J Radioanal Nucl Chem (2014) 302:673–678
DOI 10.1007/s10967-014-3255-2

Effects of digestion, chemical separation, and deposition on Po-210 quantitative analysis

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Received: 4 April 2014 / Published online: 8 June 2014

Abstract The purpose of this work was to determine polonium losses from a variety of sample types (soil, cotton fiber, and air filter) due to digestion technique, chemical separation, and deposition method for alpha energy analysis. Results demonstrated that yields from a perchloric acid wet-ash (87 ± 5 %) were similar to that from a microwave digestion (100 ± 7 %), but both were greater than the dry-ash procedure (38 ± 5 %). The polonium was separated from an SRM soil using an AG1X8 ion exchange column and deposited on a Ag disk with a recovery of 83 ± 7 % of polonium-209 (Po-209). Deposition yields without chemical separation averaged 90 ± 5 % of Po-209. The polonium-210 content was successfully measured in the three matrix types and quantitated using alpha spectroscopy.

Keywords Digestion · Po-210 · Alpha spectroscopy

Introduction

Polonium-210 (Po-210) is a decay product of the U-238 natural decay chain and has a half-life of 138.376 days. Due to its presence in the U-238 natural decay chain, Po-210 is often included in environmental studies related to sedimentation processes, food chains, aerosol behavior, atmospheric circulations [1–7]. Po-210 is also one of the most radiotoxic isotopes to humans; therefore, studies have also been done to quantitate the amount of Po-210 that humans are exposed to from tobacco leaves [8, 9], cigarettes [10, 11], foods such as shell-fish [12], and potential uptakes that can be evaluated from blood [13] and urine [14]. Various techniques have been studied for the separation of transuranic elements such as Po-210 from Pb-210, the parent isotope, in order to understand environmental contributions from each towards natural radiation doses and activities [15, 16]. The separation of Po-210 from Pb-210 has also been investigated so as to reduce radiation backgrounds for low-level detector systems [17, 18]. A thorough review of the literature associated with Polonium (Po) analysis was done by Matthews and co-workers in 2007 [19].

Polonium-210 is hard to detect from a raw, unprocessed sample because it decays only by an alpha emission (5.304 MeV, 100 %). Digestion, chemical separation and thin layer deposition processes are often required for the isotopic analysis; however, the polonium is often lost in the initial digestion because it has a relatively low volatilization temperature, 180 °C [20, 21]. Previously, a study quantitated polonium losses during a wet ash of multiple organic substances using a nitric/hydrochloric wet-ash in an open beaker, a Kjekdahl flask, and a closed-vessel microwave [22]. This work compares these results to other common digestion techniques, such as a dry-ash, an all-in-one microwave-evaporation method, and a perchloric acid wet-ash. For each digestion method investigated, a typical material (matrix) for that digestion technique was selected for comparative purposes.

In conjunction with the losses of Po from digestion, our work also investigated losses associated with the chemical separation of Po from a complex matrix, and the spontaneous deposition of Po on Ag for quantitative alpha energy analysis. Using the Polonium-209 (Po-209) tracer to determine Po recovery, a concentration of Po-210 in each sample type was calculated.

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Table 1 CEM MARS 5 soil digestion program settings

Step	Power (watts)	Ramp (min)	Pressure (Pa)	Temp (°C)	Hold (min)
1	1,600 (100 %)	3.5	2×10^6	170	0:00
2	1,600 (100 %)	3.5	2×10^6	180	30:00

Table 2 Summary of sample composition for chemical separation

Matrix (10 mL)	Po-209 (Bq)	SRM digest (mg)	H_3BO_3 present (μg)	Replicates
3 M HNO_3	0.185	0	0	4
3 M HNO_3	0.185	0	3.8	4
3 M HNO_3	0.185	1	0.38	4
3 M HNO_3	0.185	10	3.8	4
3 M HNO_3	5	100	38	4

