

AN IMPROVED IN-HOUSE METHOD FOR THE DETERMINATION OF ^{241}Am IN SURFACE SEDIMENT WITHIN THE EXCLUSIVE ECONOMIC ZONE OF EAST COAST PENINSULAR MALAYSIA

Yii Mei Wo, Hidayah Shahar and Zaharudin Ahmad

Malaysian Nuclear Agency
Bangi, 43000 Kajang, Selangor, Malaysia
Correspondence author: yii@nuclearmalaysia.gov.my

ABSTRACT

Present of ^{241}Am in the environment is being determined as part of surveillance and research programs related to nuclear activities. The separation of ^{241}Am from environmental samples was carried out against the IAEA's reference material by using an improved in-house radiochemical separation method through anion exchange column, followed by the electro-deposition on a stainless steel disc, and finally assayed on alpha spectrometry counting system. The resulting spectra showed good isolated peak, indicating a good separation of the radionuclide of interest. The analytical results were in good agreement with the certified value for IAEA-326 and IAEA-368 with the calculated U-score was 0.36 and 0.82, respectively, showing no significant difference between the experimental and certified value. Using this method, distributions of ^{241}Am in seabed surface sediment in the Exclusive Economic Zone of East Coast Peninsular Malaysia were studied. Samples were collected during June 2008 where the concentrations of ^{241}Am were found to be ranged from < 0.08 to 0.36 Bq/kg, dry weight.

Keywords: Alpha spectrometry, ^{241}Am , electro-deposition, radiochemical separation, sediment

INTRODUCTION

For the past decades, studies on radionuclides present in the environment and their possible effect, either acute or chronic, on human health had increased annually. Many of these radionuclides are artificial and can be transported over long distances from their source of emission, removed from the atmosphere, then moved into the biosphere and hydrosphere and finally into the human body through different pathways (Zaharudin et al., 2003). The accumulation of hazardous radionuclides in the environment and fallout isotopes started since the first nuclear test and had continued until now (Ugur and Yener, 2003).

Most of the radioactive elements produced in atmospheric nuclear tests will decay rapidly. However, considerable amounts of ^{241}Am , mainly formed from the decay of ^{241}Pu , still present in global fallout nowadays. Estimated activity of ^{241}Am that have been injected into the atmosphere as a consequence of nuclear tests and accidents, is around 6 TBq (IAEA, 1986; WHO, 1989). ^{241}Am attracted considerable attention from the radioecological point of view due to their high radiotoxicity (Yusoff and Bradley, 2001), long physical half-life (~ 430 years), high chemical reactivity and long residence in biological systems. Such behaviour raise concern over the safety of foods. According to Codex Alimentarius Commission in CAC/GL 5, the maximum levels for alpha emitting radionuclides in food, such as ^{241}Am , have been established at 10 Bq/kg for general consumption and at 1 Bq/kg for infant food and milk (Codex, 1989).

Present of ^{241}Am in the environment is being determined as part of surveillance and research programs related to nuclear activities. Although ^{241}Am can be measure directly at 59.54 keV energy line using gamma spectrometry system, it is not very suitable for low level ^{241}Am such as those presented in the environment due to high interference in gamma spectrometry system at energy below 100 keV (Gilmore and Hemingway, 1998). Therefore, measurement of ^{241}Am using radiochemical separations and alpha spectrometry will be a more appropriate and reliable choice.

Americium is an element in the transuranic series and it is usually dominated by the trivalent Am (III) form. Most analytical procedures for ^{241}Am tend to be long and tedious (Knab, 1979). Extensive work had been done on methods to separate ^{241}Am from other elements (Ageyev et al., 2005). Usually these methods consist of a combination of techniques such as precipitation, co-precipitation, solvent extraction and ion exchange. Several adopted methods had been tried in our laboratory but none produce satisfactory results and/or due to difficulty in getting certain chemicals. Therefore, a method obtained during IAEA fellowship training had been adopted and modified to overcome these problems.

