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IMPROVED OPTIMUM CONDITION FOR RECOVERY AND MEASUREMENT OF ^{210}PO IN ENVIRONMENTAL SAMPLES

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ABSTRACT

The aim of this study was to determine the optimum conditions for deposition of ^{210}Po and evaluate the accuracy and precision of the results for its determination in environmental samples. To improve the technique for measurement of polonium-210(^{210}Po) in environmental samples. The optimization of five factors (volume of media, acidity, removal of interference, temperature and deposition time) governing the deposition of ^{210}Po onto silver discs were investigated. The optimum conditions for deposition of ^{210}Po in the present study were achieved using plating solution of 80 mL hydrochloric acid (HCl) media at acidity of 0.5 M with the presence of 1.0 gram hydroxyl ammonium chloride and plating temperature of 90^{o}C at 600 rpm for 4 hours. The recorded recoveries obtained using ^{209}Po yield tracer in the CRM IAEA-360 and environmental samples were 85% - 98% showing that the efficiency of the new technique is a distinct improved method over existing ones. Therefore, optimization of ^{210}Po recoveries from aqueous environmental samples can be achieved with accuracy and precision in a cost effective and time saving manner using the optimum conditions preferred in the present study.

Key words, Polonium-210, spontaneous plating, optimum conditions, environmental samples

INTRODUCTION

Polonium-210 is a naturally occurring alpha-emitter with a half-life, $t_{1/2}$ of 138.376 ± 0.002 days (NuDat2.1, 2006). Polonium-210 exists in the environment as a result of decay via ^{210}Pb ($t_{1/2}$ = 22.3 years) and ^{210}Bi ($t_{1/2}$ = 5.15 days) in the ^{238}U decay series (Matthews *et al.,* 2007). Polonium occurs widely in nature and is considered as an important component of man's radiation background. Furthermore, the possibility of health effects for humans and other organisms associated with ^{210}Po arises because of its high energy alpha particle associated and has been reported to have relatively high radiation exposure (UNSCEAR, 2000). Indeed, ^{210}Po is considered to be one of the most radiotoxic naturally occurring radionuclides (Al-Masri *et al.,* 2004), and one of the most important environmental radionuclides due to its wide distribution and potential for human radiation exposure through ingestion and inhalation (Momoshima *et al.,* 2002, Martin

and Ryan, 2004). It is considered one of the most radiotoxic naturally-occurring radionuclides (Jia *et al.*, 2001;Al-Masri *et al.*, 2004a&b) and is therefore an important component of dose assessments (Henricsson *et al.*, 2011; Mathew *et al.*, 2007). The contribution of ^{210}Po to the internal radiation dose to human has been estimated to be around 8% (Nassef *et al.*, 2008). Furthermore, in conjunction with ^{210}Pb, ^{210}Po can be used to evaluate a range of environmental processes, such as sediment accumulation (Sanchez-Cabeza et al. 1999) and organic carbon fluxes in marine environment (Stewart and Fisher 2003).

Due to the above mentioned problems, ^{210}Po in the environmental samples such as air, rain water, rock, soil, marine sediment, sludge, seafood, foodstuffs and others are frequently analyzed for both emergency and routine radiation monitoring. Health physicists use the analytical results of environmental samples to estimate the amount of radioactive material present in the environment, calculating its burden for the radiological workers and general public (Becker and Dietze, 1999). These routine analysis which may involve large volumes of samples require efficient, cost effective and accurate measurement of ^{210}Po from such samples.

The suitable conditions for Polonium deposition was recognised early as a problem in determination of ^{210}Po in environmental samples. Many different optimization conditions and physical arrangements for spontaneous deposition of ^{210}Po onto silver discs have been used based on high recoveries of tracer yield (typically 80%) and satisfactory results being achieved throughout (Mathew *et al.*, 2007). This indicates the robustness of the technique. In this study optimization of five factors (deposition time, temperature, volume of media, removal of interference and acidity) governing the deposition of ^{210}Po onto silver discs were investigated.

