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The Solid Speciation of ^{90}Sr, ^{137}Cs, ^{226}Ra, ^{238}U and $^{239,240}Pu$ in Surface Soils from Sites of Special Radiological Interest in Kazakhstan

M. Burkitbayev[1]*, M. Conway[2], H. Jimenez Napoles[2], Y. Kuyanova[1], L. Leyn Vintry[2],
P.I. Mitchell[2], N.D. Priest[3], L.Pourselot[4], P.Stegnar[5], B.Salbu[6] and B. Uralbekov[1]

[1]Department of Inorganic Chemistry, Al-Farabi Kazakh National University, Almaty, Kazakhstan

[2]School of Physics, University College Dublin, Belfield, Dublin 4, Ireland

[3]School of Health and Social Sciences, Middlesex University, Hendon, London, UK

[4]IRSN, BP3 Cadarache 13108 Saint-Paul-lez Durance, France

[5]Jozef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia

[6]Norway University of Life Science, Aas, PO Box 5003, Norway

Abstract

The solid speciation of some radiologically important radioelements has been examined in soils sampled at the former Semipalatinsk (Nuclear) Test Site (STS) and at Kurdai, the site of a former major uranium mining operation – both situated in Kazakhstan. Specifically, the partitioning of radiostrontium, radiocaesium and plutonium has been examined using sequential extraction on selected soils from the test site, while the partitioning of radium and uranium has been evaluated in soils at Kurdai. The data show that at the STS, in general, little (if any) of the radiostrontium, radiocaesium and plutonium is in an exchangeable form, with the great bulk of the radiostrontium and radiocaesium in a strongly bound or refractory form. In the case of plutonium, the proportion in a strongly bound or refractory form varies from 40% to 95% depending on site and appears to be a function of the explosive yield involved. At the Kurdai ore deposit, less than 5% of the uranium and radium is in an exchangeable form, with some 35-60% of the uranium and the bulk of the radium in a strongly bound or residual form.

Introduction

STS and Kurdai Site and History

The Semipalatinsk (Nuclear) Test Site (STS), located in the north-east of Kazakhstan, was the first and one of the main proving grounds for the testing of nuclear weapons and other nuclear devices by the former Soviet Union. The area is arid with a mean annual precipitation of about 300 mm, and the sandy soil and gravel that comprise most of it is course in texture and highly permeable. Grassland cover over much of the site is comparatively sparse and is punctuated by low bushes and scrub. However, it is suitable for grazing by domestic animals and is used by semi-normadic farmers. The depth to groundwater over much of the area is only 5-10 m and the potential for serious groundwater contamination is obvious.

In the period 1949-89 inclusive, a total of 456 nuclear tests were conducted at the STS. Approximately 25% of these tests were surface or air shots, while the remainder (75%) were conducted underground [1]. Testing at the site was largely carried out in four areas, known as Technical Areas Sh, B, G and M (Fig. 1). The early surface and air tests took place in the northern Technical Area Sh, within which is located Ground Zero, site of the first Soviet atomic test in August 1949. Following the signing of the limited test ban treaty in 1963, testing moved underground, mainly to Technical Areas G (Degelen Massif) and B (Balapan). The above total of tests includes four "cratering" explosions (Chagan, Tel'kem 1, Tel'kem 2 and Sary-Uzen) carried out in order to

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*corresponding author. E-mail: chemtech@kaznu.kz

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evaluate the potential of low-yield nuclear explosions for civil engineering purposes. Three of these cratering sites, Area Sh (Ground Zero), and Area 4A (a zone contaminated by ^{90}Sr as a result of the testing of radiation dispersal devices), are the focus of interest in the present study. Explosions with a total yield of 6.62 Mt are reported as having been carried out in Technical Area Sh [2], while for the cratering explosions the reported yields were 140 kt (Chagan), 0.75 kt (Tel'kem 2) and 0.25 kt (Tel'kem 1). These yield figures will prove to be of some importance in the discussion that follows (below).