On the other hand, there are potential risks of planned and unplanned releases of ^{241}Am , from the global nuclear activities to move into Malaysian marine environment. It is therefore necessary to monitor the presence and distribution of ^{241}Am in order to evaluate their potential risk to human life. Human can be affected directly by this artificial radionuclide especially through consumption of contaminated seafood. This developed in-house method had been applied to determine the level of ^{241}Am in the Malaysian marine environment. The Exclusive Economic Zone of East Coast Peninsular Malaysia is the designated area for this study. The zone covers an area between $1^{\circ} 14.04'$ to $7^{\circ} 48.92'$ N latitude and $102^{\circ} 5.03'$ to $105^{\circ} 48.77'$ E longitude with approximately 1,150 km long and has a maximum width of 417 km, with an area around 130,000 km². It is relatively shallow with an average depth between 60 – 70 meters. (Mohsin and Mohamed, 1988).

MATERIALS AND METHODS

Reagents and Standard

Yield determinant used was ^{243}Am (SRM 4332B, National Bureau of Standards). All the other chemicals used are of Analar grade, which include Hydrochloric Acid (37%) and Nitric Acid (65%) from Merck Chemicals. 2 ml TRU resin column, 100 – 150 μm from Eichrom Ltd. and Resin Ag 1X2, 50-100 mesh form from Bio Rad Ltd. All chemicals were used as supplied.

Counting System

Canberra Alpha Spectrometry Counting System with Alpha Passivated Implanted Planar Silicon (PIPS) detectors, with active detector surface of 450 mm² and efficiency of 25% for source to detector distance less than 10 mm. The background count is less than 1 count per hour.

Sample Dissolution

To a small amount of sample (~ 1 – 2 g), ~ 5 mBq ^{243}Am was employed as yield determinant. Then, strong acid leaching was performed using combination of nitric and hydrochloric acid for few times before been evaporated to total dryness.

Column Separation

After evaporation, sample was added with 8 M HNO₃, any un-dissolved material was removed using centrifugation. A 7 cm high with 1 cm diameter resin column (Ag 1-X2, Cl⁻ form) was prepared and pre-conditioned with 8 M HNO₃ at a flow rate of ~ 5 mL/min. Then, the sample solution was passed through the column, washed with substantial amount of 8 M HNO₃. The elute and the wash from 8 M HNO₃ was collected together and evaporated to dryness.

Purification by Ion Exchange and Electro-Deposition

Sample was re-dissolved in 2 M HNO₃ and passed through a pre-conditioned 2 M HNO₃ Eichrom TRU resin column. It was further washed with 2 M HNO₃, 0.7 M HNO₃ before eluting americium using 0.05 M HNO₃. Eluted sample solution was evaporated to dryness before plating, it was electro-deposited on a stainless steel disc using a method published earlier (Yii et al., 2006). The disc is counted for 3 days using the alpha spectrometry counting system.

Study Area and Sampling

Sampling of seabed surface sediment was carried out on 10 – 30 June 2008 aboard the vessel KL PAUS at the Exclusive Economic Zone area as shown in Figure 1. Sediment samples were collected by employing PONAR grab sampler and brought back to land laboratory for further analyses. In laboraroty, samples were dried in the oven at 60 °C (to prevent losses of ²¹⁰Po for other study) until constant dry weight. Dried samples were finely grinded and sieved at 200 mesh. Then, the samples were processed using the same method as of the reference material.

