Eu, Gd, Tb, Tm, Yb and Lu in high– pure Y_2O_3 determined by INAA

ppm

Samples	Ce		Eu	Gd	Tm	Yb	Lu	Tb	
	added	determined						added	determined
Y_2O_3 870603	–	0.0826	0.0106	0.381	0.137	1.18	$\leq$0.200	–	0.199
3b=	8.20	8.08	0.0439	0.497	0.184	1.40	0.223	17.0	17.3
6=	8.20	8.27	0.0417	0.550	0.163	1.51	0.235	17.0	17.4
5=	4.10	4.13	0.0388	0.409	0.238	1.43	0.230	8.51	8.94
10=	4.10	4.17	0.0429	0.594	0.194	1.47	0.225	8.51	8.68

3 DISCUSSION

3.1 Elution curve and yield

The elution curves of La, Ce, Pr, Sm, Eu, Yb and Y were made by radioisotope tracers.

The La, Ce, Pr, Sm and Eu were quantitatively eluted in 40 ml eluate. The Y appeared after receiving 80 ml eluate. The elution order was that the light rare-earth elements appeared earlier than the heavy ones. La appeared first and Tb last. The chemical yields of rare-earth elements were 96-100%. The decontamination factor of Y was 2×10^4

3.2 Reclaimed experiment

Definite amounts of Ce, Pr and Tb were added into 99.999% Y_2O_3 and determined by above methods. The results indicate that not only pretreatment NAA but also INAA can be used to determine Ce, Pr and Tb quantitatively.

The results from the experiment with adding definite amounts of Tb and Ce showed that the two methods were comparable each other, the variance between the two methods was less than 5%.

3.3 Detection limits

The detection limit of each element in above mentioned irradiating and cooling conditions is showed in Table 3.

Table 3

The detection limits of rare— earth elements

ppm

Methods	La	Ce	Pr	Sm	Eu	Gd	Tb	Tm	Yb	Lu
Pretreatment NAA	0.021	–	1.3	0.0033	–	–	–	–	–	–
INAA	–	0.050	–	–	0.0048	0.17	0.0033	0.040	0.37	0.20

The results show that the chemical yields for separation of rare-earth elements are high and the detection limits are low. The detection limit for total rare-earth elements, except for Pr, is 0.912 ppm. This method is reliable and can be used to determine rare-earth elements in 99.9999% Y_2O_3.

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