Kazakhstan was a major supplier of uranium for the nuclear weapons and nuclear energy programmes of the former Soviet Union (USSR). It has been estimated that during the Soviet period about 40% of the total recovered uranium was extracted from hydrothermal and sedimentary deposits located in Kazakhstan. The long-term operation of uranium mining and milling enterprises produced a large volume of low-level radioactive waste in the form of rock spoil heaps, hydro-metallurgical plant tailings dumps, and basins of mine waters [4]. In recent years, these sites, usually located in proximity to settlements and main irrigation channels, have been the focus of concern amongst local populations and the subject of several international conferences.

Fig. 1. The Semipalatinsk Test Site, including locations at which soils were sampled in the present study (from: [4]).

The Kurdai U/Mo ore deposit, located in the south of the Kendyktas mountain ridge of the Zhambyl region is a case in point. This was the first industry-scale deposit to be exploited in Kazakhstan, following its discovery in 1951 by gamma survey. Over the operational period (1954-65) of ore extraction, about 6.28 million cubic meters of uranium contaminated waste in the form of rock spoil heaps was produced. These heaps and the open pit from which the ore had been extracted were subsequently abandoned for some decades. Today, the site lies partially remediated, in that spoil heaps have been re-aligned and covered with a layer of clay about a metre thick. However, the open pit from which uranium ore was extracted has remained in its abandoned state and is now filled with water, creating an artificial lake whose maximum extent is approximately 100 m (length) by 35 m (width). The nearest settlement (known locally as "Muzbel") is situated 600 m to the north of the former mine.

Purpose and Validity of Sequential Extraction Analysis

It is widely recognized that the mobility and bioavailability of a radioactive element in soil is dependent upon its physico-chemical form(s) and the characteristics of the soil itself. To define the remediation procedures necessary at sites such as those referred to above, as well as the long-term impact of residual radioactive wastes on the environs of these sites, knowledge of radionuclide partitioning and speciation in the soils and waters of these sites is an essential pre-requisite.

The most widely used approach for the determination of toxic element (including radioelement) partitioning in soils and, by extension, the soil phases responsible for toxic element fixation, is that of sequential chemical extraction, pioneered by Tessier *et al.* [5] in the late-1970s. Although many modifications of this original protocol have been applied and critically reviewed over the years [6-11], the original principle of the method remains unchanged. The method uses a succession of chemical reagents (extractants) chosen to selectively target the various geochemical phases (and associated pollutants/toxicants) likely to be affected by changes in environmental conditions within the soil/sediment. The increasing strength of successive extractants can be used to assess the potential for remobilization and bioavailability of these pollutants. Sequential extraction schemes have often been criticized because the analytical results are affected by a number of pitfalls, the most serious of which relate to the poor selectivity of the reagents used and the post-extraction resorption of released radionuclides.

It is now widely accepted that no extractant is perfectly selective, and that a reagent (extractant) used to selectively dissolve one particular geochemical phase may not succeed in completely doing so, or may also attack other phases. In addition to the choice of reagent itself, a number of other factors can influence the selectivity of the extraction, including reagent concentration, reagent to sample ratio, temperature, duration of extraction, *etc*. The results obtained must, therefore, be considered to be operationally defined, and care should be taken in interpreting or labelling the different fractions with a particular geochemical phase.

Readsorption and redistribution during extraction can be even more problematic and can lead to a serious underestimation of the fraction "removed" in a particular extraction step. Readsorption is caused by many factors including the affinity of the extracted radionuclide for the remaining undissolved phases and the ability of the extractant to inhibit resorption by these phases [12]. Despite these limitations, satisfactory results can be obtained, provided the correct extractants are chosen and the protocol employed is stringently validated.

In this study, sequential extraction was carried out using two different, though compatable, sequential extraction protocols currently employed by our respective laboratories. Both protocols consider six separate fractions, namely exchangeable (R0), acido-soluble (R1), reducible (R2), oxidizable (R3), strongly bound (R4) and residual (R5), but differ slightly in the nature and/or concentrations of the reagents used (Tables 1 and 2). For the sequential extraction of ^{90}Sr, ^{226}Ra and ^{238}U, the protocol employed was that of Tessier *et al.* (1979), developed for the analysis of the bioavailability of trace metals in soils and suspended sediments. For ^{137}Cs and ^{239,240}Pu, a modified version of the Tessier protocol, developed at one of our laboratories was deployed. This was produced to address the known problems of readsorption and redistribution of plutonium isotopes during extraction [13,14]. Summaries of the two protocols employed, including the extractants, targeted geochemical phases, and details on the duration and temperature of the extractions, are given in Tables 1 and 2.

