

## DETERMINATION OF PLUTONIUM ISOTOPES IN STANDARD IAEA REFERENCE MATERIALS BY DESTRUCTIVE ANALYTICAL TECHNIQUE

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An analytical procedure for the determination of plutonium isotopes (<sup>238</sup>Pu, <sup>239</sup>Pu and <sup>240</sup>Pu) with low detection limit was examined for reliability and accuracy. This procedure is based on chemical separation by ion exchange followed by alpha spectroscopy analysis. The technique and the suggested method were applied on ten samples of IAEA reference materials. A good agreement between the results is obtained by this procedure and the certified values were found. The suggested analytical method may be used for the analysis of contaminated samples for safeguards purposes.

**Keywords:** *Plutonium isotopes, Radiochemical separation, Destructive technique, Pu, isotopic ratio.*

### INTRODUCTION

One of the main objectives of nuclear safeguards program of IAEA is the accountancy of nuclear materials that requires the accurate mass measurement of the uranium and plutonium isotopes in the large variety of the environmental samples [1,2]. There is required to detect the undeclared radioactive nuclear materials by analyzing of isotopic fingerprints of trace amounts of nuclear materials in waste samples taken inside the examined facilities or assembled in an environment [3,5]. The detection of the isotopic ratio of <sup>240</sup>Pu to <sup>239</sup>Pu is useful to determine the source of plutonium in the environmental samples from the viewpoint of safeguards [2]. There is numerous accurate, reliable and rapid analytical methods with low detection limits to

determine the concentration of the radionuclide in environmental samples [2,6]. The majority of them uses several radiochemical procedures to separate of plutonium from the matrix. Many chemical separation techniques for plutonium based on anion exchange chromatography, solvent extraction chromatography have been investigated for analysis of spent fuel and environmental samples [8]. Plutonium is an artificial element, but it can be found in nature in very small amounts, formed by neutron reactions on uranium [9]. In many laboratories, plutonium was radiochemically separated using an anion exchange procedure which gives clean separations from the matrix of environmental samples [10].

The most important chemical properties of plutonium considering its environmental behavior are its oxidation states, solubility, hydrolysis, complexation, sorption and colloid formation reactions differ significantly from one oxidation state to another [11]. Plutonium can exist in solution in different oxidation states III, IV, V, and VI, as  $\text{Pu}^{3+}$ ,  $\text{Pu}^{4+}$ ,  $\text{PuO}^{2+}$  and  $\text{PuO}_2^{2+}$ , respectively [9].

The analytical methods commonly used for Pu determination are semiconductor alpha spectrometry (SAS), a liquid scintillation counter and mass spectrometry. Conventional alpha spectrometry and liquid scintillation counting are generally the most widely used methods, however, they are incapable of distinguishing between  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  because their alpha energies are too close to be resolved. Therefore only the total Pu activity, which is the sum of  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  was obtained [17].

Recently, separation methods based on the extraction chromatography have become increasingly popular in radiochemical analysis [6]. Our research are based on principle developed in this presentation but with the following new approaches based on the fact that, the ratio of  $^{240}\text{Pu}/^{239}\text{Pu}$  are almost constant, and on the well known equations of activity concentration. The purpose of our experiments is the determination of the concentration of  $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  in different IAEA reference material samples.

## EXPERIMENTAL PROCEDURE

The actinides of which most are alpha decaying are analyzed by alpha spectrometry (after chemical separation). The detection limit of alpha spectrometry is very low, but very thin deposited alpha sources and chemically pure elements are needed to achieve high quality spectra. Sources for alpha spectrometric measurements are usually prepared by electro deposition method [12], since this technique gives high chemical recovery, moreover a very thin layers can be deposited, which is essential for a high resolution of the peak.

### A. $^{240}\text{Pu}/^{239}\text{Pu}$ Isotopic Ratio

The ratio  $^{240}\text{Pu}/^{239}\text{Pu}$  depends on the composition of the source material and the subsequent irradiation history as shown in Table 1.