Materials and methods

Sample digestions

Standard reference material (SRM), Montana I Soil (SRM 2710a) was purchased from the National Institute of Standards and Technology (NIST). For each soil digestion, 1 g of SRM soil with and without 0.185 Bq of Po-209 ($n = 3$) were added to CEM MARS-5 microwave vessels and used a procedure developed by Douglas et al. [23]. To each vessel, 9 mL of conc. HNO_3, 3 mL of conc. HCl, and 7 mL of conc. HF were added. Chemical blanks were done in parallel with the SRM soils. All acids were of Optima grade purchased from Fisher Scientific. Samples were processed through the microwave digestion program shown in Table 1. The digested samples were then evaporated to dryness using the evaporation unit that is part of the CEM MARS-5 system, reconstituted in 15 mL of 3 M HNO_3–0.3 M H_3BO_3 and processed again through the microwave digestion procedure described in Table 1. The final solution was filtered through a 0.2 μm syringe filter and calculated to be a concentration of 66 mg soil/mL.

Three cotton swipes (Texwipe ® TX304, 4″ × 4″) were stippled with 0.37 Bq of Po-209 and allowed to dry at room temperature. One control of the same cotton material not traced with Po-209 was digested at the same time. Each swipe was placed in a 100 mL glass beaker and covered with a watch glass. The samples were muffled at 500 °C for 10 h using a Vulcan furnace. Once cool, the ash was dissolved in 4 mL of aqua regia. This solution was evaporated to dryness on a hot plate and the aqua regia treatment repeated once before transposing the solution to a chloride matrix using 3–1 mL aliquots of concentrated HCl. The final solution was brought up in 5 mL of 0.5 M HCl. One mL of the sample was spontaneously deposited on Ag disks, while 4 mL of the sample was analyzed by liquid scintillation for yielding of the digestion process.

The air filter, IPC-1478, was designed for sampling at high altitude and high velocities [24]. Three of these filters were stippled with 0.37 Bq of Po-209 and one filter control that was not stippled with Po-209 were folded and placed in the bottom of a 1 L glass beaker, wetted with 100 mL of concentrated HNO_3 and 50 mL of fuming HNO_3. Three teflon beads were added and the beakers were covered with ribbed watch glasses and allowed to sit for 2 days. The beakers were then placed on hot plates and heated gradually to allow the filter material to react with the acid. Intermittent heating over the next week reduced the acid volume which was replaced by washing down the sides of the beaker with concentrated HNO_3 or adding fuming HNO_3. Once the volume was reduced to 20 mL, $HClO_4$ was added and heated at a higher temperature to react with the residual organic material. The volume was reduced to 5 mL and then transferred to 100 mL glass beakers using 3–2 mL additions of conc. HCl. This solution was treated with 3 mL of concentrated $HClO_4$ and brought to dryness. At this time, some colored residue was present. The residue was dissolved in 3 mL of 0.5 M HCl and filtered using a 0.2 μm syringe filter.

Yielding of digestion techniques

An aliquot of each sample digestate and controls were analyzed by liquid scintillation counting (LSC). A detector control sample that contained a known amount of Po-209 in the same sample matrix as the digestate was analyzed to determine detector efficiency. The samples were corrected for background based on the activity measured in the controls and then divided by the activity originally added. Chemical blanks were also analyzed by LSC for the microwave digestion but were determined to be less than the background (5 CPM) and were therefore not corrected for in the samples.

Chemical separations using anion exchange

Samples were prepared that contained 0.185 Bq of Po-209 and varying amounts of digested SRM (0, 1, 10, or 100 mg). During the soil digestion, significant quantities of H_3BO_3 were used; therefore, a matrix blank containing no soil, but 3.8 μg of H_3BO_3 along with the Po-209 was prepared. Each sample set prepared in quadruplicate. A summary of the sample sets is shown in Table 2. Each sample was loaded onto a preconditioned BioRad AG1-X8 anion exchange column (2 mL pre-packed) in 3 M HNO_3. The column was rinsed with 10 mL of 3 M HNO_3, and the Po eluted using 10 mL of 10 % thiourea.

