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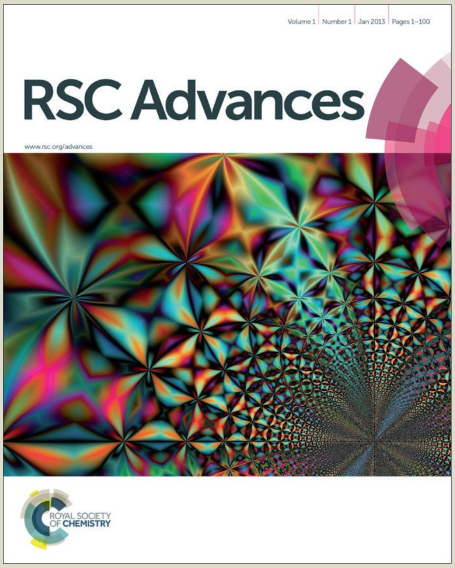

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A Trialkyl Phosphine Oxide Functionalized Task Specific Ionic Liquid for Actinide Ion Complexation: Extraction and Spectroscopic studies†

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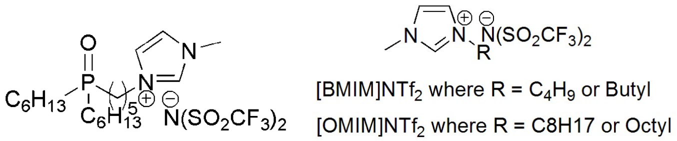
A trialkylphosphine oxide functionalized task specific ionic liquid (PO-TSIL) with NTf_2^- counter anion was synthesized and evaluated for the extraction of actinide ions such as UO_2^{2+} and Pu^{4+} from acidic feed solutions using room temperature ionic liquids (RTIL) such as [BMIM][NTf_2] and [OMIM][NTf_2] as the diluents. The extraction data were compared with those obtained with tri-*n*-octylphosphine oxide (TOPO) in the same set of RTILs. The extracted species following a cation-exchange mechanism were analyzed by the conventional slope analysis method. The nature of bonding in the extracted complexes was investigated by various spectroscopic techniques such as luminescence and UV-visible spectroscopy. Studies on the stripping and radiation stability were also carried out for possible nuclear fuel cycle applications.

Spent fuel reprocessing by the PUREX process uses TBP (tri-*n*-butyl phosphate) in *n*-dodecane as the solvent for the recovery of U and Pu for a sustainable closed nuclear fuel cycle.[1] Recent studies using room temperature ionic liquids (RTILs) are getting increasing attention for possible fuel cycle applications not only due to their low volatility and non-flammability[2] but also due to their higher metal ion extraction capability[3] and higher radiation stability.[4] Though the results are fairly encouraging in RTILs, significant aqueous solubility of the ionic liquid is reported in view of the ion-exchange mechanism involved in the metal ion extraction,[5] making their process scale application unattractive. Task specific ionic liquids (TSIL) were reported to solve this problem as the ligating group is grafted to the anionic or cationic component of the ionic liquid.[6] There are numerous reports on actinide ion extraction using TSILs with functional groups such as phosphate,[7] hydroxyamino,[8] CMPO (carbamoylmethyl phosphine oxide),[9] malonamide[10] and DGA (diglycolamide).[11]

TOPO (tri-*n*-octylphosphine oxide) is known to be a more basic extractant as compared to TBP leading to higher extraction of the metal ions at a given feed acid concentration. Solutions of TOPO in RTILs such as [BMIM]NTf_2 (1-butyl-3-methylimidazolium) or [OMIM]NTf_2 (1-octyl-3-methylimidazolium) (Fig. 1a) have been employed for the extraction of UO_2^{2+} and Pu^{4+} from HNO_3 medium.[12] To our knowledge, there is no report on the extraction of actinide ions including UO_2^{2+} and Pu^{4+} from acidic feeds using trialkylphosphine oxide functionalized TSIL (termed hereafter as PO-TSIL, Fig. 1b).



