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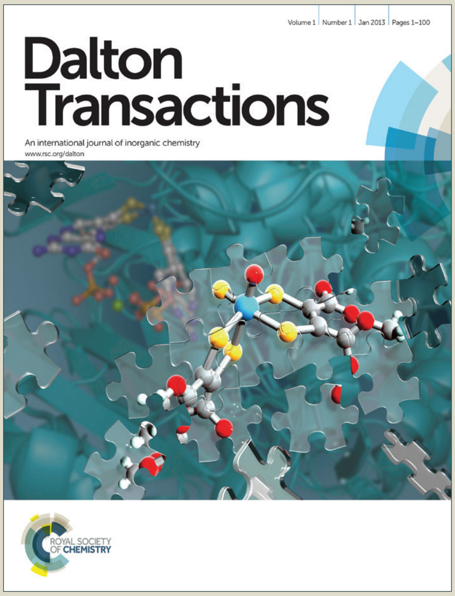

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Structural and spectrophotometric study on the complexation of Am(III) with TMOGA in comparison to the extracted complex of DMDOOGA

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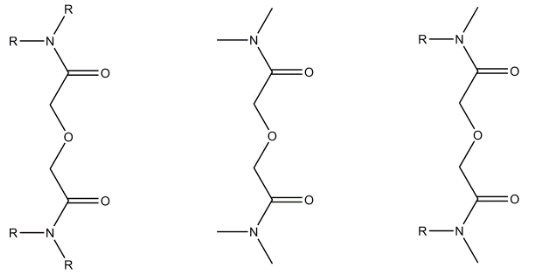
Complexation of Am(III) with tetramethyl-3-oxa-glutaramide (TMOGA, L^I) is studied by spectrophotometric titrations and single crystal X-ray diffraction. Three successive complex species, $[AmL^I]^{3+}$, $[AmL^I_2]^{3+}$, and $[AmL^I_3]^{3+}$, have been identified and their stability constants are calculated to be 3.71 ± 0.012, 5.95 ± 0.021, and 6.93 ± 0.034 respectively, from the absorption spectra collected from the titrations of Am(III) with L^I at 25°C in 1 M $NaNO_3$. Single crystals of $AmL^I_3(ClO_4)_3$ have been grown from $HClO_4$ solution containing Am^{3+} and L^I. The crystal structure of $AmL^I_3(ClO_4)_3$ shows Am(III) is coordinated by nine oxygen atoms from three L^I ligands. The deconvoluted UV-Vis absorption spectrum of $[AmL^I_3]^{3+}$ in aqueous solution is nearly identical to the diffusion reflectance spectrum of $AmL^I_3(ClO_4)_3$ in the solid state, indicating the coordination geometry of the complexes are nearly the same. In addition, to get parallel with solvent exaction, the extracted Am(III) complex with N,N'-dimethyl-N,N'-dioctyl-3-oxa-glutaramide (DMDOOGA, L^{II}) is also prepared and is studied with spectrophotometry. The similarity in UV-Vis absorption of the extracted complex of Am(III) with L^{II} and $[AmL^I_3]^{3+}$ suggests that Am(III) ion is also coordinated by three tridentate L^{II} ligands existing as $[AmL^{II}_3]^{3+}$ in the organic phase of solvent extraction.

Introduction

A huge amount of spent nuclear fuel has been accumulated during energy generation at nuclear power plants worldwide. Spent nuclear fuel consists of mainly uranium (~96%), fission products (~3%), plutonium (~1%), and very small amount of minor actinides. Chemical processing of spent nuclear fuel can recover most fission materials, mainly plutonium and uranium, and separate other actinides from fission products. The benefit of chemical processing is that the volume of radioactive hazards associated with geological disposal of long-lived radioactive isotopes can be significantly reduced and therefore, the overall cost of nuclear fuel cycle can be dramatically decreased. The further separation and treatment of radioactive waste, such as transmutation of the actinides, in particular for americium, is essential to achieve the aforementioned goal of significant volume reduction for radioactive wastes. Accordingly, a multitude of new ligands for actinide separations have been developed to recover minor actinides from high level liquid waste generated from the well-developed PUREX (acronym standing for **P**lutonium **U**ranium **R**edox **EX**traction) process[1]. Among the new ligands, N,N,N′,N′-tetraalkyl-3-oxa-glutaramide (TROGA) molecules (Scheme 1), particularly those with long-chain alkyl substituents (e.g., *n*-octyl) amide, have attracted increasing attention for selectively extracting actinide and lanthanide ions from acidic solutions in last two decades. This development results from the affinity of TROGA molecules with long-chain alkyls to actinides (An) in a full range of oxidation states (+3, +4, +5, and +6) as well as the trivalent lanthanides in solvent extraction.[2-9]

