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Sequential analysis methodology for ^{210}Po and uranium analysis by extractive liquid scintillation spectrometry

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Abstract

A methodology for separation and purification of ^{210}Po from uranium, thorium and daughters has been studied. Solvent extraction coupled with liquid scintillation analysis using HDEHP (di-(2-ethylhexyl) phosphoric acid) and TOPO (trioctylphosphine oxide) in toluene as extractive scintillator was optimized. About 95% of ^{210}Po was extracted in the organic phase of TOPO as an extracting agent from an aqueous solution in 1 M HCl, compared to 1.6% extraction by HDEHP. The methodology was validated with uranium ore tailing sample, IAEA-385 sediment and IAEA-447 Moss-soil reference materials for application to environmental samples. A minimum detectable activity of 118 mBq kg^{-1} was achieved.

Keywords ^{210}Po · TOPO · HDEHP · Liquid scintillation spectrometry · Solvent extraction

Introduction

^{210}Po ($t_{1/2}$ = 138 days) is the decay product of ^{210}Pb ($t_{1/2}$ = 22.3 years) via ^{210}Bi ($t_{1/2}$ = 5 days) in the ^{238}U decay series and widely distributed in the earth's crust, rivers, oceans, and the atmosphere. It is considered to be the most radioactive and radiotoxic amongst the 'naturally occurring radionuclides' (NORMS). Natural concentration of polonium in environment can be enhanced due to human activity like industrial growth, mining, fossil fuel combustion, phosphate fertilizers in agriculture, domestic and industrial sewage [1]. The contribution of ^{210}Po towards internal dose to human from NORMS has been estimated to be around 8% [2]. It thus becomes important to estimate activity of ^{210}Po in different environmental matrices and its transfer through the human food chain. The most widely used technique for estimation is the spontaneous deposition of polonium on a silver disc [3–6]. This method is subject to interference from oxidants, organic materials, and matrix elements that also deposit on the silver plate. These interferences can be removed by co-precipitation and separation of polonium. The most preferred method includes chemical separation of ^{210}Po from interfering radionuclides and matrix elements, prior to its deposition. Vajda et al. [7] simultaneously determined ^{210}Pb and ^{210}Po in a range of matrices including soils, sediments, and biological samples by solid phase extraction using Sr resin in hydrochloric acid solution. ^{210}Po activity was determined via LSS (liquid scintillation spectrometry) where the quench level of the sample was affected by the high acid concentration, however, if the quench is maintained low, the major advantage offered by liquid scintillation is the high counting efficiency. About 100% counting efficiencies are obtained for β emitters with energies above 100 keV and α emitters, such as ^{210}Bi and ^{210}Po respectively. Katzlberger et al. [8] determined ^{210}Pb, ^{210}Bi and ^{210}Po in natural drinking water by separating ^{210}Pb from water sample via sulphide precipitation and subsequent liquid–liquid extraction of bismuth and polonium using Polex™, an extractive liquid scintillation cocktail, from a phosphoric acid with about 90% extraction yield. L. Jokelainen et., al, [9] reported the use of three extractive scintillation cocktails, POLEX™, TOPO (trioctylphosphine oxide) and TNOA(Trioctylamine) for ^{210}Po analysis from groundwater samples. The main interfering nuclides were ^{234}U and ^{238}U, which led to incorrect results in ^{210}Po analysis, due to the co-extraction of uranium from real ground waters. In the present study, the condition for ^{210}Po recovery with 75 g L^{-1} HDEHP (di-(2-ethylhexyl) phosphoric acid) and TOPO (trioctylphosphine oxide) in toluene scintillator has been studied under varying HCl acidity conditions and optimized. The quality and reliability of

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analytical methods are of significance in the assessment of any validated analytical methods [10]. Studies for standardization of the methodology for ^{210}Po analysis using solvent extraction have been carried out using distilled water samples and validated using materials representing the different environmental matrices, like a uranium ore tailing site sample, IAEA certified reference materials viz., IAEA-385, Irish Sea sediment and IAEA-447, Moss-soil.

