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## A Rare Earth Oxides-Containing Internal Reference Material of Purified Monazite from Bangka Island

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### ABSTRACTS

Monazite mineral contains a sufficient composition of rare earth elements which are currently required widely in modern industries. Reference materials are needed to validate the measurement results, including the rare earth elements analysis. This study presents the processing of purified monazite from the PT Timah Metallurgical Unit in Muntok to become low-cost and rare earth oxides-containing internal reference material. Eight X-ray fluorescence measurements of four splits of the monazite were done for precision test and to establish its information values. The high CeO<sub>2</sub> and LaO<sub>2</sub> composition (>10%) implies the economic worth of the studied sample. Based on the acceptance criteria of  $RSD_{\text{experimental}} < 10\%$ ,  $RSD_{\text{experimental}} \leq 66\% \times CV_{\text{Horwitz}}$ , and Horrat <2, the concentration of fourteen analytes is acceptable to be set as information values. The studied monazite content resembles the other purified one of Myanmar. Its much lower phosphate composition signifies that the sample is more precious than refined monazites from Iran and Australia.

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### INTRODUCTION

Reference material is an important tool to confirm the validity of measurement procedures and results. Certified Reference Materials (CRMs) are the most preferable as they have been prepared following stringent guidelines and by proper international certifying bodies [1], [2]. However, CRMs are limited, high cost, and could not cover the large type of samples. Internal reference materials developed by a laboratory for its internal use have been produced in enabling CRM's to be used on a less frequent basis and reducing measurement cost [3], [4]. The internal reference material might be upgraded to a national

reference material after analyzed by other certified laboratories and guaranteed by the country's agency.

Rare earth elements which consist of the lanthanides group plus Scandium and Yttrium is required on modern devices fabrication. The group of elements is demanded in manufacturing permanent magnet, air craft, sensors, rechargeable battery, catalyst, and magnetic resonance imaging sensor (MRI) [5], [6], [7]. Monazite, xenotime, bastnasite, eudialyte, allanite, zircon, and apatite are the REE-bearing minerals but the first three are the major minerals currently exploited [8], [9], [10]. Monazites are found in igneous, sedimentary, and metamorphic rocks as accessory minerals. Monazite is the widely

### ARTICLE INFO

#### Article History:

Received 25 Jun 2021

Revised 09 Dec 2021

Accepted 14 Dec 2021

#### Keyword:

Monazite,

Internal reference material,

XRF,

REE.

known as Light-REE bearing mineral often present in residue and placer associated with tin mining activity in Malaysia and Indonesia [11], [12], [13], [14].

Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) and Instrumental Neutron Activation Analysis (INAA) are the most frequently used devices for REE analysis [10], [15], [16], [17], [18]. ICP-MS analysis takes a shorter time and has better detection limits but require a more complicated sample digestion procedure than INAA. Acid digestions which transformed solid sample into liquid are required in the ICP-MS preparation procedure. However, as the ore-grade monazite is a very stable phosphate mineral, it is difficult to dissolve such material in acids or alkali [19], [20]. Several wasting time and cost additional preparation steps required in measuring the chemical composition of monazite through ICP-MS method. On the other hand, X-Ray Spectrometry (XRF) is a majorly simple and effective method for analyzing samples with high element oxides content. Those factors should be the reasons for utilizing XRF in previous monazite studies [13], [20], [21], [22]. This study explains the processing of purified monazite from the PT Timah Metallurgical Unit in Muntok (Bangka Island) as an internal reference material using XRF. Chemical character of the studied material is then also compared to the other monazites from several countries.

## MATERIALS AND METHODS

### A. Sample Description

PT Timah Metallurgy Unit was built to streamline the cost of tin metal production, reduce the amount of the remaining results of the smelting, and refining process. The plant which located in Muntok, at the western of Bangka Island, is ISO9001:2008, ISO14001:2004, and OHSAS18001:2007 certified. The studied sample is pale brown and -200 mesh monazite powder from PT Timah Metallurgical Unit in Muntok as shown in **Figure 1a**. The input of this unit is the tin tailings from both offshore and onshore tin mining activities. The offshore fleet consists of eleven dredgers and ten production suction vessels which worked in waters around Bangka and Belitung Islands while the minings in the area of land were held in several locations. Zigzag, electrostatic, and magnetic separation methods are applied to purified monazite from the tin residues. According to previous studies, zigzag instrumentation separates the light and heavy fraction of the raw material whilst the electrostatic apparatus detaches the Ti-rich minerals [8], [22]. Separating monazite and iron oxide-silicate material is usually done by magnetic method [8], [14], [23], [24], [25].