The water-insoluble TROGA ligands have clear uses as extractants to recover actinide ions from acidic aqueous solutions and are being further developed for this purpose. It was recently found that water-soluble TROGA ligands with small alkyl groups (*ca.* ethyl) also have potential application for the separation of Am(III) and Cm(III) from each other.[10]



TROGA L^I:TMOGA L^{II}:DMDROGA

Scheme 1 The structure of related ligands.

Despite the promising results exhibited by using water-insoluble TROGA ligands as an extractant in the organic phase and by using water-soluble homologues as a chelate in aqueous solution, the detailed fundamental mechanisms for the preferential separation of actinides and lanthanides from other metal ions remain unclear. Analyses based on solvent extraction experiments reveal some inconsistencies and have initiated controversies over the extraction reactions and the exact nature of the extracted complexes of lanthanide and actinide ions with the TROGA ligands. The extracted complexes of Ln(III)/An(III) are expected to contain three tridentate TROGA ligands surrounding the central metal ion and providing nine oxygen donors to it, which are supported by the results from some experiments.[2-5] However, other studies have suggested that the formula of the complexes includes the coordination of four TROGA molecules.[11] In solid uranium complexes, uranyl can be coordinated by two TROGA or by one TROGA and two nitrate ions, while in the reported Ln(III) and An(IV) complexes, direct bonding of nitrate to metal ions has not been observed.[12-15] For Pu(IV) in solvent extraction, the dependency of Pu(IV) extraction on ligand concentration indicates that Pu(IV) might be coordinated by a single TROGA ligand in addition to four nitrate ions in the organic phase. Complicating the interpretation in all of the studies is the lack of knowledge on the elusive participation of nitrate in the actinide complexes with TROGA in the organic phase during solvent extraction.

The water-soluble TROGA ligands, as mentioned above, might play an essential role for the separation of Am and Cm from each other in the chemical processing of nuclear wastes. The separation of Am and Cm from each other has been a great challenge, resulting from the nearly identical chemical properties of Am(III) and Cm(III), and from the unstable oxidation states of Am other than +3 in the presence of organic materials, such as organic resins, solvents, and ligands. It is interesting that in the presence of N,N,N′,N′-tetraethyl-3-oxa-glutaramide the separation of Am(III) and Cm(III) can be improved in some extraction systems, while the separation between adjacent lanthanide ions is not significantly affected.

To develop effective processes for separating Am(III) and Cm(III) from each other, at first, ligands that can differentiate these two metal ions are required. Secondly, appropriate methods and techniques that can distinguish the subtle differences between the two metal ions are needed. Therefore, exploring the role of water-soluble TROGA ligands for the potential application to the Am(III) and Cm(III) separation should be instructive and is necessary for developing more efficient ligands.

Furthermore, to gain an insight into the mechanism of actinide and lanthanide separation by TROGA, a systematic investigation on the complexation of TMOGA (L^I in Scheme 1), a ligand in the TROGA family with R=CH_3, with lanthanide and actinide ions has been carried out in our group. In the present work, the emphasis is placed on the complexation of TMOGA with Am(III) studied by spectrophotometry and single crystal X-ray diffraction. The direct comparison of TMOGA complexation of Am(III) to Cm(III) is the subject of current research and will be reported in the future. Besides, to provide parallels to the extracted TROGA complexes, an extracted Am(III) complex with another TROGA ligand, DMDOOGA (L^{II}, shown in Scheme 1 with R=n-octyl), is prepared and the UV-Vis absorption spectrum of the DMDOOGA complex is compared with the spectra of $[AmL^I_3]^{3+}$ obtained from both aqueous solution and the single crystals.[14-16]