Various methods for the separation and determination of ^{210}Po from environmental samples have been developedsince the last decades. Selection of the most appropriate method depends on many factors including the form of the polonium, its concentration in the sample and the type of sample matrix. Deposition methods of ^{210}Po play a crucialrole in elimination of matrix interferences and influence total cost of analysis. Traditionally, the method for alpha spectrometric analysis of polonium is time consuming and generates large volumes of secondary wastes.

Furthermore, a key factor in the preparation and analysis of samples for ^{210}Po is the caution required to avoid lossesdue to volatilization (Matthews *et al.*, 2007; Babatunde, 2016).The determination of ^{210}Po at trace and ultra-trace levels with high accuracy is very important. Therefore, itsdetermination needs to be performed through the simple and rapid method whereas a chemical separation andoptimization of deposition parameters are needed. In fact any possible losses of the ^{210}Po during the analysis can be recovered by use of a tracer of ^{209}Po ($t_{I/2}$ = 102 years). It is assumed that the chemical behaviour of the tracer isidentical to the ^{210}Po. The advantage for determining of ^{210}Po radioactivity by processing the spontaneouslydeposition of ^{210}Po and its determinant yield through ^{209}Po tracer onto silver disc is the very sensitive technique. Hence, it needs a small amount of sample to be analyzed. Application of 210Po

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radioactivity in many research works needs a precise and accurate data of its radioactivity concentration. It is essential to develope radio-analytical techniquesfor ^{210}Po determination that increases the recovery yields and therefore more precise and accurate results.

The aim of this study was to determine the optimum condition for the deposition of ^{210}Po onto silver disc and to evaluate the accuracy and precision of results obtained for the determination of ^{210}Po inenvironmental samples. It is hoped that the results will contribute to the ease of ^{210}Porecovery from aqueous environmental samples in a cost effective and reliable manner.

MATERIALS AND METHODS

Reagents and equipment

In this study, double distilled water was used and all chemicals/reagents used were of analytical grade (Merck, Fluka, BDH and Fisher Scientific). Tracer standard solution of ^{209}Po obtained from the National physics laboratory, United Kingdom was used to calculate the analytical yield. Certified Reference Material, CRM (IAEA-360 from the Mediterranean Sea Sediment) was purchased from the Agency's Laboratories Seibersdorf, Physics Chemistry and Instrumentation Laboratory, Reference Materials Group, Vienna, Austria. The alpha activities of ^{209}Po and ^{210}Po were measured using silicon surface-barrier alpha detectorsOrtec EG&G through their alpha particle emission energies of 4.98 MeV and 5.30 MeV, respectively. The alpha spectrometer was calibrated using a mixed alpha standard source and the counting time was 60,000 s.

Experimental Design

Experiment designs to determine the optimum condition for deposition of ^{210}Po were carried-out based on fiveparameters as follows:

Generic Deposition procedure

For each of the parameters investigated, two replicates of 80 mL 0.5 M hydrochloric acid (HCl) solution as deposition media were prepared in plating glass beakers and each solution was spiked with approximately ~0.1 g of 0.5 Bq/g ^{209}Po tracers. Subsequently, followed by adding 1 g of Bi^{3+}carrier (10 mg/g) solution as a hold-back carrier and 1 g of hydroxyl ammonium chloride (HONH2-HCl; HAC) to avoid the interference of Fe^{3+} and Cr^{6+} during the plating process (Matthews *et al.*,2007; Babatunde*et al.*, 2016). Finally, polonium isotopes were spontaneously plated at 90^{o}C onto brightly polished silver discs at 600 revs per minute (rpm) for 4 hours. Alpha activity concentrations of polonium isotopes onto silver discs were measured by Alpha Spectrometry System for a counting time of 60,000 s.

i. plating time

The plating time for polonium deposition was performed at 2, 4, 5, 6 and 7 hours.

ii. Temperature

In this experiment, five different temperatures for polonium deposition i.e. 25 (room temperature), 40, 65, 85 and 100^{o}C were chosen.

iii. Volume of hydrochloric acid for deposition media

Two replicates of 30, 50, 65, 80, 100 and 120 mL were chosen to determine optimum plating volume

iv. Acidity of hydrochloric acid for deposition media

In order to achieve the optimum acidity for deposition of ^{210}Po, six different acidity levels of HCl media at 0 (distilled water), 0.25, 0.5, 1.0, 1.5 and 2.0 M were investigated.

v. Quantity of hydroxyl ammonium chloride to remove the interferences

In this experiment, six different quantity of hydroxyl ammonium chloride (HAC) (i.e. 0, 0.3, 0.5, 1.0, 1.25 and 1.5 g) were added during the process of polonium deposition. The addition of this chemical was to avoid the interference of Fe3+ and Cr6+ during the plating process.

