SELECTIVE AMERICIUM SEPARATION: NEW INSIGHTS INTO THE COMPLEXATION OF TRIVALENT *f*-ELEMENTS WITH SO_3-PH-BTBP

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Abstract. In the Americium selective (AmSEL) process, the N-donor ligand 3,3′,3″,3‴-([2,2′-bipyridine]-6,6′-diylbis(1,2,4-triazine-3,5,6-triyl))tetrabenzenesulfonate (SO_3-Ph-BTBP) is used to selectively strip Am(III) from a *N*,*N*,*N*′,*N*′-tetraoctyl diglycolamide (TODGA) containing organic phase loaded with Am(III), Cm(III) and lanthanides (Ln(III)). Fundamental extraction mechanism studies revealed an unusual extraction behavior of heavy Ln(III) and Y(III), which provided the motivation to investigate their complexation with SO_3-Ph-BTBP using nuclear magnetic resonance (NMR) spectroscopy and solvent extraction. NMR spectroscopy indicated the formation of the same SO_3-Ph-BTBP complex with Lu(III) in 10^{-3} mol L^{-1}, 1 mol L^{-1} and 3 mol L^{-1} DNO_3 solution. However, the complexation at high DNO_3 concentration is subject to a slower than expected complexation kinetics leading to the conclusion that the unusual extraction behavior is probably related to a kinetics effect rather than an unknown complex species. Kinetics studies using solvent extraction show a slowly increasing extraction of heavy Ln(III) and Y(III) into the organic phase which is attributed to a kinetically inhibited decomplexation of the SO_3-Ph-BTBP complexes. This effect is even more pronounced at higher HNO_3 concentration. Additionally, combinations of mono- and di-methylated TODGA derivatives with SO_3-Ph-BTBP were tested, showing a decreasing performance regarding the actinide(III)/lanthanide(III) and Am(III)/Cm(III) separation with increasing degree of methylation.

1. Introduction

Advanced reprocessing of used nuclear fuel with Am(III) separation is a challenging task. On an industrial scale, uranium and plutonium are separated *via* the Plutonium Uranium Reduction Extraction (PUREX) process for partial reuse as mixed oxide (MOX) fuel in common light water reactors [1]. Since future reactor developments aim to use a fast neutron spectrum, americium separation and reuse might be considered advantageous regarding final disposal [2]. Especially the separation of Am(III) from Cm(III) and the fission lanthanides is a challenge due to their similar chemical behavior. Therefore, solvent extraction processes have been developed using highly selective ligands [3]. As O-donor ligands such as *N*,*N*,*N*′,*N*′-tetraoctyl diglycolamide (TODGA, Fig. 1) show a high affinity towards trivalent metal ions, they are used for effective co-extraction of lanthanides (Ln(III)) and trivalent actinides (An(III)) [4]. In contrast, N-donor ligands reveal a higher selectivity of An(III) over Ln(III) [5]. Therefore, processes have been developed combining both kinds of ligands to achieve a sufficient selectivity for the separation of An(III) from fission Ln(III) as well as Am(III) from Cm(III) [6-8]. The Americium selective (AmSEL) process was developed to selectively strip Am(III) from a loaded organic TODGA phase containing An(III) and Ln(III) by using the hydrophilic N-donor ligand tetra-sodium-3,3′,3″,3‴-([2,2′-bipyridine]-6,6′-diylbis(1,2,4-triazine-3,5,6-triyl))tetrabenzenesulfonate (SO_3-Ph-BTBP, Fig. 1) [8]. While Am(III) separation from Cm(III) and the light Ln(III) (La – Gd) is possible, Wagner *et al.* recognized an unusual extraction behavior for the heavier Ln(III) (Tb – Lu) and Y(III) [8]. Metal ion extraction with TODGA usually increases with increasing nitric acid concentration [9]. However, Wagner *et al.* observed decreasing extraction of the heavy Ln(III) in the presence of SO_3-Ph-BTBP. They speculated that this remarkable behavior might originate

from the formation of SO_3-Ph-BTBP complexes in the aqueous phase with a composition differing from the proven 1:2 metal-to-ligand complexes [8,10]. To elucidate this issue, nuclear magnetic resonance (NMR) spectroscopic investigations of the complexation of Lu(III) with SO_3-Ph-BTBP were conducted. Additional solvent extraction studies were carried out to provide further insights into the heavy Ln behavior. Additionally, methylated TODGA derivatives (Me-TODGA, Me_2-TODGA, Fig. 1) were tested for their ability to improve the current AmSEL process.