(a) **(b)**

Figure 1 Structural formula of the PO-TSIL and RTILs used in the present study

It was, therefore, of interest to investigate the extraction behaviour of UO_2^{2+} and Pu^{4+} from HNO_3 medium using PO-TSIL and the results are compared with those obtained with solutions of TOPO in the ionic liquids, [BMIM]NTf_2 and [OMIM]NTf_2. The studies were directed at understanding the extraction behaviour of the metal ions and to devise separation methods. Apart from this, it was also aimed at understanding the mechanism of extraction by the conventional 'slope analysis' method along with techniques such as UV-vis spectroscopy and time resolved fluorescence spectroscopy (TRFS). The application of a newly developed solvent system for nuclear fuel cycle applications can get acceptance after the sustainability for long term reusability is established. Keeping this in mind, radiolytic stability and stripping studies were also carried out.

PO-TSIL was synthesized in two steps,[13] the first step involving preparation of the TSIL with methanesulfonate as a counter anion (Scheme S1 in Supporting Information).† In a subsequent reaction, the product PO-TSIL with bis(trifluoromethane)sulfonamide (NTf_2^-) counter anion was prepared (Scheme S2 in Supporting Information). The products were characterized by NMR and HR-MS (Supporting Information).

The extraction of actinide ions by RTILs takes longer time to attain equilibrium as compared to molecular diluents in view of factors such as higher viscosity[5b] and complex extraction mechanism.[5a] The time required to attain equilibrium distribution coefficient values for UO_2^{2+} extraction was found to be dependent on the feed HNO_3 acid concentration rather than the nature of the RTIL diluent (Supporting Information).† For the same acid concentration, Pu^{4+} extraction kinetics was faster as compared to that of UO_2^{2+} (Supporting Information).†

PO-TSIL was evaluated for the extraction of UO_2^{2+} and Pu^{4+} ions as a function of feed nitric acid concentration and the results are presented in Fig. 2. For comparison purpose, UO_2^{2+} extraction data are also presented using 0.05 M TOPO in [OMIM]NTf_2 and *n*-dodecane making the extraction systems nearly identical. As seen from the figure, the extraction mechanism of TOPO in [OMIM]NTf_2 and *n*-dodecane resemble each other and appear to involve neutral species as reported previously.[12] On the other hand, a cation-exchange mechanism appears to be valid for the extraction of the metal ion when solutions of PO-TSIL in [OMIM]NTf_2 of [BMIM]NTf_2 were used.

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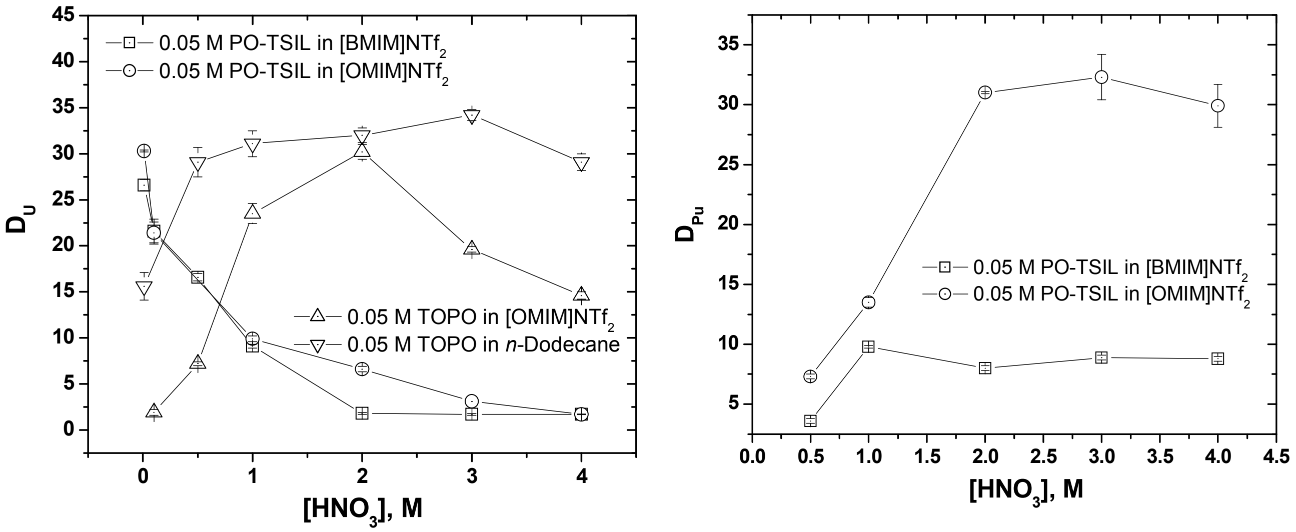