Materials and Methods

Sampling and Sample Preparation

For the purpose of sequential chemical extraction analysis, samples of soil were collected on the STS from the surrounding ring of spoil at Balapan Lake, Tel'kem 1 and Tel'kem 2, and from the centre of the lake (under water) at Tel'kem 1, in the course of a

Table 1
Sequential extraction protocol used to examine the solid partitioning of ^{90}Sr, ^{226}Ra and ^{238}U in the present study (reagent-to sample ratio 20:1)

Fraction	Targeted Phases	Extractant	Temperature, °C	Time, h
R0 Exchangeable	Weakly bound/ exchangeable	0.4 M $MgCl_2$, pH 6.5	Room	1
R1 Acido-soluble	Carbonates	1 M NH_4Ac in 25% HAc, pH 4	Room	2
R2 Reducible	Oxyhydroxides	0.1 M $NH_2OH{\cdot}HCl$ in 25% HAc	70	6
R3 Oxidizable	Organics/sulphides	30% H_2O_2 in 0.008 M HNO_3, pH 2	50	4
R4 Strongly bound	Remaining extractable	4 M HNO_3	90	4
R5 Residual	Mineral matrix	HF + $HClO_4$ + HCl or NaOH fusion		

Table 2.
Modified sequential extraction protocol used to examine the solid partitioning of ^{137}Cs and ^{239,240}Pu in the present study (reagent-to sample ratio 50:1).

Fraction	Targeted Phases	Extractant	Temperature, °C	Time, h
R0 Exchangeable	Weakly bound/ exchangeable	MilliQ water, 0.175 M Na-citrate, pH 8 with NH_4OH	Room	1
R1 Acido-soluble	Carbonates	1 M NH_4Ac, 0.175 M Na-citrate, pH 5 with HAc	Room	2
R2 Reducible	Oxyhydroxides	1 M $NH_2OH{\cdot}HCl$, 0.175 M Na-citrate, 25% HAc, pH 5 with NH_4OH	Room	2
R3 Oxidizable	Organics/sulphides	30% H_2O_2, 0.175 M Na-citrate, pH 2 with HNO_3	Room	2
R4 Strongly bound	Remaining extractable	4 M HNO_3, 0.175 M Na-citrate	90	4
R5 Residual	Mineral matrix	HF + HNO_3		

field campaign undertaken in July 2000 [15], and from Area 4A in the northern zone of the STS in July 2005. Soil sampling at Ground Zero is currently not permitted and the soil sample analysed was collected at an earlier date by an IAEA team. Soil samples were also collected from the Kurdai uranium ore mining site at two locations, the first close to the northern shore of the artificial lake referred to above and the second some 60 m to the north-east, in the course of a field mission carried out in May 2006. All samples were air-dried, homogenised and sieved through a 1-mm wire mesh prior to sequential extraction analysis.

Sequential Chemical Extraction

Wetted soil samples of 1-2 g were weighed into centrifuge tubes. A reagent-to-sample ratio of 20:1 (or 50:1) was used and each extraction was performed without heating and with continuous stirring to ensure maximum contact between soil and extractant. Following extraction, separation of the aqueous from the soil fraction was carried out by centrifugation at 5000 rpm, followed by filtration through a 0.45 µm membrane filter. Further, the soil fraction was rinsed with de-ionized water between extractions to ensure complete removal of the extractant. The extractant and rinse solution were subsequently combined and analysed for their radionuclide (*i.e.*, ^{90}Sr, ^{137}Cs, ^{226}Ra, ^{238}U and ^{239,240}Pu) content. In addition, X-Ray diffraction analysis was performed to assess the selectivity of extraction for different geochemical phases and to determine mineralogical composition.