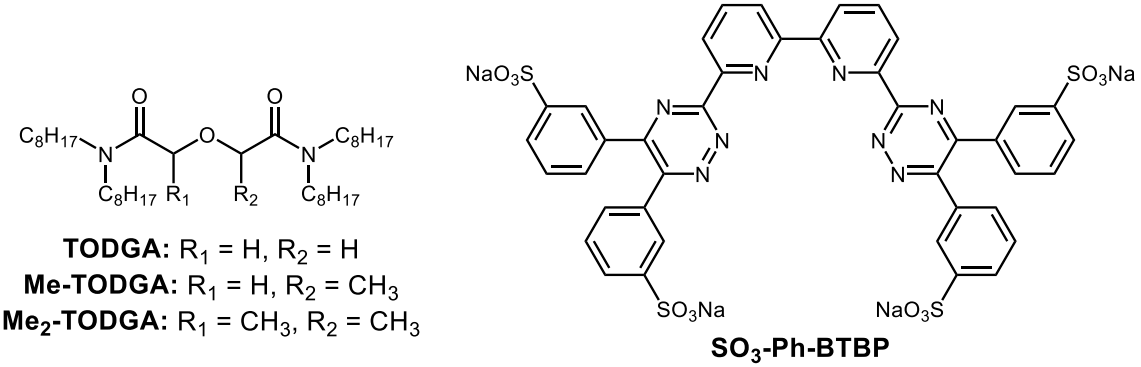

Fig. 1. Chemical structures of TODGA, Me-TODGA, Me_2-TODGA, and SO_3-Ph-BTBP.

2. Experimental

2.1 Chemicals

TODGA as well as its mono- and di-methylated derivatives Me-TODGA and Me_2-TODGA were purchased from Technocomm Ltd. (Edinburgh, UK). SO_3-Ph-BTBP was synthesized according to Müllich *et al.* [11]. Isane® IP175, an isoparaffinic diluent, was provided by the CEA (Marcoule, France). Deuterated solvents were purchased from Euriso-Top GmbH (Saarbrücken, Germany). Other chemicals were purchased from different suppliers and used as received. All chemicals were purchased in the highest available quality and had a purity of ≥ 98%.

2.2 NMR spectroscopic studies

NMR titration experiments were conducted in 10^{-3} mol L^{-1}, 1 mol L^{-1} and 3 mol L^{-1} DNO_3 solutions each including 10 vol% MeOD. The addition of 10 vol% MeOD was found to improve the resolution, as the ^1H NMR signals are exceptionally broad using DNO_3 solution. NMR spectra were recorded at 300 K using a Bruker Avance III 400 spectrometer operating at 400.13 MHz for ^1H and 100.63 MHz for ^{13}C. Further details on the NMR studies can be found elsewhere [12,13].

2.3 Solvent extraction studies

General details on the batch extraction experiments and the measuring techniques are described elsewhere [14,15]. Organic phases consisted of 0.2 mol L^{-1} (methylated) TODGA in *n*-dodecane or 5 vol% 1-octanol/Isane® IP175. Aqueous phases contained 10^{-5} mol L^{-1} Ln(III) nitrate (w/o Pm) and Y(III) nitrate as well as tracer concentrations of ^{152}Eu(III), ^{241}Am(III), and ^{244}Cm(III) in 0.7 mol L^{-1} or 3 mol L^{-1} HNO_3. For stripping (back-extraction) kinetics studies, the organic phase was first loaded with trivalent metal ions by contacting with an appropriate aqueous phase and was then shaken against fresh nitric acid solution containing 0.01 mol L^{-1} SO_3-Ph-BTBP. For extraction kinetics studies, 0.01 mol L^{-1} SO_3-Ph-BTBP was dissolved in the aqueous phase as well, and the solutions were left overnight to allow SO_3-Ph-BTBP complexation to reach equilibrium. The organic solution was pre-equilibrated with 0.7 mol L^{-1} or 3 mol L^{-1} HNO_3 solution before extraction. Distribution ratios *D* were calculated as the ratio of metal ion concentration or activity in the organic and aqueous phase with an uncertainty of ± 5% for $0.01 < D < 100$ and an uncertainty of > 5% for lower/higher *D* values.

