# DETERMINATION OF RARE EARTH AND OTHER ELEMENTS IN YEN-PHU RARE EARTH ORE AND OTHER INTERMEDIATE PRODUCTS FROM THE FLOATATION AND HYDROMETALLURGICAL PROCESS ON PORTABLE XRF Si-PIN DETECTOR

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***ABSTRACT:*** The concentration of rare earths elements such as La, Ce, Pr, Nd, Gd... and other elements as Ca, Fe, U, Th in Yen Phu rare earth ore and other intermediate products from the flotation and hydrometallurgical process was determined by using Si-PIN detector fluorescence spectrometry. The precision and accuracy of quantitative analysis was tested by standard reference materials and comparative analysis with different analytical methods. The analytical procedures were set-up and applied for the determination of rare earth and other elements in Yen Phu rare earth ore and other intermediate products from the flotation and hydrometallurgical process with high precision and accuracy.

## I. INTRODUCTION

Previously ICP methods were often used to analyze the rare earth elements, however the disadvantage of this method of analysis was time-consuming because it needed to digest the solid samples. In published articles written about the identification of rare earth elements by X-ray fluorescence method, the wavelength dispersion fluorescence systems (WD-XRF) were almost applied. In ITRRE, there was a report concerning energy dispersion fluorescence system (ED-XRF) from the year of 2000, in which some rare earth elements belonged to the "light group" was determined. However, the detector of that (ED-XRF) system was semiconductive Si(Li), and the number of rare earth elements determined has been limited. The energy dispersion fluorescence system (ED-XRF) with Si-PIN detector for direct analysis of individual rare earth elements was thus employed for the study of La, Ce, Pr, Nd, Ca, Fe, Th, U determination in Yen Phu rare earth ores with the aiming supplying the demands of rare earth ore beneficiation. The procedure was established with the low error and high precision.

## II. EXPERIMENTAL

1. Investigation of the current, voltage of X ray generator to obtain the characteristic X-ray with highest intensity of elements La, Ce, Pr, Nd, Ca, Fe, Th, U in Yen Phu rare earth ore.

**Table 1:** Dependence of characteristic X line intensity on X-ray tube current.

| No | Elements | Characteristic X-ray intensity at the voltage U = 30 kV and different X-ray tube current | | | | |
|---|---|---|---|---|---|---|
| | | I=5 μA | I=10 μA | I=15 μA | I=20 μA | I=25 μA |
| 1 | *Ca* | 4096 | 5054 | 5389 | 4903 | 4260 |
| 2 | Fe | 336524 | 461749 | 471320 | 434574 | 384631 |
| 3 | La | 5863 | 7733 | 7992 | 7245 | 6630 |
| 4 | Ce | 14343 | 18395 | 19217 | 17423 | 15003 |
| 7 | Ag-IN | 7300 | 11152 | 14597 | 17780 | 19480 |
| 8 | Ag-CO | 1446 | 2215 | 2966 | 3717 | 3917 |
| (Dead time )% | | 16.88 | 30.56 | 41.98 | 51.69 | 59.29 |

The voltage of X ray generator by 30 kV and X-ray tube current by 10 μA was chosen when analyzed of rare earths and other elements in Yen Phu rare earth ore and other intermediate products from the flotation and hydrometallurgical process on the portable XRF Si-PIN detector.

2. Quantitatively analysis methods for the determination of rare earth and other elements in Yen Phu rare earth ore and other intermediate products from the flotation and hydrometallurgical process on the portable XRF Si-PIN detector.

***Fundamental parameter method***

*Fundamental parameter method in QXAS software*

Fundamental parameter is the most versatile method for quantification in the QXAS package and suited even for completely unknown samples. Practically all modes of excitation with electromagnetic radiation in the range of X-rays can be covered and many more parameters can be selected to match the assumptions needed for the calculations with the experiment.

The sample self-absorption was corrected by the use of scatter peaks such as Compton and Rayleigh. The specially characteristic when calculating using fundamental parameters method are some factors such as absorption and enhancement correction are taken into consideration.

3. Analysis in the comparison with Standard Reference Materials and other methods.

Results received when analyses of the Yen Phu Rare earths ore by X-ray fluorescence Si-PIN detector using fundamental parameter method compare with the one when analyses by ICP-MS method.