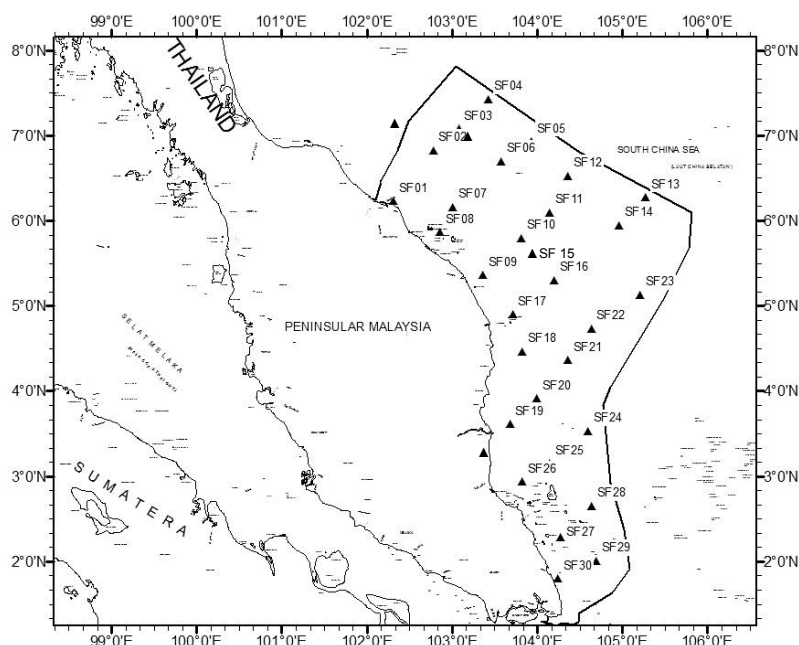


Figure 1: Map showing sampling stations

RESULTS AND DISCUSSION

In-House Method

The reliability of the method was studied using the reference material IAEA-326 and IAEA-368 as samples. The IAEA-326 sample was a black soil obtained from the Kursk region in Russia in 1990. Meanwhile, the IAEA-368 sample was collected in June 1989 in the Pacific Ocean at the Mururoa Atoll. Due to the property of the reference materials itself, during the digestion process, IAEA-368 was completely dissolved whilst IAEA-326 was only been leached, reflecting two different behaviour groups of sample. A total of 10 and 17 samples of IAEA-326 and IAEA-368, respectively, had been studied using this method. Grubbs' outlier test shows no significant outlier data (GRAPHPAD, 2008). Information on these two materials is given in Table 1.

Table 1: Information of reference material used

| Sample | Type | Reference Date | ²⁴¹ Am (95% Confidence Level) (Bq/kg) |
|----------|------------------------|--------------------------------|--|
| IAEA-326 | Soil | 31 st December 1994 | 0.16 – 0.22 |
| IAEA-368 | Pacific Ocean Sediment | 1 st January 1990 | 1.2 – 1.5 |

The resulting spectra showed no overlapping of peaks observed indicating a good separation of radionuclide of interest. The ²⁴¹Am peak and ²⁴³Am yield determinant peak can be easily identified at its respective energy regions, marked accordingly and the experimental value was calculated. From the experiments, yield determinant recoveries generally exceed 80 % indicating a good chemical separation for alpha spectrometric analysis. An example of spectra observed for reference material and sample is given in Figure 2 and Figure3, respectively.

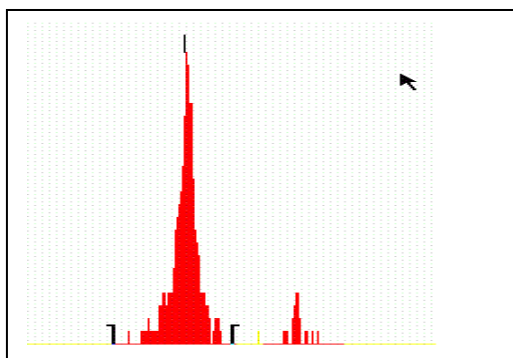


Figure 2: Reference material

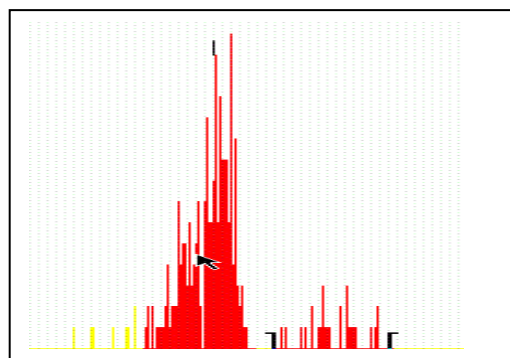


Figure 3: Sample

U-score was also calculated to determine how significant the experimental value differ from the certified value. The U-score was calculated using eq. (1) below:

$$U - score = \frac{|value_{certified} - value_{measurement}|}{\sqrt{\sigma_{certified}^2 + \sigma_{measurement}^2}} \quad (1)$$

Where $value_{certified}$ is the certified value of ^{241}Am of the reference material and $value_{measurement}$ is the value of ^{241}Am measured using this method. The $\sigma_{certified}$ is the uncertainty of the certified value whilst $\sigma_{measurement}$ is the uncertainty of the measured value.