Verification of method by Determination of ^{210}Po in environmental samples

In order to verify the optimum conditions preferred in this research, a certified reference material (CRM) IAEA-360 (Radionuclides in Mediterranean Sea Sediment) were analysed employing the optimum conditions preferred in the present study. In addition, environmental samples such as seafood and sediment collected from the Bonny/New Calabar estuary system, Niger Delta, Nigeria were also used for verifying the suitability of these optimum conditions for the deposition of ^{210}Po. Radiochemical separation of ^{210}Po was performed as detailed by (Nita Salina *et al.*, 2012). 0.5 g of fineground samples was weighed into Teflon beakers and spiked with ~0.1 g of 0.5 Bq/g ^{209}Po tracer. The samples were digested on a hot plate with concentrated mixture of mineral acid consisting of 15 mL of HF, 10 mL of HNO_3 and 5 mL of $HClO_4$ with 1 mL of H_2O_2added. Then, 5 mL of conc. HNO_3 and 1 mL of H_2O_2 were added into the samples and evaporated until insipient dryness (Yamamoto *et al.*, 1994; Martin *et al.*, 1998; Swift, 1998; Theng *et al.*, 2004; Babatunde *et al.,* 2016). Subsequently, followed by digestion with 10 mL of conc. HCl and evaporated until insipient dryness. (Precaution: the temperature for digestion, evaporation etc. should not exceed than 90^{o}C to avoid losses of polonium isotopes due to volatilization). Finally, polonium isotopes were spontaneously plated for 4 hours at 90°C onto brightly polished silver discs in 80 mL 0.5 M HCl in the presence of Bi^{3+}carrier (10 mg/g) solution as a holdback carrier and 1 g of hydroxyl ammonium chloride (HONH2-HCl) to avoid Fe^{3+} and Cr^{6+}interference during deposition. Then, the silver discs were rinsed with distilled water and with alcohol and dried. Alpha activity concentrations of polonium isotopes onto silver discs were measured by Alpha Spectrometry System for a counting time of 60,000 s. Polonium-210 activity concentrations were corrected to the sampling time.

RESULTS AND DISCUSSION

Optimum conditions for polonium deposition

i. Deposition (plating) time

Optimization of deposition times for spontaneously plating of polonium are indicated in Fig. 1. Generally, the percentages of polonium deposition with six different deposition times (0, 2, 4, 5, 6 and 7 hours) obtained from this study ranged from 0% to 99.6%. It was shown that time-consuming nature of plating procedures was most rapid of 4 hours when the highest recovery of 99.6% of polonium deposition onto silver discwas recorded. According to Godoy and Schüttelkopf 1980; Smith and Hamilton, 1984; Matthews *et al.*,2007), minimizations of the needed deposition time

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may be achieved by employing a optimum temperature, solution volume with agitation of the solution and optimum acid strengths of solution (pH 1.5 - 2). The rate of deposition is also improved by employing a large disc area, but this has the disadvantage of decreasing detection efficiency in the spectrometry system (Matthews *et al.*, 2007).