Radiochemical and Radiometric Analysis

Strontium-90

Following ashing, the separation of strontium from each fraction was accomplished by a well-established procedure based on the precipitation of strontium as carbonate, and separation of strontium from inactive and active interferences as hydroxides after the method of Margulis [16]. Typical strontium recoveries were in the range 80-90% and were determined gravimetrically. The ^{90}Sr content of each fraction was then measured by beta counting of ingrown ^{90}Y following a two-week equilibration of separated ^{90}Sr using a low-background, Doza-supplied alpha/beta counter [17].

Caesium-137

The ^{137}Cs content of each fraction was determined by high resolution gamma spectrometry using an n-type high purity germanium detector. The detector was calibrated using a mixed radionuclide standard containing ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{51}Cr, ^{113}Sn, ^{85}Sr, ^{137}Cs and ^{88}Y supplied by Cerca Framatome ANP, France (code: 7081/4).

Uranium-238 and Radium-226

The uranium content of each fraction was determined by high resolution alpha spectrometry following radiochemical separation, which involved ferric hydroxide co-precipitation, dissolution by concentrated nitric acid, separation of uranium by solvent extraction using tri-*n*-butylphosphate, and electrodeposition from an ammonium chloride/ ammonium oxalate solution onto polished stainless-steel discs. Chemical recoveries of uranium, determined using ^{232}U as yield monitor, were typically 70-90%.

The ^{226}Ra content of each fraction was also measured by alpha-spectrometry following micro-precipitation with lead sulphate. In this case ^{133}Ba was employed as the yield monitor. Radium recoveries were similar to those of uranium.

Plutonium-239,240

The plutonium content of each fraction was similarly determined by alpha spectrometry following separation and purification by ion-exchange [18], and electrodeposition, as described above. Chemical recoveries were determined using a NIST-certified ^{242}Pu standard reference solution as yield monitor and were typically in the range 60-95%.

Results and Discussion

Strontium-90

Sr-90 bound to soil can be desorbed to the liquid phase by changes in soil pH, temperature or oxidation-reduction potential, as well as by the decomposition of soil organic matter, leaching processes, etc. The same is true for other trace metals. Moreover, the biological availability of ^{90}Sr and other metals also depends on the genesis (*i.e.*, source specific characteristics) of the fallout or contamination involved. As the data for the various radioelements examined below show, different source terms lead to different levels of availability/non-availability. The data for ^{90}Sr partitioning, as determined by sequential extraction analysis, are summarized in Fig. 2.

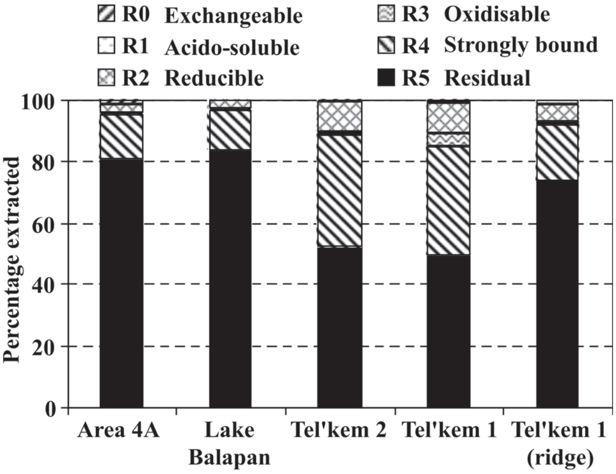

Fig. 2. The partitioning of ^{90}Sr in selected soils following sequential extraction.

Little if any of the ^{90}Sr in soil at the four distinct STS sites examined proved to be in a weakly bound, easily extractable form**.** The percentages associated with the acido-soluble and oxidisable fractions are also insignificant, though approximately 10% of the ^{90}Sr at both Tel'kem sites appears to be linked to the reducible fraction. At all four sites, at least 85% of the ^{90}Sr is found in either the strongly bound or the residual fraction, with Balapan, as expected, being the most extreme example.

However, at first sight, perhaps the most surprising finding is that at Area 4A, onto which ^{90}Sr was released following tests of radiation dispersal devices (containing liquid radioactive waste from radiochemical factories). That so much of the strontium found in soil from Area 4A at the present time should be associated with the residual and strongly bound fractions is understandable once it is appreciated that the waste released over this area contained nitric acid which destroys silicate minerals within soil, forming orthosiliconic acid. Following the polycondensation of orthosiliconic acid, polymeric silicate network structures are formed. These latter, in turn, are immobilized and strontium (and other cations) become strongly fixed within their lattice structure.