The monazite was brought to the Laboratory of Center for Geological Survey in Bandung to be prepared and analyzed using XRF. No homogeneity

preparation was needed as this work should have been done in PT Timah Metallurgical Unit at Muntok. The samples were directly divided into splits and stored in glass containers as shown in **Figure 1b**. Metal containers which possibly cause inhomogeneity during storage due to magnetism should be avoided. A total of eight samples consisting of two sub-samples from the four split containers were weighed and prepared according to the procedure of previous study [26]. The pressed pellet method was adapted in this study on behalf of flexibility and preventing particle size effect [1], [4], [26]. Pellet thickness should be not less than the stainless steel ring in avoiding incorrect detection of X-ray radiation.



(a)



(b)

**Figure 1.** a) The pale brown and -200 mesh monazite powder from Muntok as the studied material; and b) The monazite is split and stored in glass containers to prevent inhomogeneity.

### B. Precision Test

Precision depicts the sample homogeneity in which the high degree results in even distribution of analytes and particles in all parts of the sample so that it would be detected almost the same through repeated measurements [3], [27], [28]. The precision degree of any analytes could be drawn by comparing the relative

standard deviation (RSD) which is also known as the coefficient of variance (CV) to the  $CV_{Horwitz}$  value [1], [3], [26], [29]. RSD of measurements (n) is the coefficient of variation and calculated by dividing the standard deviation (SD) to the average value ( $X_R$ ) as described in Equation (1) through Equation (3) below:

$$X_R = \frac{\sum X}{n} \quad (1)$$

$$SD = \left( \frac{\sum (X - X_R)^2}{(n-1)} \right)^{0.5} \quad (2)$$

$$RSD = \frac{SD}{X_R} \quad (3)$$

$CV_{Horwitz}$  is the coefficient of variability Horwitz as RSD prediction and obtained using Horwitz function in Equation (4). The Horwitz ratio (Horrat) value is calculated by dividing the RSD of the eight subsamples measurement by  $CV_{Horwitz}$  as described in Equation (5).

$$CV_{Horwitz} = 2^{1-(0.5 \times \log X_R)} \quad (4)$$

**Table 1.** The measurement results of studied monazite chemical composition using XRF.

Analytes	Split 1		Split 2		Split 3		Split 4		RSD <sub>exp.</sub>	66% CV <sub>Horwitz</sub>	Horrat
	A	B	A	B	A	B	A	B			
<i>Concentration in wt%</i>											
CeO <sub>2</sub>	27.77	28.04	28.01	28.02	27.80	27.82	28.07	27.96	0.43	0.81	0.36
Ce	22.60	22.82	22.80	22.81	22.63	22.64	22.85	22.76	0.43	0.83	0.34
P <sub>2</sub> O <sub>5</sub>	20.09	20.11	20.06	20.14	20.05	19.98	19.85	20.02	0.45	0.85	0.36
P	8.77	8.78	8.76	8.79	8.75	8.72	8.67	8.74	0.45	0.96	0.31
La <sub>2</sub> O <sub>3</sub>	12.96	12.90	12.86	12.93	12.95	12.91	13.00	12.92	0.33	0.91	0.24
La	11.05	11.00	10.97	11.03	11.04	11.01	11.09	11.02	0.33	0.93	0.23
Nd <sub>2</sub> O <sub>3</sub>	10.80	10.86	10.84	10.77	10.91	10.75	10.91	10.87	0.56	0.93	0.40
Nd	9.26	9.31	9.29	9.23	9.35	9.22	9.35	9.32	0.56	0.95	0.39
ThO <sub>2</sub>	7.45	7.44	7.43	7.43	7.44	7.40	7.51	7.43	0.42	0.99	0.29
Th	6.55	6.54	6.53	6.53	6.54	6.50	6.60	6.53	0.42	1.01	0.28
SnO <sub>2</sub>	4.17	4.15	4.22	4.07	4.18	4.28	4.07	4.20	1.72	1.08	1.07
Sn	3.28	3.27	3.32	3.21	3.29	3.37	3.21	3.31	1.72	1.11	1.03
Pr <sub>6</sub> O <sub>11</sub>	2.65	2.62	2.69	2.68	2.66	2.63	2.65	2.54	1.77	1.15	1.02
Pr	2.19	2.17	2.23	2.22	2.20	2.18	2.19	2.10	1.77	1.19	0.99
Y <sub>2</sub> O <sub>3</sub>	2.61	2.60	2.58	2.64	2.60	2.57	2.61	2.62	0.84	1.15	0.49
Y	2.06	2.05	2.03	2.08	2.05	2.02	2.06	2.06	0.84	1.20	0.47
SiO <sub>2</sub>	1.73	1.61	1.62	1.62	1.62	1.65	1.60	1.60	2.63	1.24	1.42
Si	0.81	0.75	0.76	0.76	0.76	0.77	0.75	0.75	2.63	1.39	1.26
Sm <sub>2</sub> O <sub>3</sub>	1.62	1.61	1.60	1.60	1.63	1.61	1.62	1.62	0.66	1.24	0.35
Sm	1.40	1.39	1.38	1.38	1.41	1.39	1.40	1.40	0.66	1.27	0.35
Fe <sub>2</sub> O <sub>3T</sub>	1.61	1.62	1.64	1.61	1.68	1.65	1.66	1.62	1.56	1.24	0.84
Fe	1.13	1.13	1.15	1.13	1.17	1.15	1.16	1.13	1.56	1.31	0.80