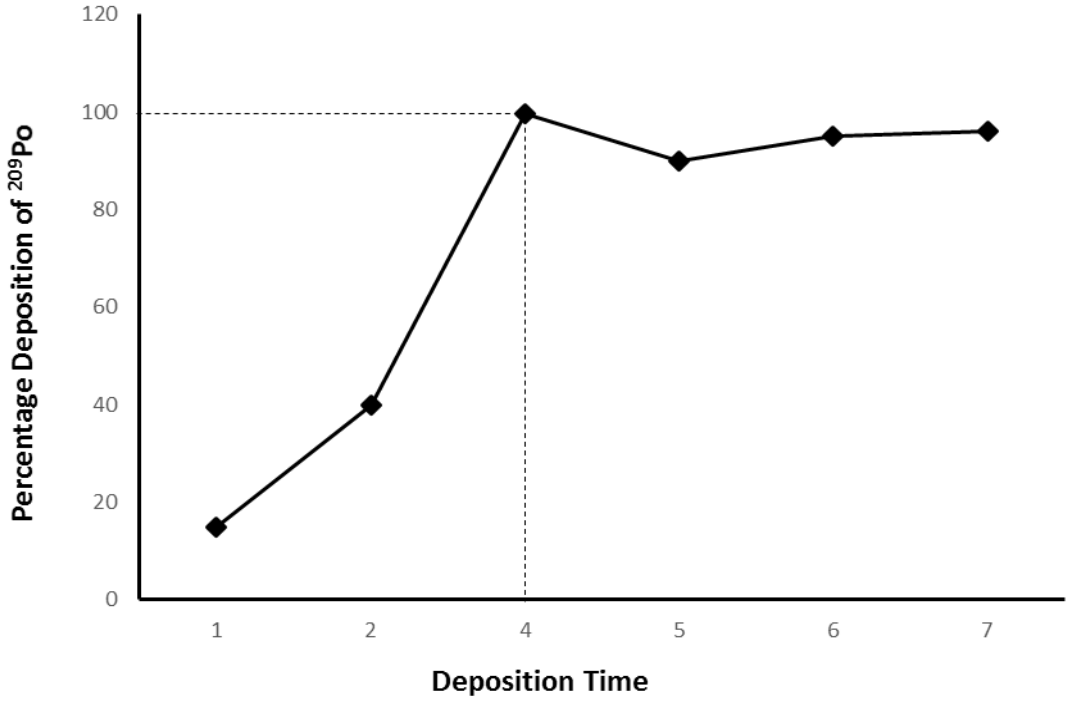

Figure1. Optimization of time (hours) for polonium deposition

ii. Temperature

The percentages of polonium deposition onto silver disc at six different level of temperature (0, 25, 40, 65, 85 and 100^{o}C) ranged from 0% to 98% as shown in Fig. 2. The highest percentage of polonium deposition (98%) occurred at temperature of 90^{o}C. This also showed that losses of polonium begin at temperatures above 100^{o}C. This is highly supported by Matthew*et al.,* (2007) and they also reported that 90% of polonium was lost at 300^{o}C. In addition, the result indicates that low yield of polonium (~50%) at the temperature point of 65^{o}C was probably due to the fact that polonium was being deposited on wall of the plating glass beakersdue to lack of appropriate kinetic energy which was achieved at 90^{o}C.

iii. Volume of hydrochloric acid for deposition media

The result of the experiments to investigate the optimization condition for plating efficiency as a function of volume of HCl media is shown in Fig. 3. The yield depositions of polonium in six different volume of aqueous HCl (30, 50, 65, 80, 100 and 120 mL) ranged from 66% to 100%. It can be observed that the curve reached a plateau at around 80 mL. This means that nearly all the polonium (100%) were deposited onto silver disc at the volume of 80 mL.