3. Results and Discussion

3.1 NMR spectroscopic investigations

NMR spectroscopic studies were performed to improve the understanding of the SO_3-Ph-BTBP complexation with heavy Ln. Fig. 2 (left) shows the ^{1}H NMR spectra for the titration of SO_3-Ph-BTBP solution into a Lu(III) nitrate solution. The complexation was studied using a mixture containing 3 mol L^{-1} DNO_3 with 10 vol% MeOD to improve the resolution of the spectra. With increasing ligand-to-metal ratio, a characteristic signal at 6.1 ppm becomes visible, indicating metal ion complexation. The last spectrum of the titration series at a ligand-to-metal ratio of 3:1 was remeasured after 16 h, showing a clear change in the spectrum. This indicates a slow complexation reaction of SO_3-Ph-BTBP with Lu. To find potential differences in the Lu(III) complexation with SO_3-Ph-BTBP depending on the acidity, NMR titrations were carried out at different DNO_3 concentrations. Comparable to the titration experiment at 3 mol L^{-1} DNO_3, Lu(III) complexation with SO_3-Ph-BTBP is also indicated by the change in ^{1}H spectra for titrations in 10^{-3} mol L^{-1} and 1 mol L^{-1} DNO_3 solution. Due to the considerable solvent influence on the ^{1}H spectra, ^{13}C spectra were used for further comparison.

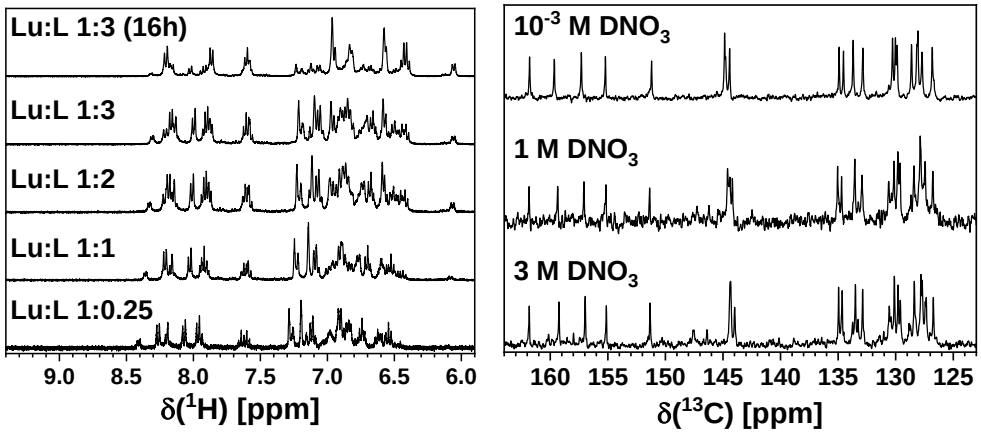

Fig. 2. Left: ^{1}H NMR spectra for titration of SO_3-Ph-BTBP (L) to Lu(III) nitrate solution at 3 mol L^{-1} DNO_3 including 10 vol% MeOD. Right: ^{13}C NMR spectra of Lu(III) complexation with SO_3-Ph-BTBP at 10^{-3} mol L^{-1} DNO_3 with Lu:L = 1:2, at 1 mol L^{-1} DNO_3 with Lu:L = 1:0.67 and at 3 mol L^{-1} DNO_3 with Lu:L = 1:3 (after 16 h), all including 10 vol% MeOD.