Plutonium-239,240

The partitioning of plutonium in a small number of soil samples from the STS was examined using the sequential extraction scheme proposed by Lucey (2003) and to which we have already made reference. The soils examined were collected close to the epicentre at Ground Zero, from the surrounding ring of spoil at the Balapan Atomic Lake and Tel'kem 2, and from the centre of the lake at Tel'kem 1. The results, summarized in Figure 3, clearly show that, with the exception of Tel'kem 1, most of the plutonium in the soils analysed appears to be in a highly refractory, non-labile form. In fact the proportion of plutonium in the residual phase was found to be about 90% at Ground Zero, 85% at Lake Balapan, 60% at Tel'kem 2 and 20% at Tel'kem 1. There is little doubt but that this pattern reflects differences in the temperature of the fireball(s) which engulfed the soil upon detonation at each of these sites, as well as differences in soil type/composition. In the case of Tel'kem 1 and Tel'kem 2, the latter is almost certainly the most relevant, as the explosive yields were both very low and quite similar (0.25 and 0.75 kilotons

TNT equivalent, respectively). Perhaps more remarkable is the observation that little plutonium is present in an exchangeable form at either Ground Zero or Lake Balapan, though almost 35% is at Tel'kem 1. What all of this implies, of course, is that at the STS little plutonium is available for uptake by plants and shrubs, thereby constraining the doses that potentially could be received by populations consuming locally grown produce or livestock.

Caesium-137

In the case of ^{137}Cs the analysis showed a consistent pattern (Fig. 4), with at least 95% of the activity at each site associated with the residual and strongly bound fractions, and little if any associated with the more available phases.

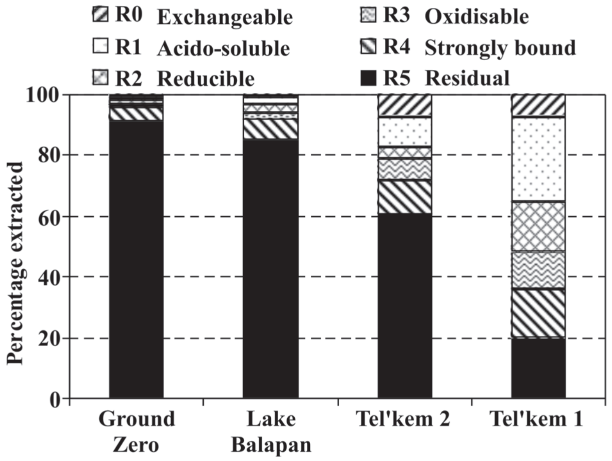

Fig. 3. The partitioning of plutonium in soils from Ground Zero, Lake Balapan, Tel'kem 1 and Tel'kem 2 upon sequential extraction analysis

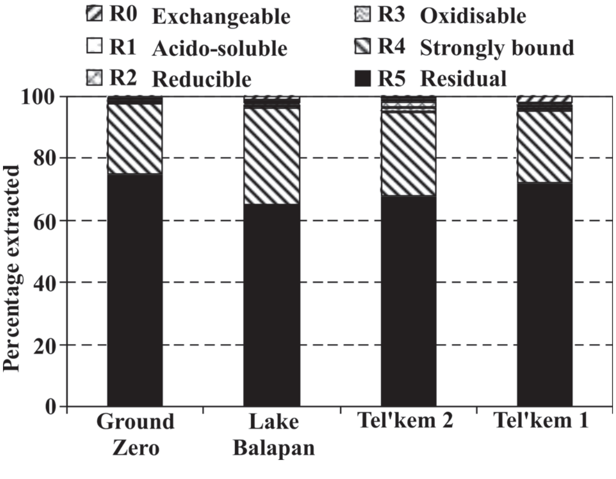

Fig. 4. The partitioning of ^{137}Cs in soils from Ground Zero, Lake Balapan, Tel'kem 1 and Tel'kem 2 upon sequential extraction analysis

Radium-226 and Uranium-238

To characterize the levels and extent of contamination at the Kurdai site, a large number of soil samples were examined. X-ray diffraction analysis indicated that the soil was mainly composed of quartz and feldspar, with sand and clay as minor constituents. Radiometric analysis showed that total ^{226}Ra and ^{238}U concentrations in these samples were in the range 410-2913 Bq·kg^{-1} and 300-654 Bq·kg^{-1}, respectively. In the majority of samples the daughter members of the uranium series were in near secular equilibrium, indicating that the soil had not been processed (*i.e.*, uranium had not been chemically leached from the soil).