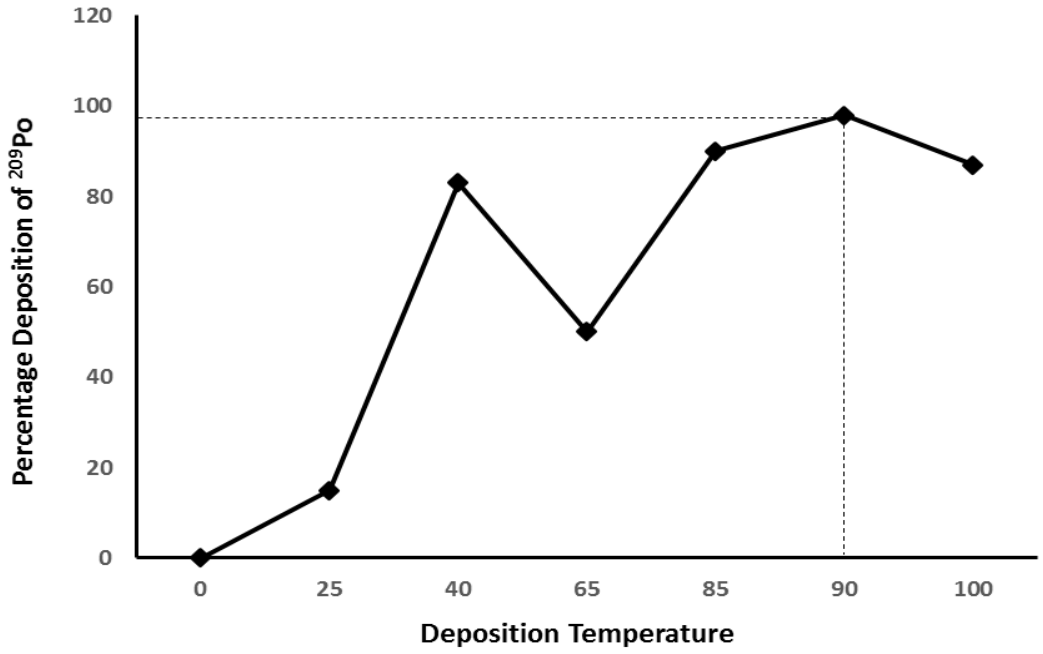

Figure 2. Optimization of temperature (^{o}C) for polonium deposition

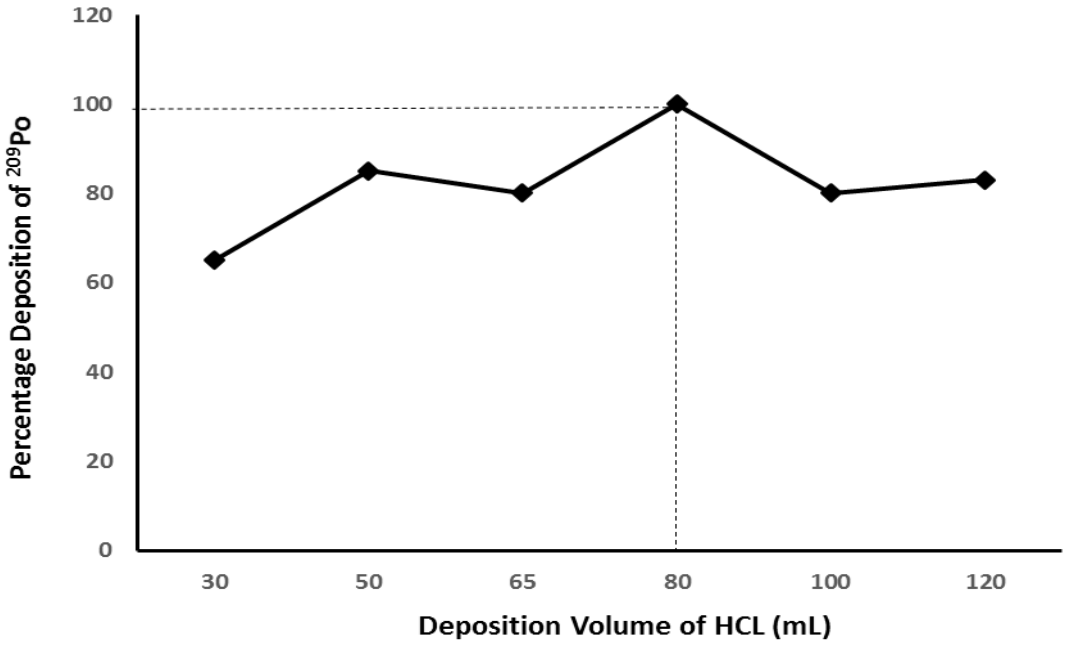

Figure 3. Optimization of acid volume (mL) for deposition media

iv. Acidity of hydrochloric acid for deposition media

The deposition of polonium in aqueous HCl at six different acidity levels (0, 0.25, 0.5, 1, 1.5 and 2 M) is shown in Fig. 4.It was shown that nearly to 90% of polonium was deposited onto silver disc in aqueous HCl at 0.5 M. This means afew percent of polonium (~10%) remains in the aqueous phase if the HCl acidity was in the range of