Materials and methods

The standard solution of uranium (30 Bq mL^{-1}) and ^{210}Pb in equilibrium with ^{210}Po (90 Bq mL^{-1}) used for spiked experiments were obtained from bureau international des Poids et mesures (BIPM), France. Working solutions were prepared by transferring a known weight of the standards followed by volumetric dilution to an appropriate working concentration. The extractant HDEHP, TOPO, naphthalene, 2,5-diphenyl-oxazole benzene (PPO), 1,4-bis (2,5-diphenyl-oxazole) benzene (POPOP), diethylenetriaminepentaacetic acid (DTPA) were obtained from Merck.

Ultra low level Quantulus 1220 LSS (Finland) was used for the measurement. It is equipped with pulse shape analysis (PSA) circuit for simultaneous quantization and discrimination of alpha and beta particles in the same sample depending on the decay time of the light pulses they produce in the liquid scintillator. Pulse shape discrimination is accomplished using a software adjustable parameter, PSA which can vary between 1 and 256 to categorize the pulses as either α events or β events and stores the events appropriately in separate multichannel analyzers (MCAs).

Preparation of extractive scintillator

Toluene based scintillator was prepared by dissolving 7 g PPO, 0.5 g POPOP and 200 g naphthalene in 1 L toluene. Extractive scintillator was prepared with two different extractants 75 g HDEHP and 75 g TOPO in toluene scintillator (7.5% V/V). Extraction recovery of ^{210}Po spiked in aqueous solutions (10 mL) with varying HCl concentrations (0.1-1.2 M) using two different extractants 0.75 M TOPO and HDEHP in toluene scintillator was studied as shown in Fig. 1. The sequential separation of $U_{(nat)}$ and ^{210}Po was carried out based on previous studies [9] and suitable extractant as observed from the current study.

Method

Standardisation of HCl concentrations for ^{210}Po extraction using TOPO and HDEHP extractant in toluene scintillator

Two sets, containing series of 10 mL distilled water were spiked with ^{210}Po standard solution (8 Bq) and they were adjusted to different concentrations of 0.1, 0.2, 0.5, 0.7, 1 and 1.2 M L^{-1} of HCl. This was transferred to separating funnels and 10 mL TOPO as extractive scintillator were added to one set and 10 mL HDEHP extractive scintillator to the other. The funnels were shaken at room temperature for few minutes and the solutions were allowed to separate. Under this condition ^{210}Po is expected to get transferred to the organic phase [11]. Following phase separation, the organic phase was sparged with argon gas for ^{222}Rn removal and reduction of chemical quenching by oxygen, for improving the pulse shape discrimination and energy resolution. Finally 5 mL aliquot of the organic phase containing ^{210}Po was counted in LSS for 1000 min at the optimized PSA [12]. Figure 1 shows the variation of ^{210}Po recovery under various HCl concentrations along with TOPO and HDEHP as extracting agents in toluene scintillator.

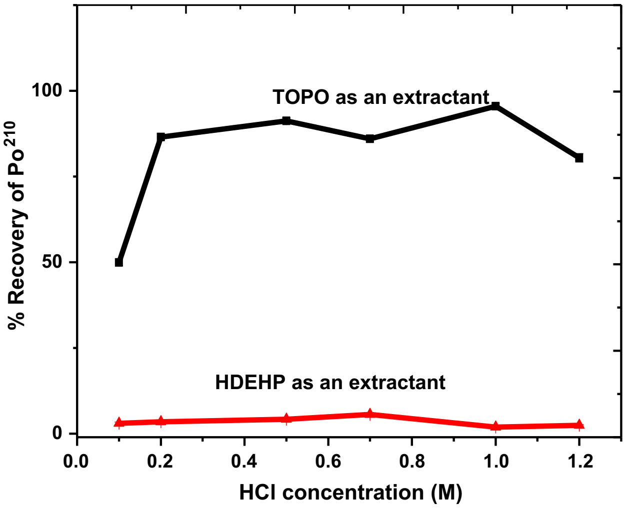

Fig. 1 Efficiency of ^{210}Po extraction by 0.75 M TOPO and HDEHP in toluene scintillator

Effect of extractant concentration (TOPO) on recovery of ^{210}Po

Experiments were carried out using different concentrations of TOPO ranging from 15 to 100 g L^{-1} in toluene scintillator, to optimize its concentration in the extractant. Distilled water was spiked with 10 Bq ^{210}Po activity and the above mentioned extraction procedure was repeated. Figure 2 shows the extraction efficiency of Po under different TOPO concentrations.