Fig. 2 (right) compares the ^{13}C NMR spectra for the SO_3-Ph-BTBP complexation of Lu(III) at 10^{-3} mol L^{-1}, 1 mol L^{-1} and 3 mol L^{-1} DNO_3 concentration. Neglecting minor shifts due to the different solvent, the spectra are identical, indicating the formation of the same complex species in 10^{-3} mol L^{-1}, 1 mol L^{-1} and 3 mol L^{-1} DNO_3 solution. This observation disproves the hypothesis by Wagner *et al.* regarding the formation of further complex species besides the known 1:2 metal-to-ligand complexes [8,10].

3.2 Kinetics experiments using solvent extraction

To further investigate the slow heavy Ln(III) complexation with SO_3-Ph-BTBP, the kinetics of trivalent metal ion stripping into the aqueous phase containing SO_3-Ph-BTBP was examined in solvent extraction studies. Stripping studies were conducted by shaking an organic phase loaded with trivalent metal ions (Ln (w/o Pm), Y, Am, Cm) against a 3 mol L^{-1} HNO_3 solution containing 0.01 mol L^{-1} SO_3-Ph-BTBP. After 7 h shaking, metal ions remained in the organic phase, as indicated by distribution ratios D > 100. Apparently, trivalent metal ion complexation with TODGA in the organic phase is stronger than the complexation with SO_3-Ph-BTBP at 3 mol L^{-1} HNO_3. Since the AmSEL system is designed to selectively strip Am from Cm and the Ln at lower nitric acid concentrations, the stripping kinetics was also investigated using 0.7 mol L^{-1} HNO_3 solution. Fig. 3 (left) shows the distribution ratios of La, Ce, Pr, and Nd as well as Am and Cm for stripping from an organic phase containing 0.2 mol L^{-1} TODGA into a 0.7 mol L^{-1} HNO_3 solution containing 0.01 mol L^{-1} SO_3-Ph-BTBP. The equilibrium is already reached after 2 min, showing a clear separation of Am from Cm ($SF_{Cm/Am}$ = 2.7) and the light Ln (e.g., $SF_{La/Am}$ = 14). This

indicates a fast decomplexation of the TODGA complexes as well as a fast complexation of Am, Cm, and the light Ln with SO_3-Ph-BTBP. The other lanthanides Sm to Lu remained in the organic phase with D > 100 within 170 min of shaking analogously to the stripping study at 3 mol L^{-1} HNO_3. As this also indicates a stronger Ln complexation with TODGA than with SO_3-Ph-BTBP, kinetics studies were carried out for the metal ion extraction into the organic phase from nitric acid solution containing SO_3-Ph-BTBP (in contrast to stripping from a loaded solvent).

Fig. 3 (right) shows the Eu, Am, and Cm distribution ratio as a function of time for extraction from 3 mol L^{-1} HNO_3 solution containing 0.01 mol L^{-1} SO_3-Ph-BTBP into an organic solution with 0.2 mol L^{-1} TODGA. Usually, the equilibrium for the metal extraction with TODGA (without SO_3-Ph-BTBP) is established very fast [16]. Here, however, the distribution equilibrium is reached after ca. 20 min for Eu and ca. 60 min for Am and Cm, showing a strong inhibition of the metal extraction in the presence of SO_3-Ph-BTBP. Interestingly, the Am/Cm selectivity is shifted in the beginning, as D_{Am} is higher than D_{Cm} for < 60 min. Probably, the decomplexation of the [Cm(SO_3-Ph-BTBP)$_2$] complex is kinetically more inhibited than the [Am(SO_3-Ph-BTBP)$_2$] complex.