Calculated U-score value for each sample was summarized in Table 2 and 3. A U-score of $u < 1.64$ means that the value measured does not differ significantly from the certified value (Khater et al., 2002). The meaning of U-score values is presented in Table 4.

Table 2: Experimental value and U-score for IAEA-326

| Date | ^{241}Am (Bq/kg) | | U-score |
|----------------|---------------------------|-----------------------|-------------|
| | Value | 2σ Uncertainty | |
| 22 May 2007 | 0.1497 | 0.0184 | 1.28 |
| 22 May 2007 | 0.2337 | 0.0288 | 1.31 |
| 04 Sept 2007 | 0.2279 | 0.0278 | 1.15 |
| 04 Sept 2007 | 0.2768 | 0.0336 | 2.52 |
| 30 Nov 2007 | 0.1769 | 0.0221 | 0.41 |
| 30 Nov 2007 | 0.1680 | 0.0208 | 0.69 |
| 30 Nov 2007 | 0.2215 | 0.0271 | 0.96 |
| 30 Nov 2007 | 0.1966 | 0.0243 | 0.20 |
| 30 Nov 2007 | 0.1652 | 0.0205 | 0.78 |
| 30 Nov 2007 | 0.2009 | 0.0246 | 0.34 |
| Average | 0.2017 | 0.0248 | 0.36 |

Table 3: Experimental value and U-score for IAEA-368

| Date | ^{241}Am (Bq/kg) | | U-score |
|----------------|---------------------------|-----------------------|-------------|
| | Value | 2σ Uncertainty | |
| 26 June 2007 | 1.5422 | 0.1848 | 1.09 |
| 26 June 2007 | 1.1851 | 0.1426 | 0.99 |
| 26 June 2007 | 1.5878 | 0.1900 | 1.34 |
| 26 June 2007 | 1.1903 | 0.1426 | 0.96 |
| 26 June 2007 | 1.3861 | 0.1657 | 0.21 |
| 26 June 2007 | 1.4452 | 0.1728 | 0.55 |
| 04 Sept 2007 | 1.3296 | 0.1592 | 0.12 |
| 04 Sept 2007 | 0.8462 | 0.1019 | 3.18 |
| 04 Sept 2007 | 0.9186 | 0.1102 | 2.70 |
| 04 Sept 2007 | 1.2473 | 0.1499 | 0.61 |
| 04 Sept 2007 | 1.4348 | 0.1722 | 0.49 |
| 10 Dec 2007 | 1.0357 | 0.1260 | 1.93 |
| 10 Dec 2007 | 0.9100 | 0.1099 | 2.75 |
| 10 Dec 2007 | 0.9820 | 0.1218 | 2.27 |
| 10 Dec 2007 | 1.5748 | 0.1896 | 1.27 |
| 10 Dec 2007 | 1.4652 | 0.1777 | 0.67 |
| 10 Dec 2007 | 1.5222 | 0.1830 | 0.98 |
| Average | 1.2129 | 0.1460 | 0.82 |

Table 4: Physical meaning of the U-score values

| Condition | Probability | Status |
|-------------------|------------------------|--|
| $u < 1.64$ | Greater than 0.1 | The reported value does not differ significantly from the certified value |
| $1.95 > u > 1.64$ | Between 0.1 and 0.05 | The reported value probably does not differ significantly from the certified value |
| $2.58 > u > 1.95$ | Between 0.05 and 0.01 | It is not clear whether the reported value differ significantly from the certified value |
| $3.29 > u > 2.58$ | Between 0.01 and 0.001 | The reported value is probably significantly different from the certified value |
| $u > 3.29$ | Less than 0.001 | The reported value significantly differs from the certified value |

Usually, minimum six replicates data were required to ensure the validity of data (LGC Limited, 2001). Therefore, from the average experimental value of ^{241}Am , U-score was calculated to be 0.36 and 0.82 for IAEA-326 and IAEA-368, respectively, showing no significant difference between the experimental and certified value. This shows that the method is reliable for determining ^{241}Am in environmental samples. Generally, the experimental values for ^{241}Am are close to the certified value. The slight differences can be attributed to the fact that the certified value of the reference material is a calculated average value by the IAEA from the pool of different values obtained from measurements using several different techniques.