Selective separation of ^{210}Po and Uranium (U)

Pilot experiments were conducted to examine the extraction of $U_{(nat)}$ in the presence of ^{210}Po in aqueous phase at 1 M L^{-1}

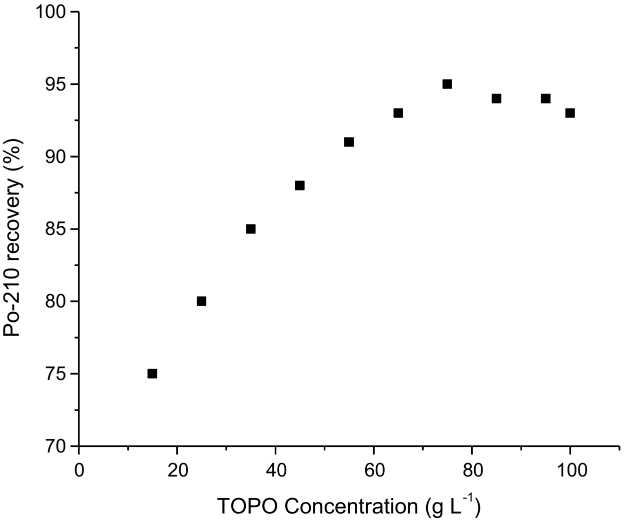

Fig. 2 ^{210}Po extraction recovery under various TOPO concentrations

HCl with TOPO as extracting agent. 10 mL of distilled water was spiked with $U_{(nat)}$ (3.17 Bq) and ^{210}Po (8 Bq) activity. 10 mL of the extractive scintillator was added and the procedure described above was repeated. An aliquot of the organic phase was counted in a LSS and spectrum is shown in Fig. 3 which is a composite spectrum of $^{238+235+234}$U and ^{210}Po as a result of solvent extraction indicating that $U_{(nat)}$ and ^{210}Po were transferred into the organic phase and that U also gets extracted along with ^{210}Po under the above mentioned conditions. Therefore, above mentioned solvent extraction procedure was modified to sequentially isolate $U_{(nat)}$ and ^{210}Po. To standardise this, 10 mL aqueous solution in 1 M HCl was spiked with $U_{(nat)}$ (3 Bq) and ^{210}Po (9 Bq) and subjected to solvent extraction with 10 mL toluene scintillator with HDEHP as an extracting agent with a few minutes equilibration time. Under this condition, U was expected to be transferred to the organic phase and ^{210}Po remain in the aqueous phase. 10 mL of the aqueous phase was decanted into another separating funnel and subjected to solvent extraction again 10 mL toluene scintillator with TOPO as an extracting agent for a few minutes.^{210}Po was expected to be transferred to the organic phase which was counted by LSS. Figure 4a, b shows the spectra when $^{238+235+234}$U and ^{210}Po are present individually and not interfering after sequential separation.