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0.5 - 1 M. Thecurve also showed that deposition of polonium was decreasing with the increasing of HCl acidity until 1.5 M due to polonium being extracted by organic substances or other interference in media solution. However, at higher acidity levels above 1.5 M, Po recovery increased again Fig. 4. This means that the acidity for maximum recovery of Po according to this experiment would be either at pH level 0.5-1.0 or 2.0-3.0 M. Acidity of 3.0 has been reported as optimum for Po deposition by other authors (Jia et al. 2000; Babatunde *et al.*, 2016).Although, nearly all the polonium (95%) deposited onto silver disc at the HCl acidity of 2 M, due to economic factor, it was concluded that the acidity of HCl at 0.5 M was the bestcondition for deposition media of polonium. Furthermore, it was strongly supported by Matthews *et al.*, (2007) thatthe certain sample types (e.g. biological samples, sediments) the polonium may be auto-deposited from diluted HCli.e. 0.5 M in the presence ascorbic acid or hydroxyl ammonium chloride and/or citrate without any prior chemicalseparation.

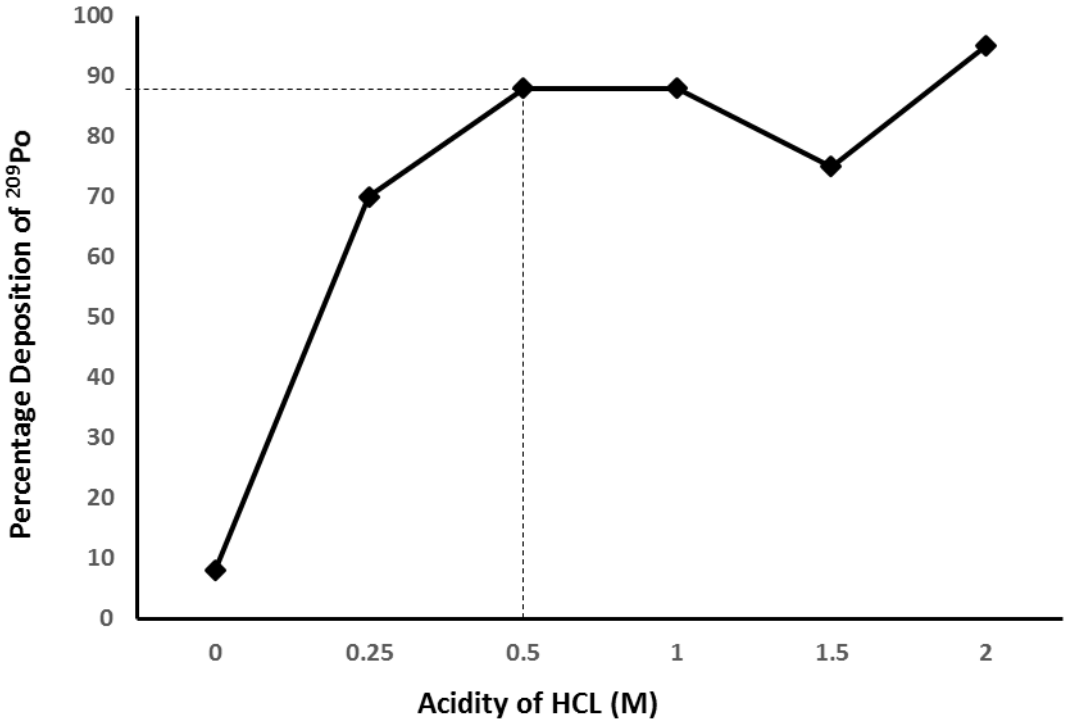

Figure 4. Optimization of HCl acidity (M) for deposition media

v. Quantity of hydroxyl ammonium chloride to remove the interferences

A key feature of source preparation beyond this initial dissolution is the prevention of interference by competingions in the deposition process, particularly Fe^{3+} and Cr^{6+} which can otherwise cause heavy deposits on the platingsilver disc and subsequent loss of counting efficiency and resolution during the a-spectrometric measurement(Matthews *et al.*, 2007). As a result of this problem, hydroxyl ammonium chloride (HAC) is employed for thoseinterferences (Flynn, 1968; Clayton and Bradley, 1995; Ham *et al.*, 1997; Chen *et al.*, 2001; Jia et al., 2004; Babatunde *et al.*, 2016). Inaddition, hold-back carrier of stable Bi^{3+} was also added to prevent