**Table 1.**  $^{240}\text{Pu}/^{239}\text{Pu}$  ratios from different sources.

Source	$^{240}\text{Pu}/^{239}\text{Pu}$
Integrated weapon test fallout	0.18
Weapon production	0.01-0.07
Power reactors	0.23-0.67
Chernobyl accident	0.4
Soil from the Semipalatinsk nuclear test site (SNTS)	0.04-0.07

Nuclear weapons construction requires a low  $^{240}\text{Pu}/^{239}\text{Pu}$  ratio ( $<0.07$ ). After detonation this ratio increases due to neutron capture, the exact value depending on the test parameters and the type of weapon detonated. For this reason, the  $^{240}\text{Pu}/^{239}\text{Pu}$  ratio in weapon test fallout varies between 0.03 and 0.35 [16]. Table 2 summaries the information compiled by the indicated working group for the value of the measured  $^{240}\text{Pu}/^{239}\text{Pu}$  ratio in different IAEA reference material samples.

**Table 2.** The sum of the information compiled by the indicated working group.

Sample	$^{240}\text{Pu}/^{239}\text{Pu}$	Reference
IAEA-375	0.220±0.010	Cheol-SU Kim et al. [17]
IAEA-300	0.190±0.020	
IAEA-6	0.180±0.010	
	0.191±0.005	J. E. McAninch et al . [18]
	0.170±0.004	
	0.180±0.004	
IAEA-135	0.204±0.027	S. H. Lee et al. [19]
	0.210±0.027	
	0.192±0.043	
	0.246±0.043	J. E. McAninch et al . [18]
	0.211±0.004	
	0.213±0.006	
IAEA-134	0.212±0.008	S.H. Lee et al. [19]
IAEA-385	0.182±0.020	IAEA-AQCS [20]
IAEA-414	0.210±0.010	
	0.230±0.050	

### B. Sample preparation

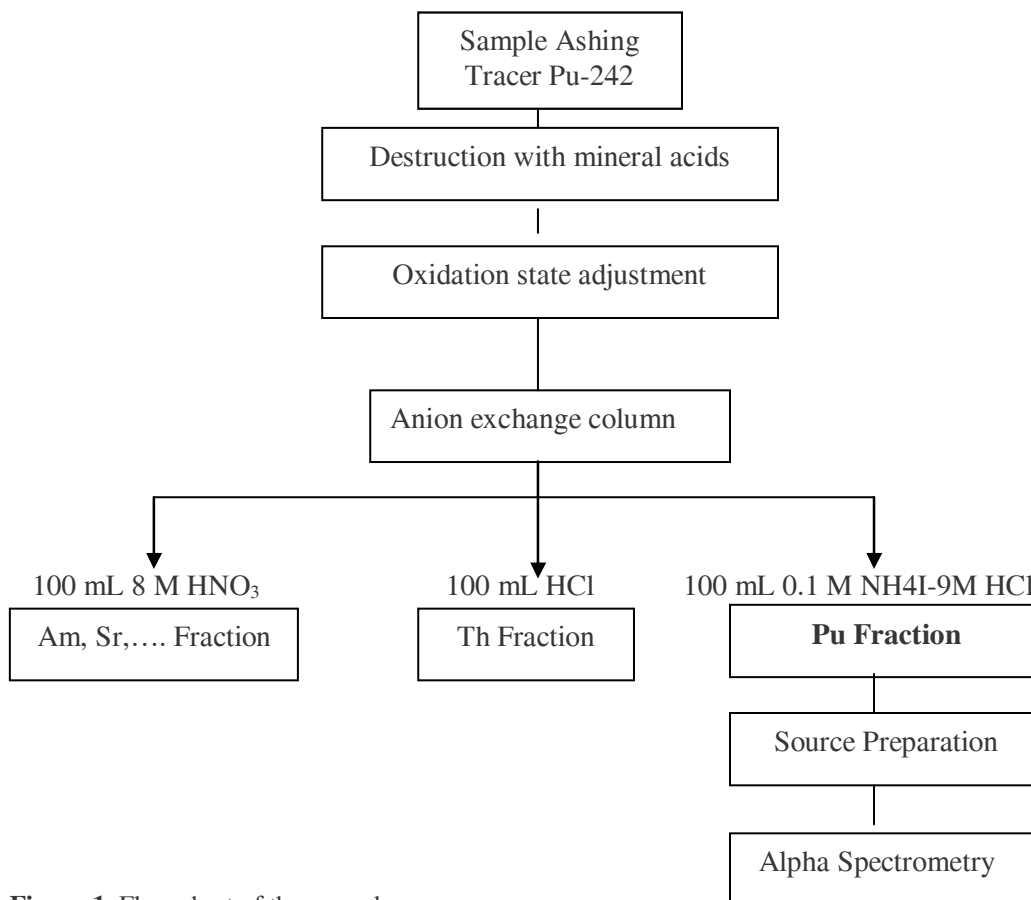
Ten IAEA reference material samples were selected in this study. 12-g weight from IAEA-6, 10-g weight from IAEA-375, 0.16-g weight from IAEA-135, 12-g weight from IAEA-300, 5-g weight from IAEA-385, were treated in addition to three samples from IAEA-414 each of 50-g, and two samples from IAEA-134 each sample has 1-g of dry weight were also treated. For QC purposes, a blank sample was also