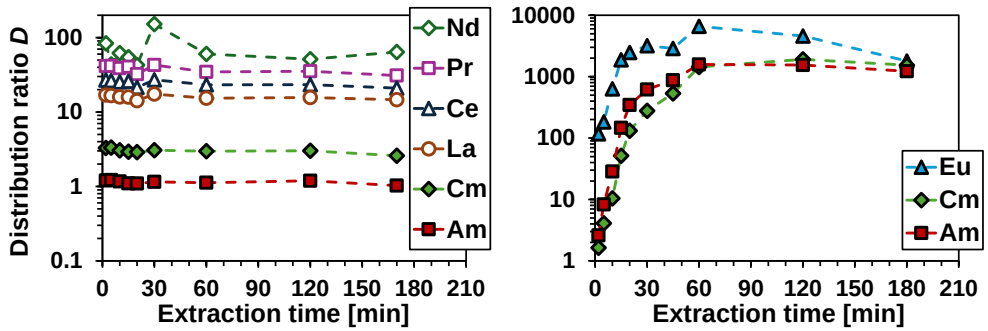

Fig. 3. Distribution ratios of different Ln(III), Am(III), and Cm(III) as a function of extraction time for stripping into 0.7 mol L^{-1} HNO_3 (left) or extraction from 3 mol L^{-1} HNO_3 solution (right), shaking at 22°C ± 1°C with 2500 rpm. Org. phase: 0.2 mol L^{-1} TODGA in 5 vol% 1-octanol/Isane® IP175, pre-equilibrated with 0.7 mol L^{-1} HNO_3 (left) and 3 mol L^{-1} HNO_3 (right). Aq. phase: 0.01 mol L^{-1} SO_3-Ph-BTBP in 0.7 mol L^{-1} HNO_3 (left) and in 3 mol L^{-1} HNO_3 (right).

The kinetically inhibited SO_3-Ph-BTBP decomplexation becomes even more apparent for the heavy Ln (Tb – Lu). Fig. 4 compares the Ln(III) and Y(III) distribution ratios for extraction into an organic TODGA phase from 0.7 mol L^{-1} HNO_3 (left) and 3 mol L^{-1} HNO_3 solution (right), each containing 0.01 mol L^{-1} SO_3-Ph-BTBP. In both cases, metal extraction is strongly inhibited, with the inhibition being more pronounced for heavier Ln. Since metal extraction using TODGA is usually fast [16], this kinetics inhibition is related to the decomplexation of the [Ln(SO_3-Ph-BTBP)]$_2$ complexes. Interestingly, the inhibition of the heavy Ln and Y extraction from 3 mol L^{-1} HNO_3 (Fig. 4, right) seems to be stronger than from 0.7 mol L^{-1} HNO_3 solution (Fig. 4, left), indicated by the lower increase of the distribution ratios over the same extraction time.

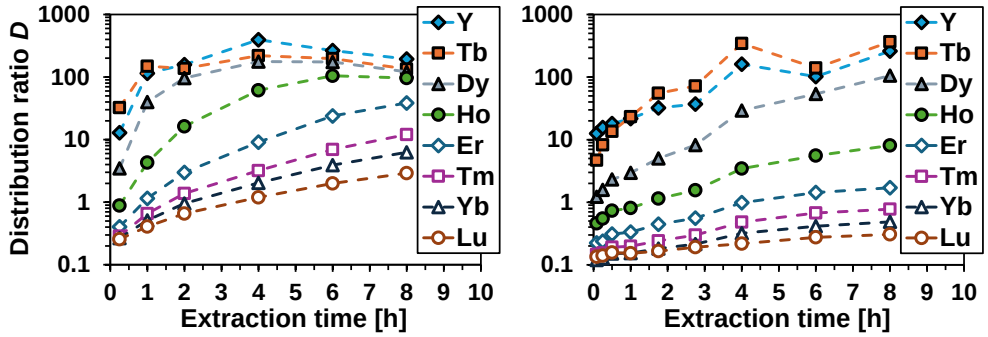

Fig. 4. Distribution ratios as a function of extraction time for extraction of Ln(III) (Tb-Lu) and Y(III) from HNO_3 solution, shaking at 22°C ± 1°C with 2,500 rpm. Org. phase: 0.2 mol L^{-1} TODGA in 5 vol% 1-octanol/Isane® IP175, pre-equilibrated with 0.7 mol L^{-1} HNO_3 (left) and 3 mol L^{-1} HNO_3 (right). Aq. phase: 0.01 mol L^{-1} SO_3-Ph-BTBP in 0.7 mol L^{-1} HNO_3 (left) and in 3 mol L^{-1} HNO_3 (right).