Spontaneous deposition of polonium

Spontaneous deposition was accomplished using Ag disks (2.54 cm outer diameter, 0.03 cm thick). The disks were cleaned using a hot solution of $NaHCO_3$ and NaCl in water. Once cleaned and dried, each disk had one side covered with teflon tape. The prepared disk was placed into the beaker containing the final solutions which contained 0.5 M HCl and 10 % thiourea (for samples eluted from the anion exchange). The solution with the disk was heated between 90 and 95 °C and stirred for 2 h. After 2 h, the disks were rinsed with water and allowed to air-dry.

Alpha detection

Spontaneously deposited samples on the Ag disks were counted for 4 days using Canberra's Alpha Analyst^{TM} integrated alpha spectrometer equipped with Canberra's Passivated Implanted Planar Silicon detectors with 450 mm^2 active surface areas. All data was acquired and analyzed with Apex-Alpha^{TM} software suite. Regions of interest were manually chosen for each spectrum and count data was background subtracted and reported without correcting for individual detector efficiency. The 2.54 cm diameter disks were not calibrated with the alpha detection system; therefore, a standard was prepared by stippling 0.185 Bq of Po-209 on a 2.54 cm stainless steel disk with the same thickness. This standard was used to determine detector efficiency 12.8 % for the analyzed geometry. The CPM was determined for the standard and the samples. The percent yield was based on the sample's CPM divided by the standard's CPM. This method assumes no loss of Po-209 in the production of the standard.

Results and discussion

The goal of this work was to determine the Po loss using three digestion techniques with three common sample types of soil, cotton swipe material, and air filters. Each material was spiked with a known amount of Po-209 prior to digestion, and the recovery post digestion was determined based on LSC analysis. The SRM 2710a, Montana I Soil, was digested using a CEM MARS-5 microwave digestion–evaporation system and the recovery of the Po-209 was 100 ± 7 %. The yield from the microwave was high and is attributed to the closed system microwave and the built-in evaporation system used. The microwave system by CEM allows for 12 different reaction vessels to be digested or evaporated at one time. During the evaporation process, one of the vessels is monitored for temperature, and the assumption is the other vessels behave the same way. As the volume of liquid in the monitored vessel decreases, the temperature reading of the thermometer decreases, when the change in the temperature exceeds 20 °C, the microwave system shuts down. There is a potential for additional loss if all the vessels do not dry at the same rate and some vessels get hotter than others. This is not expected to have occurred since all samples were prepared in the same fashion. Previous work by Henricsson et al. [22], used a similar microwave digestion technique on various organic materials (i.e. leaves, lichen, peat, and algae), and determined an average polonium recovery of 82.3 ± 7.6 %. Rather than using the built in evaporation chamber provided by CEM, Henricsson used a hot plate to control the evaporation temperature for each of the samples prepared for alpha deposition. Our method using the built in evaporation chamber potentially provides a more rapid digestion with better yields of Po, assuming all samples behave similarly under the evaporation chamber. The quantitative yields of sediment digestion using a microwave system was also observed by Sanchez-Cabeza and co-workers [25] using a slightly different but similar combination of acids.

The cotton swipe was digested using a dry-ash followed by an acid dissolution of the ash. This process caused the highest loss of Po with a Po recovery of 38 ± 5 %. This high loss is expected since the muffling process exceeds 500 °C in an open system, which is well above the 180 °C volatilization temperature of Po. These results are consistent with reports by Cleary and co-workers that dry-ashed rat tissues and found that Po can be lost at temperatures as low as 100 °C and full volatilization occurs around 800 °C [26].