treated with the studied samples. The samples were dry ashed with the blank in a porcelain crucibles in an electric muffle furnace up to 600 °C for 24 hours to eliminate any organic matter and burn off all carbonaceous material [13].

Samples were placed into porcelain beakers for digestion process. To each beaker of the studied samples including the reagent blank, about 0.9175 dpm of  $^{242}\text{Pu}$  tracer is added as a yield tracer to evaluate chemical recovery. All reagents used were of analytical grad and solutions were prepared in deionized water [14]. The samples and the blank were digested in a Teflon beakers with 30 ml conc.  $\text{HNO}_3$  and a few drops of 30 %  $\text{H}_2\text{O}_2$  was added to destroy any organic matter. The solution is kept and labeled as solution A. After centrifuge the sample solution, the resulting precipitate was digested again by adding 30 ml 8 M  $\text{HNO}_3$  for five hours. Then the second centrifuge was done. The resulting solution is kept and labeled as solution B, while the resulting precipitate was discarded. Solution B was combined with solution A with an addition of 20mg  $\text{Fe}^{3+}$  as  $\text{Fe}(\text{NO}_3)_3$  in 1M  $\text{HNO}_3$  and deionized water to get 100 mL total volume. The pH was adjusted from 7 to 8 until dark red precipitate appears. After digesting the sample using a hot plat for at least 30 minutes, and centrifuge the sample solution, the precipitate (precipitate A) was kept, the resulting solution is labeled as solution C, then about 5 mL conc.  $\text{HCl}$  was added to solution C to get a  $\text{pH} < 1$  followed by the addition of  $\frac{1}{2}$  ml  $\text{Fe}(\text{NO}_3)_3$  as before then the pH was adjusted again from 7 to 8. The sample solution (solution C) was digested for 30 minutes on the hot plate, and centrifuged the resulting solution (solution D) was discarded, and the resulting precipitate was labeled as (precipitate B). The two precipitates were combined together (precipitate A, & B) and dried in an oven at 60°C and redissolved with the addition of 80 mL 8 M  $\text{HNO}_3$ . About 200 mg  $\text{NaNO}_2$  was added to the solution, and heated until yellow fumes appear, after that the sample was cooled. The flow chart of the procedure is depicted in figure 1.

The chromatography column was prepared and conditioned before introduction of the sample solution. 14 cm column length was selected and the anion exchange resin type AG1-X8, 100-200 mesh was used. 50 mL 8 M  $\text{HNO}_3$  was added for conditioning the column. The sample solution was loaded on the conditioned column with an adjusted flow rate of (1 drop/6 seconds) under gravity flow. 100 mL 8 M  $\text{HNO}_3$  is added to the column and the fractions of Am, Sr, Po, U, Cs, ... were received and kept. In the mean time Thorium fraction was striped from the same column by passing 100 mL 10 M  $\text{HCl}$  solution through it. Finally, the plutonium fraction was eluted from the column by passing 100 mL 0.1M  $\text{NH}_4\text{I}$ -9M  $\text{HCl}$  through it. After that a few drops of  $\text{H}_2\text{O}_2$  were added to oxidize plutonium to Pu-IV. The eluted plutonium fraction from the last stage were evaporated to near dryness. After dryness a 5 mL conc.  $\text{HNO}_3$  with a few drops from  $\text{H}_2\text{O}_2$  were added and evaporated to complete dryness. After complete dryness a 10 ml conc.  $\text{HCl}$  was added and evaporated to dryness. After dryness a 50 ml 10 M  $\text{HCl}$  are added and the second purification column was prepared. 10 cm column length was selected with the same type of resin as before. For conditioning the column 30 mL 10 M  $\text{HCl}$  was added. The sample solution was loaded and 40 mL 10 M  $\text{HCl}$  was added to the column. The receiver was changed and 20 mL 10 M  $\text{HCl}$  was added to the column. The received fraction was kept. Finally plutonium was eluted from the