(a) **(b)**

Figure 2 Dependence of metal ion extraction on acid concentration. (a) UO_2^{2+} – TSIL system; (b) Pu^{4+} – TSIL system

Careful observation of the extraction profiles of UO_2^{2+} and Pu^{4+} (Fig. 2) in PO-TSIL suggests that while the extraction of UO_2^{2+} decreased with HNO_3 concentration, an entirely opposite trend was seen for Pu^{4+}. Contrary to the report of ion-pair species extraction for UO_2^{2+} extraction by TOPO-RTIL solvent system, the present results indicate a cation extraction mechanism with possible extracted species as $UO_2(TSIL)_x(NTf_2)_y{}^{(2+x-y)+}{}_{IL}$. On the other hand, Pu bearing species were neutral ion-pairs of the type, $Pu(TSIL)_n(X)_{4+n}$, where $X^- = NTf_2^-$ or NO_3^- (vide infra).

In order to further ascertain the nature of the extracted species, it was required to determine the number of PO-TSIL units present in the extracted species. Conventional 'slope analysis' (Supporting Information) suggested the presence of two units of PO-TSIL in the Pu^{4+} bearing extracted species in both $[OMIM]NTf_2$ and $[BMIM]NTf_2$, thereby prompting us to suggest us to propose extraction of species of the type, $Pu(TSIL)_2(X)_6$ and the extraction equilibrium can be given as:

$$Pu^{4+} + 2\ TSIL^+_{IL} + 6\ X^- = Pu(TSIL)_2(X)_{6\ IL} \qquad (1)$$

where the species with the subscript 'IL' represents those present in the RTIL phase and those without any subscript refer to those in the aqueous phase. On the other hand, 'slope analysis' suggested that while two units of PO-TSIL were present in the UO_2^{2+} bearing species in $[OMIM]NTf_2$, a mixture of 1:2 and 1:3 (M:L) species were extracted for $[BMIM]NTf_2$ (Supporting Information)[†]. These results suggested that while $UO_2(TSIL)_2(X)_y{}^{(4-y)+}$ (where y < 4) may be extracted in $[OMIM]NTf_2$, a mixture of $UO_2(TSIL)_2(X)_y{}^{(4-y)+}$ and $UO_2(TSIL)_3(X)_y{}^{(5-y)+}$ (where y < 5) may be extracted in $[BMIM]NTf_2$. Form these, UO_2^{2+} extraction may involve the following eqn.

$$UO_2^{2+} + y\ X^- + x\ TSIL^+_{IL} + (2+x-y)\ NMIM^+_{IL} = UO_2(TSIL)_x(X)_y{}^{(2+x-y)+}{}_{IL} + (2+x-y)\ NMIM^+ \qquad (2)$$

where, x may be 2 or 3 and NMIM is BMIM or OMIM. As shown below, the evidence for two species in case of UO_2^{2+} ion extraction is also given from luminescence spectroscopy.