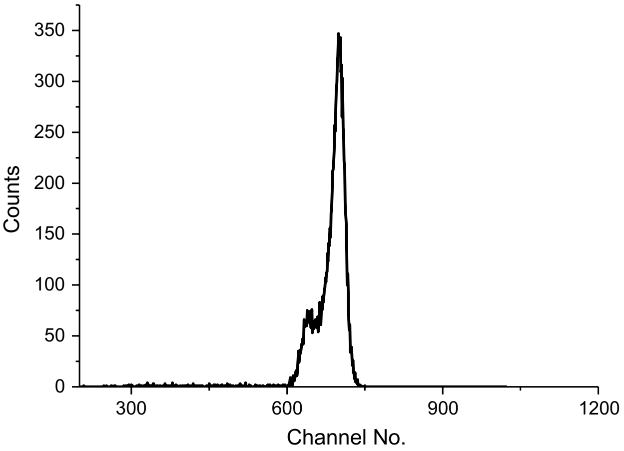

Fig. 3 Composite spectrum of U and ^{210}Po with only TOPO extraction

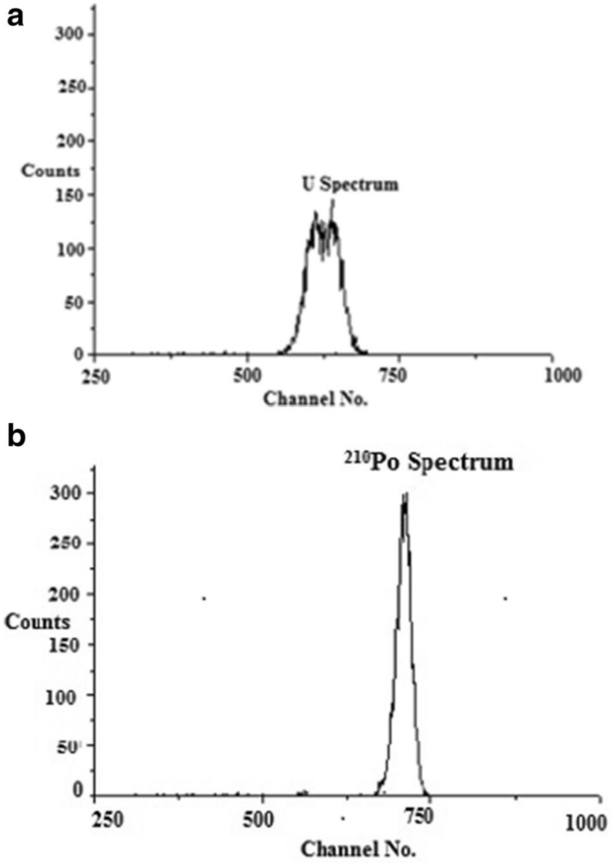

Fig. 4 **a** U and **b** ^{210}Po individual spectra after sequential separation

^{210}Po solvent extraction in the presence of ^{226}Ra

^{226}Ra is the decay product of naturally occurring radionuclide U which is present in almost all matrices of the environment along with ^{210}Po. Solvent extraction of ^{210}Po also quantitatively extracts ^{226}Ra, an α emitter which causes interference in the estimation of ^{210}Po. An aliquot of 1 mL ^{226}Ra standard (5.5 Bq) along with its daughters were evaporated to dryness with repeated addition of 1 mL concentrated HCl under an IR lamp. After complete

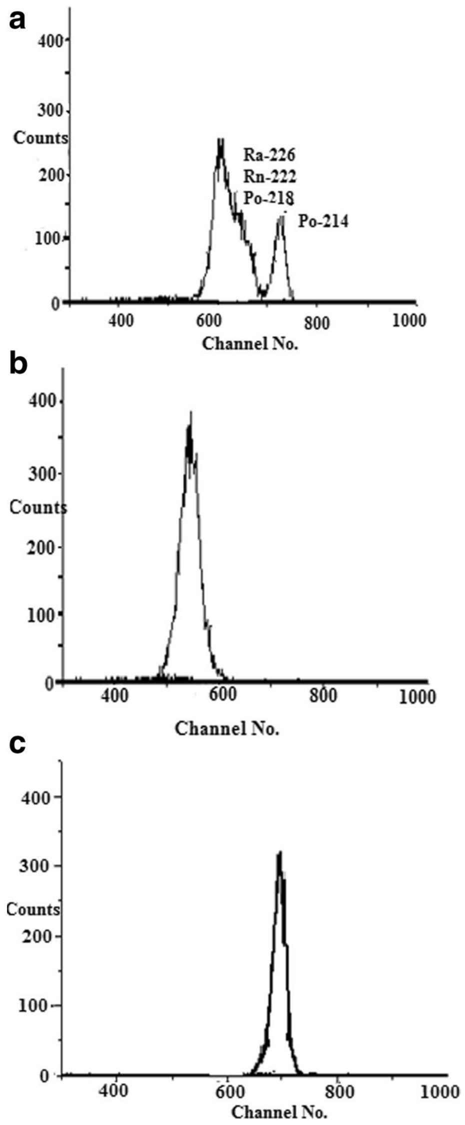

Fig. 5 **a** ^{226}Ra spectrum with ^{222}Rn and its short lived daughters after solvent extraction. **b** ^{226}Ra spectrum after removal of short lived daughters by complete evaporation. **c** ^{210}Po spectrum after solvent extraction ^{226}Ra