To allow comparison of both data sets, equilibrium distribution ratios of Ln and Y under both conditions were determined by long-term experiments. Equilibrium was reached within 7 days. Higher equilibrium distribution ratios of the heavy Ln and Y were found for extraction from 3 mol L^{-1} HNO_3 compared to 0.7 mol L^{-1} HNO_3 solution, as expected [9]. Therefore, it is concluded that the decomplexation of the SO_3-Ph-BTBP complexes with the heavy Ln and Y is kinetically more inhibited in 3 mol L^{-1} HNO_3 than in 0.7 mol L^{-1} HNO_3 solution. Further investigations are planned to better understand the decomplexation mechanism and thus the kinetics inhibition.

3.3 Comparison of TODGA, Me-TODGA, and Me_2-TODGA with SO_3-Ph-BTBP

Table 1. Eu/Am and Cm/Am separation factors for batch extraction using 0.2 mol L^{-1} (methylated) TODGA in n-dodecane against HNO_3 solution without and in presence of 0.01 mol L^{-1} SO_3-Ph-BTBP, shaking for 15 min at 22°C ± 1°C with 2,500 rpm.

	Without SO_3-Ph-BTBP			With SO_3-Ph-BTBP		
	TODGA	Me-TODGA	Me_2-TODGA	TODGA	Me-TODGA	Me_2-TODGA
$SF_{Eu/Am}$	6.6	4.6	2.2	630	240	37
$SF_{Cm/Am}$	1.5	1.4	0.9	2.6	2.0	1.2

Mono- and di-methylated derivatives Me-TODGA and Me_2-TODGA (Fig. 1) show a shift in the Ln(III) extraction pattern [16]. Therefore, the replacement of TODGA by its mono- and di-methylated derivatives was tested for potential improvement of the AmSEL process. Batch extractions were conducted shaking 0.2 mol L^{-1} TODGA, Me-TODGA and Me_2-TODGA solutions in *n*-dodecane for 15 min against different HNO_3 solutions with and without addition of 0.01 mol L^{-1} SO_3-Ph-BTBP. Without SO_3-Ph-BTBP, distribution ratios increased with increasing HNO_3 concentration in the order Me_2-TODGA < Me-TODGA < TODGA, as known from literature [16,17]. All three extractants exhibit a small Eu/Am selectivity, increasing in the same order Me_2-TODGA < Me-TODGA < TODGA, as shown in Table 1. Moreover, TODGA and Me-TODGA show a similar small selectivity regarding the Cm/Am separation, whereas Me_2-TODGA shows no Cm/Am selectivity (Table 1), probably caused by the existence of two different diastereomers with contrasting Cm/Am selectivity [15].