$$Horrat = \frac{RSD}{CV_{Horwitz}} \quad (5)$$

## RESULTS AND DISCUSSION

### Research Results

All analytes value of this study is  $\gg 1,000$  ppm (0.1%) which RSD<sub>experimental</sub> should not be more than 10% based on Commission Regulation (EU) No. 836/2011. Moreover, it is stated the other acceptable criteria are that the RSD<sub>experimental</sub>  $\leq 66\% \times CV_{Horwitz}$  and Horrat  $< 2$  [1], [30]. Taking into account the monazite analysis data in this study, almost all of the experimental values were lower than their correspondent theoretical acceptable, except for the ZrO<sub>2</sub> with Horrat of 2.45 as shown in **Table 1**.

Gd <sub>2</sub> O <sub>3</sub>	1.16	1.18	1.17	1.21	1.12	1.21	1.17	1.16	2.49	1.30	1.27
Gd	1.01	1.02	1.01	1.05	0.97	1.05	1.01	1.01	2.49	1.33	1.25
ZrO <sub>2</sub>	1.01	0.91	0.94	1.01	1.02	1.02	0.93	1.02	4.91	1.34	2.45*
Zr	0.75	0.67	0.69	0.75	0.75	0.75	0.69	0.75	4.91	1.40	2.34*
Al <sub>2</sub> O <sub>3</sub>	0.77	0.73	0.73	0.72	0.71	0.72	0.70	0.70	3.06	1.40	1.46
Al	0.40	0.38	0.38	0.37	0.37	0.38	0.36	0.37	3.06	1.55	1.32
TiO <sub>2</sub>	0.76	0.76	0.79	0.76	0.80	0.82	0.83	0.80	3.43	1.38	1.66
Ti	0.46	0.46	0.48	0.46	0.49	0.49	0.50	0.48	3.43	1.49	1.54

\*The value outside the acceptance criteria.

## B. Information Value of the Internal Reference Material

Uncertainty in a set of measurements depicts an estimation of the range of inaccuracy. The uncertainty of reference values is taken into account due to possible inhomogeneity between measurements and/or within the analyzed material. Uncertainties are basically expressed as the standard deviations of the set of analysis [4], [31], [32]. Lower uncertainty indicates a better degree of reference values. Optimizing sample preparation and enlarging the number of analysis are a couple of options to reduce the degree of uncertainty.

ISO and IAG guidelines are established in determining the reference values of any reference materials. The reference values are the averages of a mean of measurement results of the different certified laboratories. On the other hand, information values of composition which provide for information purpose only do not comply with the ISO protocols completely [1]. The information values of each analyte in this study are the average results of eight splits measurement with the uncertainty value as shown in **Table 2**. In order to upgrade its status to become a higher degree of reference material in the future, the studied monazite should be sent and analyzed by other certified laboratories.

**Table 2.** Information values of composition of analytes in the studied monazite.

Analytes	Information value (%)		Analytes	Information value (%)	
CeO <sub>2</sub>	27.9363	± 0.0145	Y <sub>2</sub> O <sub>3</sub>	2.6038	± 0.0005
P <sub>2</sub> O <sub>5</sub>	20.0375	± 0.0083	SiO <sub>2</sub>	1.6313	± 0.0018
La <sub>2</sub> O <sub>3</sub>	12.9288	± 0.0018	Sm <sub>2</sub> O <sub>3</sub>	1.6138	± 0.0001
Nd <sub>2</sub> O <sub>3</sub>	10.8388	± 0.0037	Fe <sub>2</sub> O <sub>3T</sub>	1.6363	± 0.0007
ThO <sub>2</sub>	7.4413	± 0.0010	Gd <sub>2</sub> O <sub>3</sub>	1.1725	± 0.0008
SnO <sub>2</sub>	4.1675	± 0.0051	Al <sub>2</sub> O <sub>3</sub>	0.7220	± 0.0005
Pr <sub>6</sub> O <sub>11</sub>	2.6400	± 0.0022	TiO <sub>2</sub>	0.7900	± 0.0007