^{241}Am Distribution in EEZ

^{241}Am concentration at each location as well as the physical and chemical parameters such as salinity, temperature and DO were also measured as supporting parameters were summarized in Table 5. Concentrations of ^{241}Am were found to be ranged from < 0.08 to 0.36 Bq/kg dry weight.

Due to usual low concentration in environmental sample, quantitative determination of ^{241}Am using Gamma Spectrometry system is not that reliable as compared to Alpha Spectrometry system. Moreover, the complicated chemical separation technique also discourage the study on this radionuclide when compared to ^{137}Cs and $^{239+240}\text{Pu}$ by other researchers. Not much past studies on ^{241}Am could be found. Generally, activity concentrations of ^{241}Am found in this study is similar to that reported in Gokova Bay, which ranged from 0.003 to 0.33 Bq/kg dry weight (Ugur and Yener, 2003). Meanwhile, it were considerable high when compared to the area that is free from nuclear activities such as in Antarctica, where ^{241}Am ranged from 0.0024 to 0.044 Bq/kg dry weight (Desideri et al., 2003).

On the other hand, the finding is lower when compared to areas that having active nuclear activities such as reported by Yamamoto et al. (1990) and Schertz et al. (2006). Yamamoto et al. (1990) reported that the concentration of ^{241}Am in the surface sediment at Nyu Bay at the coastal area of the Sea of Japan is about 1.5 Bq/kg dry weight, whileas Schertz et al.(2006) reported that the concentration of ^{241}Am in the surface lake sediment at an alpine wetland, France is about 2 Bq/kg dry weight.

The different between ^{241}Am found in this study when compared to elsewhere could probably due to the differences of particle affinity and biological uptake of these nuclides in the marine

ecosystem (Hong et al., 1999) and also nuclear activities in that area. McCubbin et al. (2004) also saying that ^{241}Am is highly particle reactive where it will rapidly removed from the water to the seabed sediment. Ocean circulation is also expected to play an important role in distribution of this radionuclide.

Table 5. Date of sampling, sampling location coordinates, physical and chemical parameters, activity concentrations of ^{241}Am in surface sediments

| Station | Date | Latitude | Longitude | Temp °C | Salinity PSU | pH | Turbidity | $^{241}\text{Am} \pm 2\sigma$ (Bq/kg) |
|---------|----------|--------------|---------------|---------|--------------|------|-----------|---------------------------------------|
| SF01 | 18.06.08 | 06° 13.99' N | 102° 19.00' E | 30.91 | 32.66 | 8.18 | 2.800 | 0.15 ± 0.02 |
| SF02 | 17.06.08 | 06° 50.04' N | 102° 47.04' E | 30.46 | 33.04 | 8.23 | 2.600 | < 0.08 |
| SF03 | 17.06.08 | 07° 05.03' N | 103° 04.99' E | 30.29 | 33.17 | 8.22 | 2.700 | 0.19 ± 0.02 |
| SF04 | 17.06.08 | 07° 25.98' N | 103° 26.01' E | 30.04 | 32.98 | 8.21 | 2.700 | 0.16 ± 0.02 |
| SF05 | 16.06.08 | 06° 56.09' N | 103° 56.04' E | 30.03 | 33.53 | 8.18 | 2.633 | 0.14 ± 0.02 |
| SF06 | 16.06.08 | 06° 42.14' N | 103° 35.17' E | 30.11 | 33.57 | 8.21 | 2.700 | 0.17 ± 0.02 |
| SF07 | 16.06.08 | 06° 10.00' N | 103° 01.00' E | 30.25 | 33.27 | 8.19 | 2.600 | 0.24 ± 0.03 |
| SF08 | 18.06.08 | 05° 52.10' N | 102° 51.92' E | 30.41 | 33.17 | 8.20 | 2.667 | 0.16 ± 0.02 |
| SF09 | 20.06.08 | 05° 22.06' N | 102° 21.97' E | 28.66 | 33.98 | 8.16 | 2.650 | < 0.08 |
| SF10 | 14.06.08 | 05° 48.20' N | 103° 48.98' E | 30.26 | 33.75 | 8.23 | 2.550 | < 0.08 |
| SF11 | 14.06.08 | 06° 06.16' N | 104° 09.11' E | 30.03 | 33.55 | 8.19 | 2.667 | 0.15 ± 0.02 |
| SF12 | 14.06.08 | 06° 32.01' N | 104° 22.11' E | 29.59 | 33.69 | 8.20 | 2.800 | 0.26 ± 0.03 |
| SF13 | 13.06.08 | 06° 16.98' N | 105° 16.99' E | 29.85 | 33.58 | 8.21 | 2.650 | 0.14 ± 0.02 |
| SF14 | 13.06.08 | 05° 57.15' N | 104° 58.13' E | 29.63 | 33.57 | 8.20 | 2.550 | 0.25 ± 0.03 |
| SF15 | 12.06.08 | 05° 29.08' N | 104° 29.02' E | 29.61 | 33.29 | 8.22 | 2.500 | 0.27 ± 0.03 |
| SF16 | 12.06.08 | 05° 18.50' N | 104° 12.60' E | 29.68 | 33.38 | 8.20 | 2.600 | 0.28 ± 0.03 |
| SF17 | 20.06.08 | 04° 54.12' N | 103° 42.98' E | 29.94 | 33.97 | 8.15 | 2.700 | < 0.08 |
| SF18 | 11.06.08 | 04° 28.14' N | 103° 49.98' E | 29.76 | 33.65 | 8.16 | 2.575 | 0.21 ± 0.03 |
| SF19 | 22.06.08 | 03° 37.07' N | 103° 41.08' E | 29.50 | 34.01 | 8.15 | 2.800 | 0.23 ± 0.03 |
| SF20 | 22.06.08 | 03° 55.10' N | 104° 00.05' E | 29.72 | 33.91 | 8.22 | 2.750 | 0.18 ± 0.02 |