**Table 2:** Analysis of Yen Phu Rare earth Ore by XRF and ICP-MS method.

| No | Element | Unit | Results (%) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | *YA-XRF* | *YA-ICPMS* | *Relative Error (%)* | *YB-XRF* | *YB-ICPMS* | *Relative Error (%)* | *YC-XRF* | *YC-ICPMS* | *Relative Error (%)* |
| 1 | Ca | % | 0.8 | 0.77 | 3.90 | 0.56 | 0.51 | 9.80 | 0.47 | 0.43 | 9.30 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2 | Fe | % | 9.72 | 9.54 | 1.89 | 8.78 | 9.1 | 3,52 | 8.08 | 7.57 | 6.74 |
| 3 | Y | % | 5.794 | 5.31 | 9.11 | 5.94 | 5.57 | 6.70 | 6.834 | 6.40 | 6.78 |
| 4 | La | % | 1.212 | 1.13 | 7.26 | 1.24 | 1.13 | 9.65 | 1.342 | 1.23 | 9.11 |
| 5 | Ce | % | 2.468 | 2.35 | 5.02 | 2.57 | 2.37 | 8.57 | 2.824 | 2.61 | 8.20 |
| 6 | Pr | % | 0.467 | 0.47 | 0.64 | 0.46 | 0.47 | 2.34 | 0.499 | 0.51 | 2.16 |
| 7 | Nd | % | 2.324 | 2.12 | 9.62 | 2.05 | 2.10 | 2.43 | 2.487 | 2.26 | 10.04 |
| 8 | Sm | % | 0.577 | 0.54 | 6.85 | 0.61 | 0.54 | 12.96 | 0.616 | 0.58 | 6.21 |
| 9 | Gd | % | 0.736 | 0.66 | 11.52 | 0.74 | 0.67 | 13.28 | 0.715 | 0.76 | 5.92 |
| 10 | Dy | % | 0.867 | 0.84 | 3.21 | 0.84 | 0.87 | 2.99 | 1.043 | 1.01 | 3.27 |
| 11 | Er | % | 0.58 | 0.54 | 7.41 | 0.61 | 0.55 | 10.91 | 0.725 | 0.66 | 9.85 |
| 12 | Yb | % | 0.48 | 0.42 | 14.29 | 0.47 | 0.44 | 6.82 | 0.565 | 0.52 | 8.65 |
| 13 | Th | % | 0.286 | 0.27 | 5.93 | 0.41 | 0.39 | 5.13 | 0.37 | 0.40 | 7.50 |
| 14 | U | % | 0.044 | 0.04 | 10.00 | 0.05 | 0.04 | 6.82 | 0.048 | 0.044 | 9.09 |

From the table, it can be found that resulted analysis is reliable compare with reference material standard. The Relative Error bethwen compared methods are less then 10%.

**Table 3:** Analysis of a rare earth samples (M1) by Si-PIN fluorescence in comparison with results received from Japan laboratory (ICP-OES).

| **Elements** | **Unit** | **Results (%)** | | |
|---|---|---|---|---|
| | | *XRF* | *Japan* | *Relative Error (%)* |
| **Ca** | % | 4.49 | 4.4 | 2.00 |
| **Mn** | % | 1.10 | 1.18 | 7.27 |
| **Fe** | % | 3.04 | 3.54 | 9.26 |
| **Y** | % | 0.23 | 0.22 | 4.35 |
| **Ba** | % | 6.18 | 6.55 | 5.99 |
| **La** | % | 11.20 | 11.53 | 2.95 |
| **Ce** | % | 14.94 | 15.8 | 5.76 |
| **Pr** | % | 1.32 | 1.58 | 7.48 |
| **Nd** | % | 4.50 | 4.59 | 2.00 |
| **Sm** | % | 0.48 | 0.47 | 2.08 |
| **Th** | % | 0.1066 | 0.114 | 6.94 |
| **U** | % | 0.061 | 0.059 | 3.28 |

From the table 3, it can be found that the diffence between analytical result of M1 rare earth sample, which was done by Si-PIN X-ray fluorescence and that obtained in Japan laboratory (ICP-MS) are less then 10%.

## III. CONCLUSION

Project has solved the following issues

1. The technical parameter of Si-PIN spectrometry was investigated. The studies found that when the voltage of X ray generator by 30kV and X-ray tube current by 10 μA was chosen will give the characteristic X-ray with the highest intensity.

2. The selection of X-ray line fluorescence and the fitting of spectrum was performed when analyzed of rare earths and other elements in Yen Phu rare earth ore and other intermediate products from the flotation and hydrometallurgical process on the portable XRF Si-PIN detector.

3. Some quantitatively analysis methods used in X-ray fluorescence such as calibration curve and fundamental parameter was studied. The detection limit of rare earth elements such as La, Ce, Pr, Nd... was received.

## REFERENCES

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