## B Monazites Content Comparison

The high P<sub>2</sub>O<sub>5</sub> composition of the sample implies the phosphate mineral character. There are at least four types of monazite based on geochemistry content, namely, monazite-Ce, monazite-La, monazite-Nd, and monazite-Sm. The studied sample should be classified as Monazite-Ce (Ce,La,Nd,Th)PO<sub>4</sub> on the high Ce abundance. Monazite-Ce is the most common type of this mineral and generally correlated with tin tailings around the world, i.e. Indonesia, Malaysia, Thailand, Australia, North America, and Nigeria [33], [34], [35]. In comparison to other monazites from several countries, the studied sample depicts higher concentration of Ce<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub>, Nd<sub>2</sub>O<sub>3</sub>, SnO<sub>2</sub>, Pr<sub>6</sub>O<sub>11</sub>, and ZrO<sub>2</sub> as shown in **Table 3**. The chemical abundant

of monazite from the metallurgical unit in Muntok relatively almost equal with the one from Myanmar, except that a few amounts of tin (SnO<sub>4</sub>) is detected in the studied sample. Although the Iranian monazite had been purified through acidic digestion, leaching, filtration, and calcination procedures [22], the existence of some ferrous minerals is indicated on the SiO<sub>2</sub> and Fe<sub>2</sub>O which should be separated to raise its market price. The economic value of monazite from Australia is reduced by the large phosphate concentration which exceeds 29%. Filtration, acids leaching, and alkaline leaching methods from previous studies might be adopted to solve this problem [23], [24]. **Table 3**. Comparison of chemical composition (wt%) of the studied monazite to the others from several countries.

	Monazite location			
	Metallurgical Unit, Muntok - Bangka	Marvast, Yazd - Iran <sup>a</sup>	Mongmit Myitsone - Myanmar <sup>b</sup>	Victoria - Australia <sup>c</sup>
CeO <sub>2</sub>	27.94	10.10	27.21	18.25
P <sub>2</sub> O <sub>5</sub>	20.04	12.20	19.34	29.02
La <sub>2</sub> O <sub>3</sub>	12.93	6.30	10.73	8.19
Nd <sub>2</sub> O <sub>3</sub>	10.84	6.60	8.90	7.19
ThO <sub>2</sub>	7.44	0.20	8.16	9.27
SnO <sub>2</sub>	4.17	-	-	-
Pr <sub>6</sub> O <sub>11</sub>	2.64	1.50	1.77	1.49
Y <sub>2</sub> O <sub>3</sub>	2.60	0.20	0.89	-
SiO <sub>2</sub>	1.63	23.60	4.26	0.11
Sm <sub>2</sub> O <sub>3</sub>	1.61	-	1.02	4.70
Fe <sub>2</sub> O <sub>3T</sub>	1.64	27.20	1.25	0.11
Gd <sub>2</sub> O <sub>3</sub>	1.17	-	0.66	3.62
ZrO <sub>2</sub>	0.98	-	0.56	-
Al <sub>2</sub> O <sub>3</sub>	0.72	1.40	-	-
TiO <sub>2</sub>	0.79	0.70	-	-

## CONCLUSION

Monazite from PT Timah Metallurgical Unit in Muntok was analyzed for generating an internal reference material. The monazite was divided into splits and stored in glass containers. Eight samples from four splits were analyzed using XRF for the oxides content measurement. The sample is highly valuable on its high plenty CeO<sub>2</sub> and LaO<sub>2</sub> composition that exceed 10%. According to the acceptance criteria of  $RSD_{\text{experimental}} < 10\%$ ,  $RSD_{\text{experimental}} \leq 66\% \times CV_{\text{Horwitz}}$ , and  $H_{\text{orrat}} < 2$ , the concentration of fourteen analytes are reasonable to become information values. The average of eight samples measurement was taken as information values while uncertainty was counted from the analyte's variance. The studied monazite composition resembles the other purified mineral of Myanmar. According to its much lower phosphate composition, the purified monazite from Bangka is more precious than refined monazites from Iran and Australia.

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## ACKNOWLEDGMENTS

The authors thank the Head of the Center for Geological Survey, Bandung for the permission of data publication. We were greatly supported by PT Timah to enter the Metallurgical plant at Muntok and to complete our other works on Bangka Island. Thanks also go to Mr. Purnama Sendjaja, Mr. Joko Subandrio, and Mr. Verry Edi Setiawan which helped a lot regarding geological interpretation. Mrs. Irfanny Agustiany S.Sc., Ms. Citra Okta Hagia and Ms. Indah Yuni Prasetyawati assisted us in laboratory works.

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