Data on the solid partitioning of uranium and radium in two distinct soil samples, one taken at lakeside and the second ~60 m inland, are presented in Fig. 5. The data reveal that uranium is distributed fairly evenly over all fractions with the exception of the exchangeable, which proved to be very small. In contrast, the bulk of the ^{226}Ra appears to be associated with the strongly bound and residual fractions, with relatively little in the exchangeable fraction. Also, almost twice as much uranium and radium is associated with the strongly bound and residual fractions of soil 2 compared to soil 1.

Conclusions

The data show that at the STS, in general, little (if any) of the radiostrontium, radiocaesium and plutonium is in an exchangeable form, with the great bulk of the radiostrontium and radiocaesium in a strongly

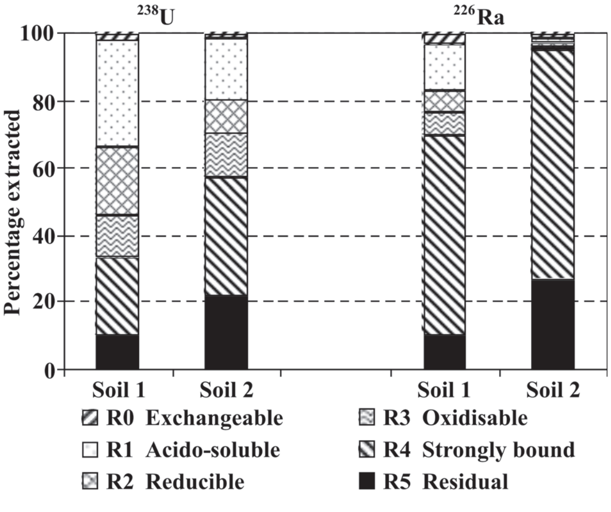

Fig. 5. The partitioning of uranium and radium in distinct soils from the Kurdai mining site, following sequential extraction.

bound or refractory form. In the case of plutonium, the proportion in a strongly bound or refractory form varies from 40–95% depending on site, and appears to be a function of the explosive yield involved and, by extension, the temperature of the resulting fireball. Izrael [19] has noted that besides the temperature of the fireball, there are other factors to be considered when assessing the physical and chemical fractionation of different radionuclides (and activation products) including the type of explosion products and inert material used, the thermodynamic conditions in the explosion zone, and the thermal and physical properties of the environmental material in this zone. Fractionation commences when evaporated matter condenses, resulting in selective capture of isotopes of certain elements by the liquid phase at the time of radioactive particle generation. The most refractory elements are condensed first within liquid particles of the soil (or any other local material) and are distributed within these particles, while the more volatile elements condense later, often after solidification of these particles. This will be particularly important for ^{137}Cs since most production ocurrs in the rapidly cooling nuclear cloud as a consequence of the decay of the primary fission product gas – ^{137}Xe. This may provide an explanation for the similar speciation of this radionuclide at all explosion sites.

At the Kurdai ore deposit, two distinct, well-characterised soils were examined which exhibited significant differences in the partitioning of uranium and radium, with almost twice as much uranium and radium associated with the strongly bound and residual fractions of soil 2 compared to soil 1. This is consistent with the results of mineralogical analysis, which showed that the mineral content (*i.e.*, chlorite – a group of minerals consisting of the hydrated silicates of aluminium, iron and magnesium; and amphibole - a group of volcagenic minerals consisting of silicates of calcium, iron, magnesium, sodium and aluminium) of soil 2 is twice as high as that of soil 1, confirming that these minerals retain and immobilize a very significant fraction of both elements. Overall, some 35-60% of the uranium and the bulk of the radium are in a strongly bound or residual form, whereas less than 5% of the uranium and radium are in an exchangeable form.

Acknowledgements

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