evaporation, scintillation cocktail was added to one set and LSS measurement was carried out. Figure 5b depicts ^{226}Ra spectrum after removing its short lived daughters by evaporation. Spectrum (a) is the case when radon is not purged from Ra and (c) is the case when ^{226}Ra and ^{210}Po both are present together. DTPA has been reported to act as a masking agent and reduces interference of ^{226}Ra in $U_{(nat)}$ [12] estimation and was used in the current studies to eliminate ^{226}Ra interference in ^{210}Po measurement. In order to standardize method for removal of ^{226}Ra interference an aliquot of 1 mL ^{226}Ra standard (5.5 Bq) along with its daughters and ^{210}Po was evaporated repeatedly to dryness and made into 10 mL 1 M L^{-1} HCl, followed by the addition of 1.5 mL 0.01 M DTPA as a masking agent. Solvent extraction was carried out using 10 mL TOPO based toluene extractive scintillator. After phase separation, 5 mL aqueous phase was collected in a 20 mL polyethylene vial for LSS measurement at optimized PSA and the spectrum was analysed to confirm complete removal of ^{226}Ra and ^{222}Rn. Figure 6 shows flow chart of the standardised methodology chalked out for sequential separation of $U_{(nat)}$ and ^{210}Po.

Validation of methodology

Certified reference materials including IAEA-385, Irish Sea sediment, IAEA-447, Moss-soil were analyzed for ^{210}Po using the optimized conditions. In addition, ore tailings sample from Jaduguda was also analyzed to validate the method. 1 g each of the samples was digested in Teflon beaker on a hot plate with concentrated mixture of 100 mL aquaregia. The procedure was repeated 3 times. The extracts from each leaching were collected and treated with perchloric acid for destroying the organic matter and volume of the samples were reduced by controlled heating on hot plate. The extracts were centrifuged at about 1500 rpm for 5 min and the supernatant was collected. The residual undissolved solid was washed 2–3 times, with 5 ml 0.1 M L^{-1} HCl and supernatant was added to the main extract. The cumulative supernatant was evaporated to near dryness at temperatures below 90 °C. The dry residue was dissolved and stock solution prepared in 1 M L^{-1} HCl. 10 mL aliquot from the stock solution of each sample containing was transferred to a separating funnel. 1.5 mL 0.01 M DTPA was added and the procedure described as above was followed. The activity concentration of ^{210}Po in the certified reference materials and ore tailing and other samples are presented in Table 1. Sediment samples were analysed by the conventional radiochemical method and also by the standard method developed in the current study. Figure 7 shows a typical spectrum from LSS of separated ^{210}Po from an ore tailing pond sample.

Minimum detectable activity (MDA)

The Currie equation given below provides an estimate of the MDA which is proportional to the standard deviation of the background activity for a specific volume of analyte/sample. Background sample was prepared similar to the real samples. MDA was evaluated using [13],

$$\mathrm{MDA}(\mathrm{Bq\,mL^{-1}}) = \frac{2.71 + 4.65\sqrt{C_\mathrm{B}}}{\mathrm{VET}} e^{\lambda \Delta t} \quad (1)$$

where C_B = Counts in ^{210}Po region of interest for blank when counted for time T, T = counting time (min); V = volume