column by passing 100 mL 0.1 M  $\text{NH}_4\text{I}$ -9M HCl solution through it. The plutonium fraction was evaporated to near dryness, after dryness 10 ml conc.  $\text{HNO}_3$  in addition to a few drops from  $\text{H}_2\text{O}_2$  were added. Then the plutonium fraction was digested and 1 mL 0.3 M  $\text{Na}_2\text{SO}_4$  was added to prevent plutonium to stick with the wall of the beaker, and the solution was evaporated to dryness. After dryness plutonium source was prepared by electro deposition.



**Figure 1.** Flow chart of the procedure.

Platinum wire of horizontal helix shape adjusted at 3mm distance from the disc was used as anode. The sample was electroplated at 1.2 A for one hour. Occasional adjustment may be maintain the current at 1.2 A during the plating period [12]. The sample was counted by alpha spectrometry for recovery before discarding the plating solution and washing from columns [15].

The alpha spectrometry system equipped with a total of 80 silicon surface barrier semiconductor detectors was used in this work for determining alpha emitting radionuclides. Seven EG & G Ortec system are in operation currently, three have 16 alpha spectrometry chambers each ( model no. 576), connected to an amplifier, a

multichannel analyzer and a multiplexer, the other four are integrated Octec PC systems with 8 chambers each. The program used for spectrum analysis and data evaluation was alphavision provided by the EG &G Ortec company.

## RESULTS AND DISCUSSION

### Treatment of spectroscopy data

To evaluate the accuracy of our suggested method ten IAEA reference material samples were analyzed. The results of analyses are presented in Table 3. We took an average value of 0.2 for the ratio  $^{240}\text{Pu}/^{239}\text{Pu}$  for the same type of IAEA reference material samples measured by different authors as shown in Table 2. However, we used the general activity equation (1) to calculate the percentage of the plutonium isotopes  $^{240}\text{Pu}$  and  $^{239}\text{Pu}$  in each sample as a mass percent and by knowing the half life values of  $^{239}\text{Pu}$ , and  $^{240}\text{Pu}$ . The specific activity calculation was carried out depending on that suggestion. The results obtained using this method for calculating the concentration of the  $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$ , and  $^{240}\text{Pu}$  were in a good agreement with the certified values.

The quality control in this study was done by using the u-test. The criteria was to get a value of  $U \leq 3.29$  to pass the accuracy test as given in equation (2).

$$A=N\lambda \quad (1)$$

$$U_{\text{test}} = \frac{|\text{Value}_{\text{Certified}} - \text{Value}_{\text{Analyzed}}|}{\sqrt{\text{Unc.}_{\text{Certified}}^2 + \text{Unc.}_{\text{Analyzed}}^2}} \quad (2)$$

It was found that almost all the analyzed samples passed this QC test, Table 4. This indicates that the suggested analytical procedure in this study showed a good accuracy and precision.

## CONCLUSION

The analytical technique based on chemical separation of plutonium isotopes by ion exchange followed by alpha spectrometry analysis was developed which allows the determination of the plutonium isotopes ( $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$ , and  $^{240}\text{Pu}$ ) with detection limit of 0.01 Bq/kg for Pu-238, 0.005 Bq/kg for Pu-239, and 0.1 Bq/kg for Pu-240.

The technique was applied for the analysis of IAEA reference materials there was found a good agreement in the range from 0 to 41% between our results and the certified values. The suggested method would be useful for the determination of plutonium isotopes by using the conventional alpha spectrometry.

The technique could be used successfully to analyze the plutonium isotopes in waste and environmental samples, also for the testing of samples containing very low activity of plutonium for safeguards purposes.