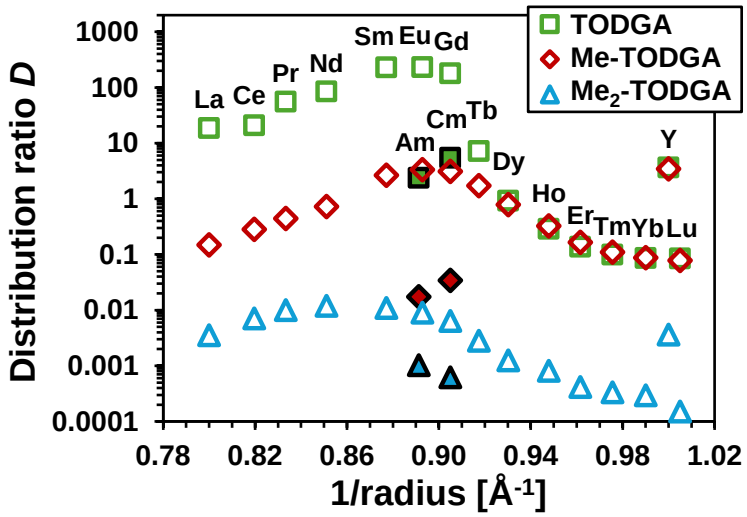

Fig. 5. Distribution ratios of Ln(III) (w/o Pm) and Y(III) (open symbols), as well as Am(III), and Cm(III) (closed symbols) as a function of the inverse ionic radius for nine-fold coordination [18-20] for batch extraction using 0.2 mol L^{-1} TODGA (□), Me-TODGA (◇) and Me_2-TODGA (Δ) in n-dodecane and 0.01 mol L^{-1} SO_3-Ph-BTBP in 1 mol L^{-1} HNO_3 solution, shaking for 15 min at 22°C ± 1°C with 2,500 rpm.

When adding SO_3-Ph-BTBP, trivalent metal ions were increasingly withheld in the aqueous phase, resulting in lower distribution ratios compared to extraction without SO_3-Ph-BTBP (Fig. 5). Due to the

higher An/Ln as well as Am/Cm selectivity of BTBP-type ligands, separation factors increased in presence of SO_3-Ph-BTBP (Table 1). However, the combination of SO_3-Ph-BTBP with the methylated TODGA derivatives results in inferior extraction performance, as the Eu/Am as well as Cm/Am selectivity decreases with increasing degree of methylation in the order TODGA > Me-TODGA > Me_2-TODGA. Fig. 5 compares the distribution ratios of Ln (w/o Pm), Y, Am, and Cm as a function of the inverse ionic radius for nine-fold coordination [18-20] for the different batch extractions using (methylated) TODGA and SO_3-Ph-BTBP at 1 mol L^{-1} HNO_3. The same trend (Me_2-TODGA < Me-TODGA < TODGA), as for the extraction without SO_3-Ph-BTBP, is apparent. A clear separation of Am(III) and Cm(III) from the light Ln(III) is observed due to the stronger SO_3-Ph-BTBP complexation of the An(III) compared to the Ln(III). In all cases, a decrease of the heavy Ln distribution ratios is observed, as described by Wagner *et al.* using TODGA and SO_3-Ph-BTBP [8]. The equilibrium for the heavy Ln was not reached within 15 min shaking, due to the kinetically inhibited [Ln(SO_3-Ph-BTBP)$_2$] decomplexation, as discussed above. The same observation was made for the Am(III) and Cm(III) distribution ratios at > 1 mol L^{-1} HNO_3. Considering the lower extraction performance of the methylated TODGA derivatives in combination with SO_3-Ph-BTBP as well as their inferior An/Ln and Am/Cm selectivity in comparison to the combination of TODGA with SO_3-Ph-BTBP, it must be concluded that the methylated TODGA derivatives do not improve the current AmSEL process.

4. Conclusions

NMR spectroscopic and solvent extraction studies were performed to improve the understanding of the An(III) and Ln(III) (de-)complexation with SO_3-Ph-BTBP in the AmSEL system. NMR spectroscopy indicated the existence of the same SO_3-Ph-BTBP complexes with Lu(III) in 10^{-3} mol L^{-1}, 1 mol L^{-1} and 3 mol L^{-1} DNO_3 solution. Kinetics studies using solvent extraction at 0.7 mol L^{-1} HNO_3 showed fast SO_3-Ph-BTBP complexation with Am(III), Cm(III) and the lanthanides La(III) – Nd(III) in stripping mode. The extraction of heavy Ln(III) (Tb – Lu) and Y(III) from 0.7 mol L^{-1} and 3 mol L^{-1} HNO_3 solution into the organic phase was found to be kinetically inhibited, which is related to a slow decomplexation reaction of the SO_3-Ph-BTBP complexes. This effect was more pronounced for extraction from 3 mol L^{-1} HNO_3 than from 0.7 mol L^{-1} HNO_3 solution. Mono- and di-methylated TODGA derivatives showed no improvement regarding An/Ln or Am/Cm selectivity.

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