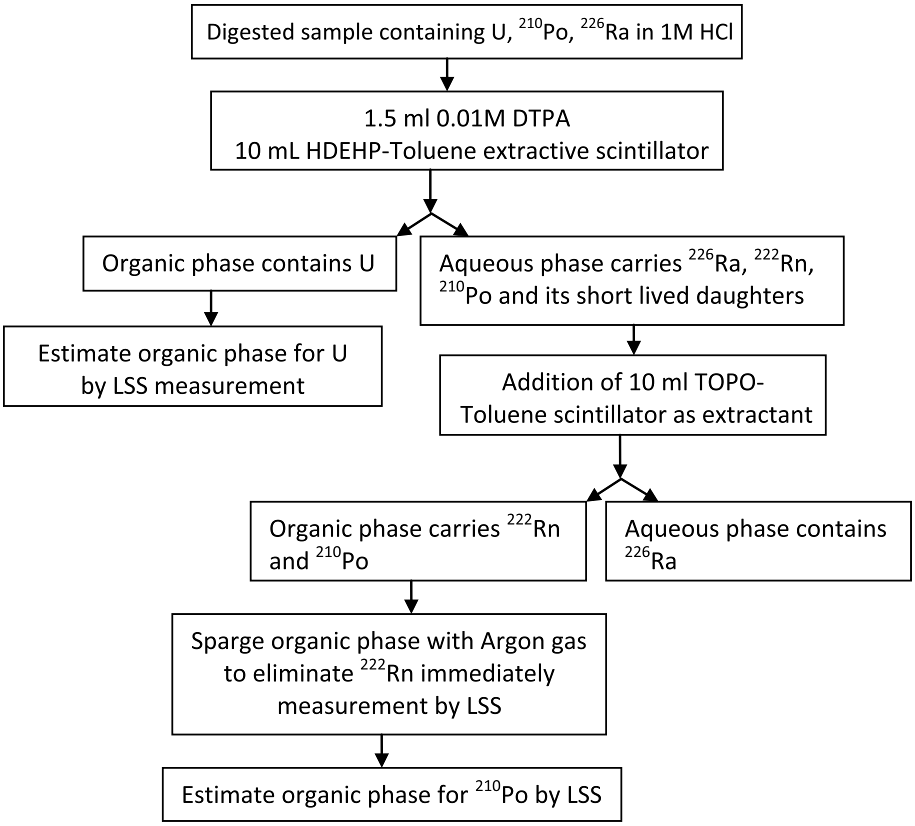

Fig. 6 Schematic procedure for sequential separation of U and ^{210}Po

Table 1 Activity of ^{210}Po in certified reference and experimental materials

Sample	Certified Activity (95%confidence) (Bq kg^{-1})	Experimental ± σ (Bq kg^{-1})
IAEA-385, Irish Sea sediment	32.9 (31.2–35.3)	35.0 ± 1.6
IAEA-447, Moss-soil	423 (413–433)	395.0 ± 7.8
Ore Tailing, Jaduguda	15	14.3 ± 0.1

Samples	α Spectrometry	LSS method
Sediment 1	31.8 ± 4.1	33.5 ± 5.1
Sediment 2	43.6 ± 5.7	44.9 ± 6.7
Sediment 3	41.5 ± 5.4	43.1 ± 6.4
Sediment 4	45.5 ± 5.9	47.3 ± 7.1
Sediment 5	53.6 ± 6.9	55.2 ± 8.3
Sediment 6	53.1 ± 6.1	55.3 ± 8.3

(mL)/weight of sample analyzed, E = counting efficiency; Δt = time delay between the ^{210}Po separation from its parent to the time of counting.

MDA of 118 mBq kg^{-1} based on a background of 50 counts for 60,000 s counting time for α peak for about 1 g of sample.

Results and discussion

The possibility of using toluene based scintillator in combination with complexing extractants like TOPO and HDEHP for the separation and extraction of ^{210}Po from U and ^{226}Ra has been studied.