The air filter sample was digested using a nitric acid and perchloric acid wet-ash. Although this digestion technique can be rigorous, the solution is maintained and refluxed around 200 °C and the reactions are well monitored. Therefore, the recovery of this method was much higher and consistent (87 ± 5 %) when compared to the dry-ash. Henricsson et al. [22] also investigated an acid wet ash in an open beaker using 65 % nitric acid, 37 % hydrochloric acid, and hydrogen peroxide. Recovery using this method averaged 69.3 ± 12.1 %, which is only slightly lower than the perchloric acid wet-ash used in this study. A similar study by Cunha and co-workers also digested marine sediment using a nitric acid-peroxide solution and deposition of Po onto Ag disks with yields of Polonium-208 of 44–63 % [27]. Several other studies have used acid digestions with minimal losses [19]; however, as is similar with this study, the time is often on the order of days to weeks.

Certain digestion techniques are only applicable for specific sample matrices; however, results from this study indicate the perchloric wet-ash or a closed-vessel microwave acid digestion system could provide limited polonium losses under the right conditions. The microwave system has the benefit of being rapid, but the perchloric acid wet-ash is able to be well monitored and can easily be adjusted for larger sample sizes.

Relatively clean sample types, such as filter papers or cotton swipes, do not necessarily require chemical clean-up prior to deposition of Po onto a Ag disk for alpha energy analysis. This spontaneous deposition technique has been commonly used for Po analysis for the last 40 years and has shown time and time again to be a reliable and high yield process [19]. Due to the simplicity of these samples, no reducing agents were added.

For complex sample matrices, such as soil, it is beneficial to have a chemical separation of Po-210 from other isotopes or elements that might interfere with alpha energy analysis. Common separation techniques include solvent extraction, extraction chromatography, and ion exchange chromatography [19]. Douglas et al. [28] developed a chemical separation of Au from a mixed activation/fission product sample using anion exchange This method demonstrated that Po can be eluted in the same fraction as Au with minimal contamination from other elements and in a small volume of only 10 mL. This is more ideal than previous studies that have used ion exchange and had large elution volumes [6, 29, 30]. This method was applied to the samples with the digested soil to separate Po from the multiple elements present that could potentially spontaneously deposit along with the Po, for example Fe. The recovery of the Po-209 from the soil samples through the chemical separation and spontaneous deposition was 83 ± 7 %. When combined with the loss from the digestion, this method recovered 83 ± 9 % of the original Po-209 on average. This value was used to calculate the amount of Po-210 measured in the SRM at each of the soil masses (i.e. 1, 10, and 100 mg). The Po-210 activity per gram of soil was calculated for each soil mass. The 1 mg soil sample had a high uncertainty due to the very low mass of soil (four orders of magnitude less than what is recommended by NIST for concentration analysis) and was therefore not used in the calculation of the average activity per gram. The samples containing 10 and 100 mg of soil digest were averaged and the activity per gram was determined to be 0.14 ± 0.06 Bq/g. Along with the soil samples, chemical blanks were also processed through the microwave digestion, chemical separation, and deposition ($n = 4$). The Po-210 content was below the minimum detection amount (MDA) of the alpha detector and was calculated to be <0.2 mBq. Samples were not corrected for the chemical blanks because it cannot be assumed that the chemical blanks would behave in the same way as the samples, particularly in the digestion process. Therefore, the quantitation of Po-210 would be less than the reported value. The certificate for SRM 2710a did not provide a Po-210 activity or concentration, therefore, no comparison was available.