The extraction of other important actinide and fission product radionuclides present in radioactive wastes such as Am^{3+} (representative trivalent minor actinide), Eu^{3+} (representative trivalent lanthanides), Cs^+ and Sr^{2+} was also studied. The extraction data (Table 1) suggested extraction of only Pu^{4+} and UO_2^{2+} while the other metal ions were not extracted to any significant extent. Although Sr^{2+} was not extracted at all, Cs^+ was extracted to a very small extent. Careful analysis of the extraction data indicated Pu^{4+} was preferentially extracted as compared to UO_2^{2+} in the PO-TSIL in both the RTIL diluents. Secondly, $[OMIM]NTf_2$ was found to be a better diluent as compared to $[BMIM]NTf_2$. Finally, while TOPO was a better extractant as compared to PO-TSIL in $[OMIM]NTf_2$, an opposite trend was seen in $[BMIM]NTf_2$. Though many TSILs result in enhanced extraction as compared to analogous ionic liquid based solvent systems such as the diglycolamide-functionalized TSIL,[11] results similar to the one observed in the present study with $[BMIM]NTf_2$ as the diluent, where the TSIL led to lower extraction as compared to analogous RTIL based solvent, was also reported by us using a CMPO-functionalized TSIL.[9b] Furthermore, the selectivity changes with the aqueous phase

Table 1 Distribution data of some important actinide and fission product element ions, present in the HLW solution, using PO-TSIL and TOPO based solvent systems from 1 M HNO_3.

Solvent system[a]	Distribution coefficient					
	Am^{3+}	Pu^{4+b}	UO_2^{2+}	Eu^{3+}	Cs^+	Sr^{2+}
PO-TSIL in $[BMIM]NTf_2$	$<1\ x10^{-3}$	9.8 ± 0.1	9.12 ± 0.10	$<1\ x10^{-3}$	0.067	$<1\ x10^{-3}$
PO-TSIL in $[OMIM]NTf_2$	$<1\ x10^{-3}$	13.5 ± 0.4	9.90 ± 0.30	$<1\ x10^{-3}$	$<1\ x10^{-3}$	$<1\ x10^{-3}$
TOPO in $[BMIM]NTf_2$	$<1\ x10^{-3}$	3.05 ± 0.05	3.63 ± 0.11	$< 1\ x10^{-3}$	0.005	$<1\ x10^{-3}$
TOPO in $[OMIM]NTf_2$	$<1\ x10^{-3}$	135.5 ± 16.1	22.9 ± 1.1	$< 1\ x10^{-3}$	$<1\ x10^{-3}$	$<1\ x10^{-3}$

[a] Concentration of the extractant: 0.05 M PO-TSIL in the respective ionic liquid. [b] $NaNO_2$ was used as the holding oxidant.

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nitric acid concentration. As seen from Fig. 2, the extraction of Pu^{4+} is significantly higher as compared to that of UO_2^{2+} at 2 – 3 M HNO_3 the selectivity becoming about 10 times (D_{Pu}/D_U) with [OMIM]NTf_2 at 3 M HNO_3. The higher extraction of Pu^{4+} vis-à-vis UO_2^{2+} is in sharp contrast to the extraction trend observed with TBP in the PUREX process. The viscosity of the TSIL phase was found to decrease which is attributed to water extraction (Supporting Information). † As the solvent extraction studies are carried out from aqueous feeds, the RTIL phase is always containing the co-extracted water. Therefore, the distribution and the selectivity data always refer to the water saturated ionic liquid phase.

Table 2 Stripping data (obtained after 1 h equilibration time) from PO-TSIL extracts obtained from 1 M HNO_3

Stripping agent	% Stripping			
	[BMIM]NTf_2		[OMIM]NTf_2	
	UO_2^{2+}	Pu^{4+}	UO_2^{2+}	Pu^{4+}
8 M HNO_3	66.3	--	56.8	--
0.05 M EDTA + 1 M Guanidine carbonate	99.9	98.4	99.2	99.3
1 M Na_2CO_3	87.7	--	99.2	--
0.5 M Oxalic acid	--	99.9	--	99.5