**Table 3.** Analytical results of IAEA-reference material for  $^{238}\text{Pu}$ ,  $^{239}\text{Pu}$ , and  $^{240}\text{Pu}$ 

Reference Material	Measured Activities for IAEA-Reference Material Bq/kg			IAEA-Certified Reference Material Bq/kg		
	This Work					
	$^{238}\text{Pu}$	$^{239}\text{Pu}$	$^{240}\text{Pu}$	$^{238}\text{Pu}$	$^{239}\text{Pu}$	$^{240}\text{Pu}$
IAEA-135	50.59±1.7	13.42±0.31	246.58±5.69	43 (41.6 – 45)	11(10.58-11.65)	202.0 (194.42-214.148)
IAEA-300	0.212±0.01	0.184±0.004	3.381±0.079	0.150 (0.140 - 0.360)	0.183 (0.178-0.188)	3.367 (3.262-3.462)
IAEA-385	0.48±0.03	0.153±0.005	2.813±0.087	0.47	0.1538	2.8262
IAEA-375	0.048±0.001	0.0141±0.007	0.259±0.012	0.071	0.015 (0.014 - 0.018)	0.285 (0.246-0.322)
IAEA-6	0.022±0.003	0.0399±0.001	0.735±0.0186	---	0.0537 (0.050-0.057)	0.9863 (0.910-1.053)
IAEA-414#1	0.0199±0.0014	0.0057±0.0002	0.1056±0.0036	0.0230 (0.0220-0.0250)	0.0062 (0.0058-0.00645)	0.1138 (0.1072-0.1186)
IAEA-414#2	0.0180±0.0030	0.0057±0.0002	0.1043±0.0003	0.0230 (0.0220-0.0250)	0.0062 (0.0580-0.0065)	0.1138 (0.1072-0.1186)
IAEA-414#3	0.0247±0.0017	0.0062±0.0002	0.1138±0.0039	0.0230 (0.0220-0.0250)	0.0062 (0.0058-0.0065)	0.1138 (0.1072-0.1186)
IAEA-134#1	3.91±0.01	0.8898±0.017	16.324±0.314	3.1( 3.0-3.4)	0.7740	14.226
IAEA-134#2	2.33±0.192	0.7330±0.023	13.464±0.419	3.1( 3.0-3.4)	0.7740	14.226

**Table 4.** U -test for the samples analyzed using alpha spectrometry for plutonium isotopes.

Sample code	U-test of $^{238}\text{Pu}$	Commentes	U-test of $^{239}\text{Pu}$	Commentes	U-test of $^{239}\text{Pu}$	Commentes
IAEA-135	3.20	Accepted	4.10	Not Accepted	3.90	Not Accepted
IAEA-300	0.12	Accepted	0.13	Accepted	0.11	Accepted
IAEA-385	0.20	Accepted	0.16	Accepted	0.10	Accepted
IAEA-375	1.60	Accepted	0.13	Accepted	0.62	Accepted
IAEA-6	*	---	3.7	Not Accepted	3.25	Accepted
IAEA-414#1	1.50	Accepted	2.30	Accepted	1.20	Accepted
IAEA-414#2	1.50	Accepted	2.60	Accepted	1.58	Accepted
IAEA-414#3	0.74	Accepted	0.05	Accepted	0.00	Accepted
IAEA-134#1	4.00	Not Accepted	5.90	Not Accepted	1.70	Accepted
IAEA-134#2	2.80	Accepted	1.70	Accepted	0.60	Accepted

\*Theoretical value not available in the IAEA-AQCS report.

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## تعيين نظائر البلوتونيوم في عينات الوكالة المرجعية باستخدام تقنية تحليل اتلافي

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تم اختبار طريقة تحليلية من وجهة نظر دقة القياس والوثوق بنتائجها وذلك لتعيين نظائر البلوتونيوم بأقل قيمة للحد الأدنى للكشف.

تعتمد هذه الطريقة علي طرق الفصل الكيميائي الإشعاعي بواسطة التبادل الأيوني وعدها وتحليلها بعد ذلك بالمطياف الألفي.

طبقت هذه التقنية المقترحة للحساب لعدد عشر عينات مرجعية من الوكالة الدولية للطاقة الذرية. وقد أظهرت النتائج توافقاً جيداً بين النتائج المحسوبة بهذه الطريقة المقترحة وبين النتائج المعتمدة من الوكالة