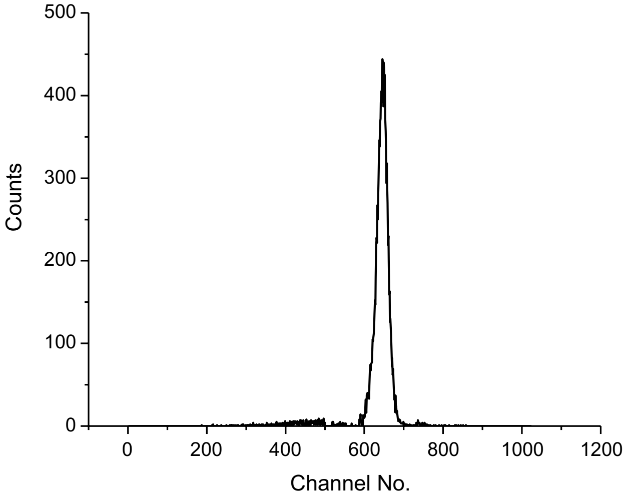

Fig. 7 ^{210}Po spectrum for ore tailing, Jaduguda

Effect of HCl concentration on ^{210}Po using TOPO and HDEHP extractants

Figure 1 illustrates the recovery of ^{210}Po under various HCl concentrations using TOPO and HDEHP in toluene scintillator as extractants. It is clear that as the concentration of HCl is increased from 0.1 M to 1 M ^{210}Po recovery increases and falls at higher concentrations. It was observed that only about 1.6% of spiked ^{210}Po was extracted from the aqueous phase under same HCl concentrations with HDEHP as an extractant. Therefore, TOPO has superior extraction capability (95%) for ^{210}Po at 1 M HCl extraction than HDHEP.

Effect of extractant concentration

The extraction behaviour of Po under various TOPO extractant concentrations is illustrated in Fig. 2. ^{210}Po extraction efficiency in the organic phase increased from 75 to 95% for TOPO concentration from 15 to 75 g L^{-1}and then decreased with further increase in the TOPO concentration from 85 to 100 g L^{-1}. Maximum extraction efficiency of about 95% is observed at 75 g L^{-1} of TOPO and 1 M HCl. After that no further increase in the efficiency is observed.

This is because of two factors: the concentration gradient of ^{210}Po-complex and the viscosity of the organic phase. As reported by Dzygiel et al. [14], the flux (J) of the species through an interface layer of thickness (dl) is related to its diffusion coefficient D and concentration gradient (dc) by Ficks law:

$$J = -D\frac{dc}{dl} \tag{2}$$

From the above equation it is clear that high flux can be obtained when high concentration gradient and diffusion coefficient are maintained. But diffusion coefficient depends on viscosity of the extractant in the organic solvent (η) at temperature, T and the radius (r) of the migrated species according to Stokes–Einstein relation:

$$D = \frac{KT}{6\pi\eta r} \tag{3}$$

Using above two equations, the relation between viscosity and concentration gradient of TOPO is obtained as:

$$\frac{dc}{dl} = \left(\frac{-6\pi rJ}{KT}\right)\eta \tag{4}$$

Therefore an increase in TOPO concentration results in increase of the complex flux however at concentrations above, 75 g L^{-1}, the viscosity of the solution increases and might retard the transfer of Po-TOPO complex at the interface due to increase in layer thickness. Thus by increasing the concentration, the amount of ^{210}Po complex that could be extracted into the organic phase tends to increase only up to a certain extent [15].

Selective separation of ^{210}Po from U$_{(nat)}$ and ^{226}Ra

As uranium is ubiquitously present in natural environmental matrices it is expected to interfere in the measurement of ^{210}Po along with its daughters like ^{226}Ra, ^{210}Pb, ^{222}Rn etc. To confirm the selective separation and extraction of polonium in the presence of uranium and radium was conducted. TOPO based extractive scintillator at 1 M HCl was used for extraction and organic phase was counted. Radiochemical separation indicated that about 80% U was co-extracted along with ^{210}Po under this condition. Broader α peak in the spectrum also indicated the extraction of $^{238+235+234}$U with ^{210}Po. Figure 3 shows the spectrum of the organic phase having $^{238+235+234}$U and ^{210}Po both. Based on the results from α measurement with LSS, the peak between channels 600 and 650 in the LSS spectrum was of $^{238+235+234}$U isotopes and α peak, between 650-750 channels corresponded to ^{210}Po. As the peaks were close by they could not be resolved. Therefore, above mentioned solvent extraction procedure was modified to remove uranium and its daughters. The procedure illustrated in Fig. 6 was followed for sequential separation of $^{238+235+234}$U and ^{210}Po. The separation and extraction recovery for spiked ^{210}Po and $^{238+235+234}$U in TOPO in toluene organic phase was 95 and 0.4% respectively. Remaining 99.6% $^{238+235+234}$U in the aqueous phase was recovered by extracting with HDEHP.