codeposition of radiobismuth (Flynn, 1968; Babatunde *et al., 2016*). Thus, further study of the effects of various matrix elements on theauto-deposition procedure and on the necessity for a prior separation step for different sample types would behelpful.

In line with the above concerns, result of the study on optimization conditions with six different quantity of hydroxyl ammonium chloride (0,0.3, 0.5, 1, 1.25 and 1.5 g) is shown in Fig. 5. The yields of polonium deposition onto silver disc with six differentweight of HAC ranged from 7% to 89%. It was shown that the highest deposition yield of polonium i.e. 89%was achieved at addition of 1.0 g of HAC. It was also shownthat the yields of polonium decreased withthe increasing of weight of HAC (> 1 g). Thus, it can be concluded that 1 g ofHAC was sufficient to remove any possible interferences of Fe^{3+} and Cr^{6+} during the deposition of polonium.

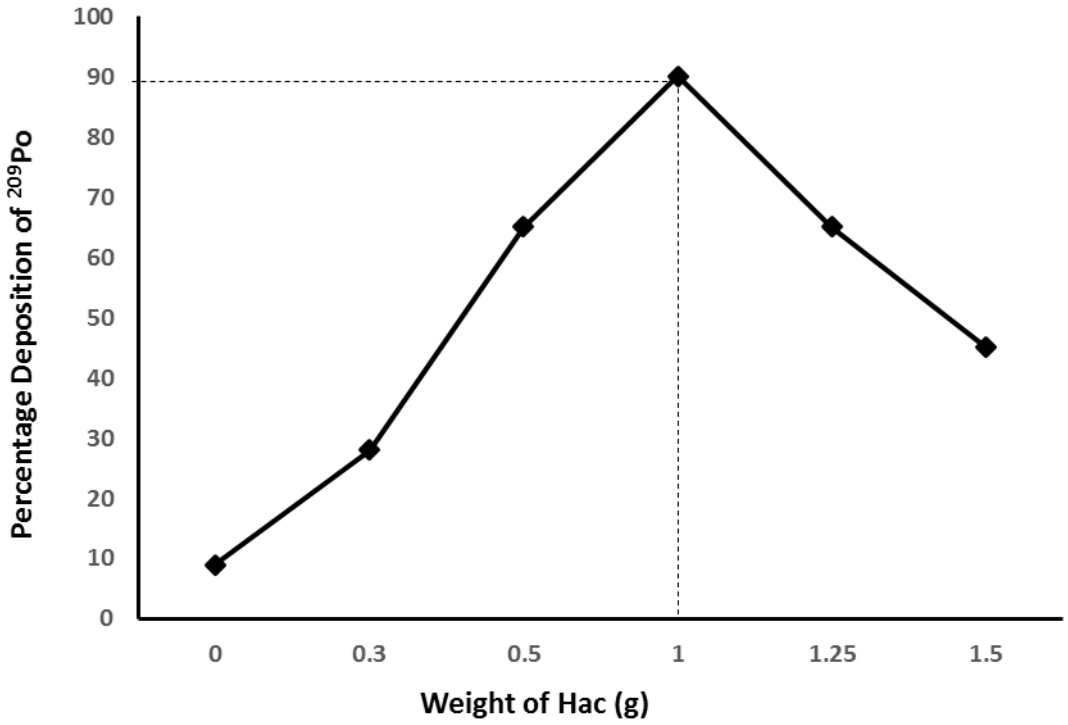

Figure 5. Optimization of HAC weight (g) to remove the interferences during deposition process