Table 3 Percent yield (%) of Po-209

	Digestion	Chemical separation and deposition	Deposition only	Total yield
SRM 2710a	100 ± 7	83 ± 7	N/A	83 ± 9
Cotton swipe	38 ± 5	N/A	87 ± 4	33 ± 5
Air filter	87 ± 5	N/A	92 ± 6	80 ± 7

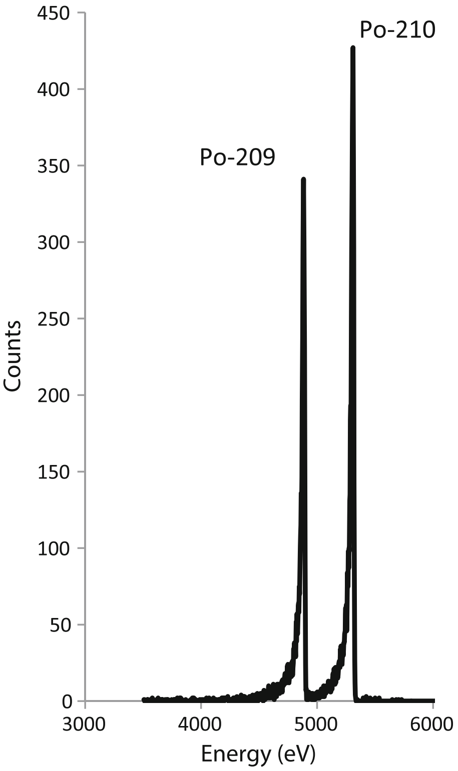

Fig. 1 Example alpha spectrum for Po-209/210

The cotton swipe and the air filter samples did not undergo chemical separation because there is limited potential for the presence of interfering isotopes in these samples. Therefore, after digestion these sample sets went directly to spontaneous deposition. The yield of Po-209 through the deposition was calculated based on the amount of Po-209 remaining after the digestion. Therefore the air filter, which was digested using the perchloric acid wet-ash had a yield with 92 ± 6 % and the cotton swipe, which was digested using the dry-ash, had a yield of 87 ± 4 %.

The total yield of Po-209 for each sample set was calculated based on the incremental yields from digestion, chemical separation (for soil samples only), and spontaneous deposition. These yields are summarized in Table 3. The soil sample and the air filter had similar yields; 83 ± 9

Table 4 Po-210 yield corrected concentrations

	Po-210 (Yield corrected, mBq/g)
Microwave chemical blank	<0.2 mBq
1 mg SRM 2710a	273 ± 50
10 mg SRM 2710a	126 ± 12
100 mg SRM 2710a	147 ± 60
Average for SRM 2710a[a]	137 ± 40
Cotton swipe	0.7 ± 0.3
Air filter	0.05 ± 0.01

[a] Average calculated using the 10 and 100 mg samples only

and 80 ± 7 %, respectively. The cotton swipe had an expected yield of 33 ± 5 %, with the majority of the loss being attributed to the high temperature dry-ash digestion.

Based on the total yield of Po-209 for each sample type, the activity of Po-210 was calculated using alpha energy analysis. The cotton swipe contained 0.7 ± 0.3 mBq/g, and the air filter contained 0.04 ± 0.02 mBq/g. This value is the total Po-210 contributed by the sample and the process. Because the digestion methods used the same high quality acids as the microwave digestion, the assumption is made that the chemical contribution of Po-210 from the wet-ash and dry-ash would also be below the MDA of the detector (<0.2 mBq). An example of the Po-209/210 alpha spectrum is shown in Fig. 1. The Po-210 concentrations are summarized in Table 4.

Conclusion

Polonium losses during digestion are minimized by using a controlled and well monitored perchloric acid wet ash or a closed-vessel microwave system. An open system dry-ash resulted in low Po recovery due to the high temperatures. Chemical separations using anion exchange and spontaneous deposition of Po on Ag have high yields and can be utilized for the low level detection and quantitation of Po-210.

Acknowledgments The work was supported by the Office of Defense Nuclear Nonproliferation (DNN), U.S. Department of Energy, and Pacific Northwest National Laboratory, which is operated by Battelle Memorial Institute for the U.S. Department of Energy under contract DE-AC05-76RLO1830. The team at PNNL gratefully acknowledges the assistance of Dr. Matthew O'Hara at PNNL for his support with the microwave digestion process.

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