| | | | | | | | | |
|------|--------------|-----------------|------------------|-------|-------|----------|-------|----------------|
| SF21 | 23.06.0 8 | 04° 22.16' N | 104° 22.07' E | 29.64 | 33.84 | 8.2 4 | 2.650 | 0.17 ± 0.02 |
| SF22 | 23.06.0 8 | 04° 44.19' N | 104° 38.44' E | 29.63 | 33.83 | 8.2 0 | 2.700 | 0.28 ± 0.03 |
| SF23 | 12.06.0 8 | 05° 08.10' N | 105° 12.90' E | 29.89 | 33.48 | 8.2 2 | 2.700 | 0.36 ± 0.04 |
| SF24 | 23.06.0 8 | 03° 32.08' N | 104° 36.00' E | 29.75 | 33.73 | 8.2 3 | 2.650 | 0.17 ± 0.02 |
| SF25 | 24.06.0 8 | 03° 09.14' N | 104° 09.04' E | 29.03 | 33.98 | 8.1 7 | 2.667 | 0.24 ± 0.03 |
| SF26 | 26.06.0 8 | 02° 56.13' N | 103° 49.97' E | 29.21 | 34.03 | - | 2.850 | 0.14 ± 0.02 |
| SF27 | 24.06.0 8 | 02° 16.94' N | 104° 16.97' E | 29.25 | 33.93 | 8.2 4 | 2.700 | 0.22 ± 0.03 |
| SF28 | 24.06.0 8 | 02° 39.18' N | 104° 38.91' E | 29.74 | 33.99 | 8.1 8 | 2.750 | 0.23 ± 0.03 |
| SF29 | 25.06.0 8 | 02° 00.55' N | 104° 41.97' E | 29.48 | 33.78 | 8.2 3 | 2.800 | < 0.08 |
| SF30 | 25.06.0 8 | 01° 48.04' N | 104° 15.03' E | 30.91 | 32.66 | 8.1 6 | 5.967 | 0.22 ± 0.03 |

CONCLUSIONS

In this work, the activity of ^{241}Am obtained using the improved in house method shows no significant difference from the certified value for both reference materials. Since the spectrum shows good peak separation with activity value in agreement and high recovery of yield determinant, it can be concluded that the method is reliable for analysis ^{241}Am in environmental samples. Concentrations of ^{241}Am in seabed surface sediment in the Exclusive Economic Zone of East Coast Peninsular Malaysia were found to be ranged from < 0.08 to 0.36 Bq/kg, dry weight.

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