Method verification using CRM and environmental samples

The measured activity concentrations 210Po in blank, IAEA-360 and its certified value are presented in Table 1. Theanalysis method used for the measurement of ^{210}Po using using the improved with theanalytical yield ranged from 85% to 98% by the recovery of ^{209}Po. The measured activity of ^{210}Po in the CRM was found to be in the range of 32.6 - 34.2 Bq/kg dry wt. (average: 33.5 Bq/kg dry wt.), while the value of ^{210}Po in CRM was reported from 31.2 Bq/kg dry wt. to 35.3 Bq/kg dry wt with average 32.9 Bq/kg dry wt. Meanwhile, all the ^{210}Po activity concentrations in the blank were below the detection limit. Therefore, the results in general showed reliable and good

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agreement between these the measured and certified value of IAEA-360 as estimated in the 95% confidence interval. This confirms that the optimum conditions preferred in the present research can be used for optimum recovery and precise measurement of Po in environmental samples.

Table 1. Activity concentrations of 210Po in the Certified Reference Material IAEA-385 (Radionuclides in Irish Sea Sediments) (Nita Salina et al., 2012)

IAEA-360 (CRM)	Measured activity	Certified value (Bq/kg dry wt)	95% confidence interval (Bq/kg dry wt)	Analytical Yield (% Recovery)	Blank (Bq/kg dry wt)
Analysis 1	33.7			91.2	BDL
Analysis 2	33.8			95.2	BDL
Analysis 3	32.6	32.9	31.2-35.3	90.1	BDL
Analysis 4	34.1			98.2	BDL
Analysis 5	34.2			91.2	BDL

BDL = Below Detection Limit

This method was also verified using seafood samples from Bonny/New Calabar estuary system, Niger Delta, Nigeria as reported by Babatunde *et al.,* (2015). The analytical yield (recovery) was recorded to be in the range of 85% - 94% for (Table 2). The values of ^{210}Po in seafood samples analysed here and those reported by Babatunde *et al.,* (2015) were not different at statistical significant level of P = 0.261. This high recovery and correlation between measured and reported values indicates that the optimum conditions preferred in the present research for determination of ^{210}Po in environmental samples was reliable, accurate and precise.

Table 2. Activity concentrations of ^{210}Po in seafood samples collected from Bonny/New Calabar estuary system, Niger Delta, Nigeria

Samples	Common name	^{210}Po concentration (Bq kg^{-1}dry wt) Measured	Analytical Yield (%Recovery)	^{210}Po concentration (Bq kg^{-1}dry wt) Babatunde *et al., (*2015)
Mollusc				
T.fuscatus var.radula	periwinkle	162±2	89.5	163±4
T.fuscatus var.fuscatus	periwinkle	178±1	85.5	174±1
Ergeria radiata	clam	242±4	85.6	238±5
Thais sp	dog whelk	140±4	90.1	138±5
G. gasar	oyster	126±2	94.0	123±4
Crustaceans				
Macrobrachium sp	shrimp	72±5	92.0	73±3
Penaeus notialis	cray fish	121±2	93.8	123±3
Portonus sp	crab	81±3	93.5	84.5±3
Fin fish				
Ethmalosa fimbriata	bonga fish	228±5	92.5	223±4

This work was performed to study the optimum condition for the deposition of ^{210}Po onto an silver disc and to evaluate accuracy and precision of this method for determination of ^{210}Po in environmental samples. The optimum conditions for deposition of ^{210}Po were achieved in short time of 4 hours at 90^{o}C. It was ideally performed in 80 mL hydrochloric acid (HCl) media at acidity of 0.5 M with the presence of 1.0 gram hydroxyl ammonium chloride (HAC). These optimum conditions were verified using environmental samples and CRM with known ^{210}Po activities. The results in general showed reliable and good agreement between activity of ^{210}Poin measured environmental samples and known ^{210}Po activity already reported for the same samples. There was also good agreement between results of the measured ^{210}Po activity in IAEA-360 and the certified values of ^{210}Po activity in IAEA-360within 95% confidence interval. The recorded recoveries (analytical yield) obtained using ^{209}Po tracers in CRM and environmental samples were 85% - 98% thus; the efficiency of the new technique is a distinct optimised conditions for efficient and accurate recovery of 210Po from

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environmental samples in cost effective andreliable manner and should be promoted.

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