Figure 4a, b illustrates sequentially separated $^{238+235+234}$U and ^{210}Po spectrum obtained from α measurement indicating successful isolation of ^{210}Po. The extraction process was repeated to ensure complete separation and good recovery. Mass balance of the spiked activity confirmed the efficient recovery of individual elements and the purity of separated elements was confirmed by α spectrometry.

During the experiments it was observed that ^{226}Ra was also getting co-extracted with ^{210}Po under the optimized conditions. Also sometimes ^{222}Rn and its short-lived daughters if present in the sample or in radioactive equilibrium with ^{226}Ra were interfering and their peaks were obtained in the LSS spectrum. An α peak observed between channels 700 and 780 (Fig. 5a), is from ^{214}Po, a daughter nuclide of ^{222}Rn with an α energy greater than 7 MeV. In order to remove the interfering α nuclides, ^{222}Rn and its daughters and to determine ^{226}Ra interference, two aliquots of 1 mL ^{226}Ra standard (5.5 Bq) in equilibrium with its daughters was evaporated to dryness with repeated addition of 1 mL concentrated HCl under a heat lamp. After complete evaporation, scintillation cocktail was added to one set and LSS measurement was carried out. Figure 5 b demonstrates spectrum obtained after α measurement of the first aliquot indicating that no other α radionuclide besides ^{226}Ra. After ^{222}Rn removal, no ^{214}Po peak was found in the spectrum, which confirmed the removal of ^{222}Rn and its short lived daughters. Hence, to estimate ^{226}Ra interference in ^{210}Po measurement, the second aliquot was spiked with ^{210}Po activity (5 Bq) after complete dryness containing only ^{226}Ra. 10 mL.

1 M HCl was added followed by 1.5 mL 0.01 M DTPA as a masking agent. It was also subjected to solvent extraction by the addition of 10 mL TOPO based toluene extractive scintillator. After phase separation, 5 mL organic phase was measured. Based on the measurement 95% ^{210}Po and the spectrum it was confirmed that ^{226}Ra and ^{222}Rn was successfully separated from ^{210}Po (Fig. 5c).

Validation of method using Certified Reference Materials

Sequential extraction procedure as illustrated in Fig. 6 was followed to separate and extract polonium in the organic phase in the presence of interferences. The reliability of the optimized method has been checked by comparing the results of the ^{210}Po analysis, performed on samples containing known values of ^{210}Po with certified values.

The measured activity concentrations of the certified reference materials and sediment samples are shown in Table 1. The measured activity of ^{210}Po in IAEA-385, Irish Sea sediment and IAEA-447, Moss-soil was found to be 35.0 ± 1.6 and 395 ± 7.8 Bq kg^{-1} respectively. While the certified values were 32.9 and 423 Bq kg^{-1} respectively. The results show good agreement between the measured and certified values of IAEA-385 and IAEA-447 reference materials. Figure 7 shows ^{210}Po spectrum of ore tailing indicating no interference from U$_{(nat)}$ and its daughters.

Ore tailing sample from Jaduguda was observed to contain markedly elevated activity of ^{210}Po 14.3 ± 0.1 Bq kg^{-1}. The ^{210}Po in the sediment samples measured by the conventional method and the current procedure of extractive LSS are comparable.

Conclusion

The present study reports the optimized condition for selective separation and extraction of ^{210}Po from environmental samples. TOPO proved to have superior extraction capability (95%) over HDEHP towards ^{210}Po under 1 M HCl concentration. About 0.4% interference of $^{238+235+234}$U and 1.43% of ^{226}Ra was observed in estimating ^{210}Po by solvent extraction. The method was standardised by adopting two extractants, initial HDHEP step to separate uranium and a second extraction with TOPO to separate ^{210}Po and eliminate interferences due to naturally occurring radium isotopes, radon and daughters. The results indicate good agreement between the measured and certified values of IAEA certified materials and ore tailing, Jadugoda within 7% deviation. It was found to be suitable for estimation of uranium and ^{210}Po in matrices like sea water, sediment and biological samples.

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