Stripping of the metal ions is an integral part of the evaluation any solvent systems being developed for actinide extraction not only for the recovery of the metal ions but also from recycling of the solvent point of view. Several complexing agents were tested for the stripping of both UO_2^{2+} as well as Pu^{4+} and the results are presented in Table 2. Apart from these, stripping of UO_2^{2+} was also attempted using 8 M HNO_3 which was found not to be very effective. Effective stripping of UO_2^{2+} (>99%) was seen with 0.05 M EDTA in 1 M guanidine carbonate which was reported previously not to be very effective for the back extraction of rare earth metal ions from CMPO ionic liquid extracts.[14] While Pu^{4+} could be effectively stripped using 0.5 M oxalic acid, Na_2CO_3 solutions was effective for U stripping only from [OMIM]NTf_2. The lower stripping efficiency from [BMIM]NTf_2 was attributed to higher acid uptake in that medium which destroyed part of the carbonate decreasing its effectiveness (Supporting Information). The stripping kinetics was slow similar to the extraction kinetics (Supporting Information).

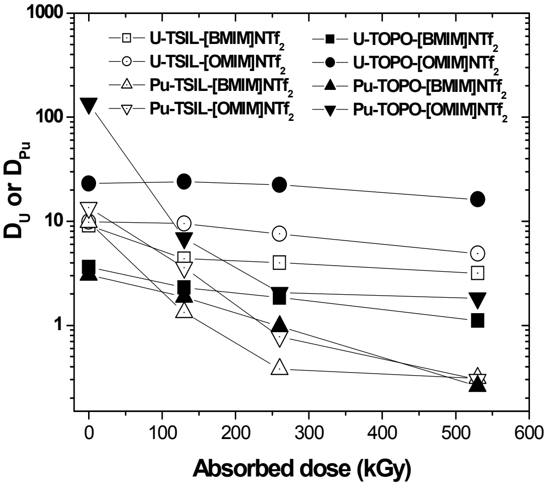

Figure 3 Effect of irradiation on extraction of Pu(IV) and U(VI), aq. phase: 1 M HNO_3,equilibration time: 3 h, [TSIL] or [TOPO]: 0.05 M.

Radiolytic stability of the TSIL was ascertained by carrying out the extraction of UO_2^{2+} ion after exposing the former to an absorbed dose of 530 kGy. The results (Fig. 3) indicated that though the extraction of Pu^{4+} was affected to a very large extent, that of UO_2^{2+} was not affected to such an extent. For U extraction, degradation was lower with [OMIM]NTf_2 than those seen with [BMIM]NTf_2. For comparison purpose, solutions of TOPO in ionic liquids were subjected to similar treatment and the results showed their relatively higher radiolytic instability as compared to the TSIL (Fig. 3).

UV-visible spectra of UO_2^{2+} extracts in TOPO and PO-TSIL in both [BMIM]NTf_2 and [OMIM]NTf_2 are presented in Fig. 4 along with the spectra of uranyl nitrate solution which shows that the spectra of the extracts are nearly identical suggesting equivalent complexation by both the ligands, although a red shift was observed compared to the hydrated uranyl ion indicating strong complexation by the phosphine oxide group in TOPO or PO-TSIL and one or more nitrate anions. Our previous work on UO_2^{2+} extraction using TOPO in ionic liquids has suggested extraction of species of the type, $(UO_2.2TOPO)^{2+}(NTf_2^-)_2$. Considering the similarities between the spectra of the TOPO and PO-TSIL extracts of U, and also considering cation exchange mechanism, species of the type $(UO_2.2TSIL)^{4+}$ may be proposed. Additional evidence for the nature of the extracted species comes from ESI-MS spectra (Supporting Information).†

A look at the photoluminescence spectra of uranyl ion in aqueous HNO_3 medium and the extracts generated after complexation of UO_2^{2+} ion with PO-TSIL in [BMIM]NTf_2 and [OMIM]NTf_2; and TOPO in [BMIM]NTf_2 (Fig. 5) revealed that the nature of the extracted species were entirely different in all the cases not only due to the shifts of the emission maxima but also the differences in the relative intensities of the vibronic structures in the emission profiles (Supporting Information). The symmetric stretching frequency of U-O bonds of uranyl moieties were found to be 793 cm^{-1} and 409 cm^{-1} , respectively for

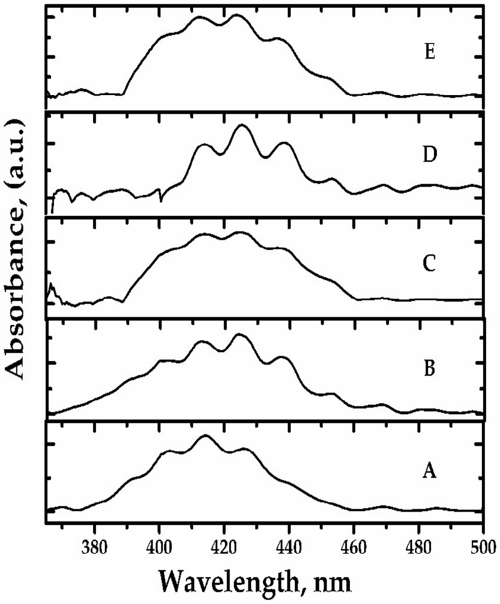

Figure 4 UV-visible spectra of (A) uranyl nitrate solution, and U extracts in (B) PO-TSIL-[OMIM]NTf_2, (C) TOPO-[BMIM]NTf_2, (D) TOPO-[OMIM]NTf_2, (E) PO-TSIL-[BMIM]NTf_2. U concentration: 5.0×10^{-3} M; Extractant concentration: 0.05 M

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aqueous uranyl ion and U-TOPO extract in [BMIM]NTf_2 which were in good agreement with that for UO_3, xH_2O[15] and UO_2^{2+}-TOPO complex in [OMIM]NTf_2 reported in literature.[12] The U-PO-TSIL complex showed the vibronic stretching frequencies at 446 cm^{-1} and 498 cm^{-1} in [OMIM]NTf_2 and [BMIM]NTf_2, respectively. The decrease in the stretching frequency in complexed forms compared to the aqueous uranyl ion can be attributed to the decrease in bond strength of U-O bonds in O=U=O moieties on complexation. The oxygen to uranyl charge transfer was found to be unfavoured by the approach of 2 ligands molecules along the equatorial plane of the uranyl ion. The symmetric stretching frequency of uranyl oxygen obtained from the emission spectra revealed that in the UO_2^{2+} TSIL complex, the U-O bonds were shorter for the PO-TSIL complex compared to that of analogous complex with TOPO. Due to the larger size of the TSIL, their approach towards metal ion is sterically hindered which favoured the oxygen to uranium charge transfer and strengthening the U-O bonds of uranyl moieties. Similarly, the ground state frequency of symmetric stretching vibration of $UO_2Cl_4^{2-}$ in [DMIM]Br[16] and [BMIM]NTf_2[17] were reported as approximately 750 cm^{-1} and 825 cm^{-1}, respectively. The luminescence decay profiles (Supporting Information, Fig. S9) revealed the presence of single species in case of aqueous uranyl ion, U-TOPO extract in [BMIM]NTf_2 and U-PO-TSIL extract in [OMIM]NTf_2 whereas for U-TSIL extract in [BMIM]NTf_2 showed the bi-exponential nature of the decay profile revealing the presence of two species.

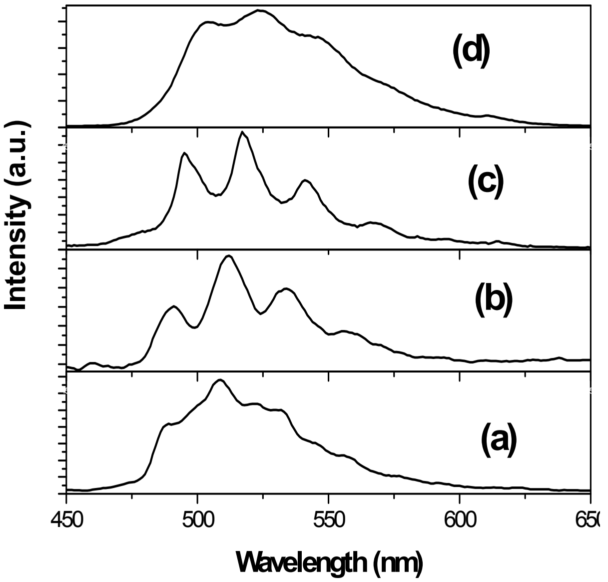

Figure 5 Luminescence spectra of (a) aq. uranyl, (b) U(VI)-TOPO extract in in [BMIM]NTf_2 (c) U(VI)-PO-TSIL extract in [OMIM]NTf_2 and (d) U(VI)-PO-TSIL extract in [BMIM]NTf_2

In conclusion, the PO-functionalized TSIL has been used for the extraction of actinides for the first time. The solvent extraction data, with the PO-TSIL suggested significant extraction of both UO_2^{2+} and Pu^{4+} and almost no extraction of the trivalent (lanthanide or actinide), bivalent or monovalent ions. The stripping of the extracted actinide ions was effective with suitable stripping agents such as EDTA in guanidine carbonate for both UO_2^{2+} and Pu^{4+} and oxalic acid for Pu^{4+}. Though these results appear promising, the insufficient radiation stability, particularly for Pu^{4+} extraction suggest that the PO-TSIL may not be considered as a suitable alternative for TBP in the PUREX process.

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† Electronic Supplementary Information (ESI) available: Synthesis of TSIL, Solvent extraction data, etc. See DOI: 10.1039/b000000x/

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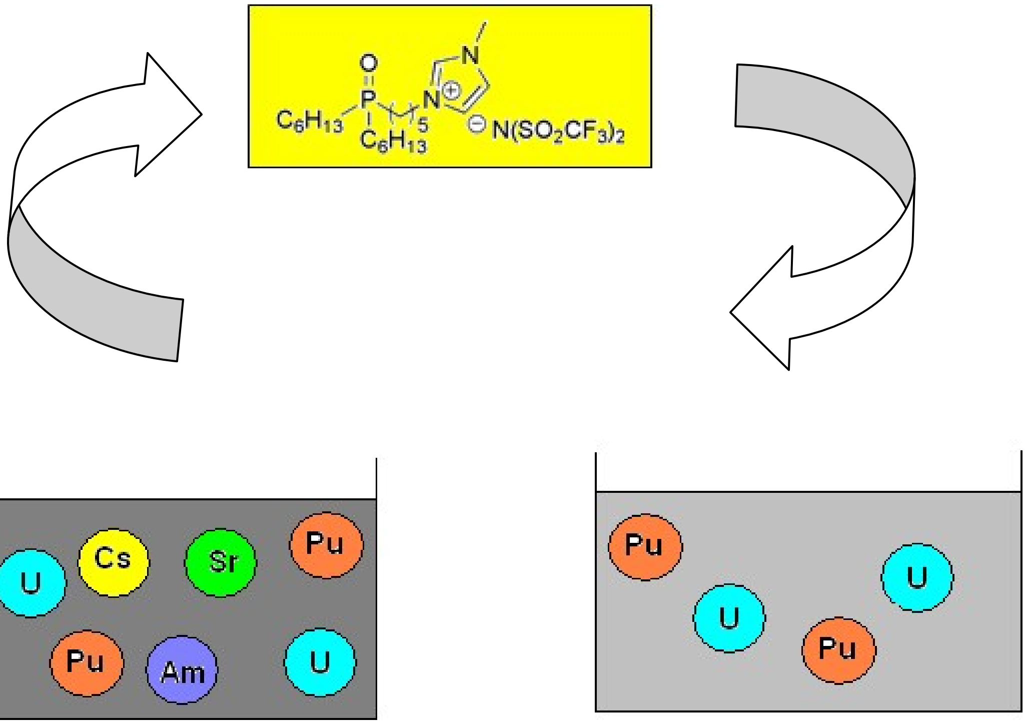
GRAPHICAL ABSTRACT



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