To distinguish the coordination properties from the resulting complexes, UV-Vis absorption spectrophotometry is employed utilizing the prominent Am(III) ion band at about 503 nm. The absorption band is very sensitive to the coordination environment, being shifted or split by the presence of ligands. Therefore, UV-Vis spectrophotometry is used characterize the complexation of Am(III) with two aforementioned TROGA ligands by monitoring the variation of the band at about 503 nm.[17]

Experimental

Chemicals and Preparation of Am(III) Samples

The two TROGA ligands used in study, the first of which is N,N,N',N'-tetramethyl-3-oxa-gutaramide (L^I), was previously prepared in this laboratory; whereas the second, oily N,N'-dimethyl-N,N'-dioctyl-3-oxa-glutaramide (L^{II}) was prepared by a similar method and its purity was determined to be > 98% by H^1-NMR.[16,18] Other chemicals were reagent grade or higher.

Milli-Q water was used in preparing all solutions. Carrier-free ^{243}Am was used in this work. The samples of Am(III) in 1 M $NaNO_3$ solution were prepared as follows. A 0.1 mL solution of 0.01 M HCl containing about 0.01-0.1 mg of Am(III) was dried in a small vial at about 150 °C on a hot plate and the residue was dissolved with 0.5 mL of 0.01 M HNO_3. The drying and dissolving process was repeated twice to convert $AmCl_3$ to $Am(NO_3)_3$. The residue was then dissolved with 1 mL of 1 M $NaNO_3$ of pH 3. The concentration of Am(III) was determined by measuring the intensity of the absorption band of Am(III) at 503 nm with molar-absorptivity of 423 cm^{-1} M^{-1} in 1 M $HClO_4$.[17]

Absorption Spectra

The absorption spectra of Am(III) were collected on a Cary6000i spectrometer with 0.05 nm intervals and 0.2 nm spectral band width. Quartz cells of 10 mm path length were used. The temperature of the samples was controlled within ± 0.2 °C with a temperature controller and a water bath. The stability constants of the Am(III) complexes were calculated by nonlinear least-squares regression using the Hyperquad program.[19]

Single Crystal X-ray Diffractometry.

Pale pink crystals of $AmL^I_3(ClO_4)_3$ were obtained from 0.1 mL of 1 M $HClO_4$ solution containing 0.05 mg of Am(III) and 0.2 mg of TMOGA by slow evaporation. The X-ray diffraction data were collected at 200 K using a Bruker APEXII CCD detector and D85 diffractometer with synchrotron radiation at beamline 11.3.1 of the Advanced Light Source at Lawrence Berkeley National Laboratory. Intensity data were collected within one hour using Bruker Apex 2 software.[20] Intensity data integrations, cell refinement and data reduction were performed using the Bruker SAINT software package.[21] Absorption correction was made with SADABS.[21] Dispersion factors (f' and f'') at 16 keV for C, N, O and Am atoms were calculated using CROMER for Windows through WinGX.[23, 24] The structure was solved with direct methods using SHELXS and refined using SHELXL.[25] All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed geometrically on the carbon atoms then constrained and refined using a riding model. Geometrical and displacement parameter restraints were used to model the perchlorate counter ions. The crystal data and structure refinement for the complex are shown in Table 1.

Table 1. Crystal data and structure refinement for $Am(L^1)_3(ClO_4)_3$.

Complex	$Am(L^1)_3(ClO4)_3$
Chemical formula	C_{24} H_{48} Cl_3 N_6 Am O_{21}
Formula weight	1106.03
Temperature	200(2) K
Radiation, wavelength	Synchrotron, 0.7749 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell parameters	a = 19.659(4), α = 90° b = 13.415(3) Å, β = 100.608(3)° c = 15.608(3), γ = 90°
Cell volume	4045.9(15) $Å^3$
Z	4
Calculated density	1.816 g/cm^3
Absorption coefficient	2.631 mm^{-1}
F(000)	2192
Crystal color and size	Pale pink block, 0.040 × 0.030 × 0.020 mm^3
Reflections for cell refinement	5389 (range 2.30 to 27.89°)
Data collection method	Bruker APEXII CCD
Range for data collection	2.602 to 29.855°
Index ranges	h −24 to 25, k −17 to 17, l −19 to 19
Completeness	99.7% (to theta = 29.000°)
Reflections collected	17209
Independent reflections	4462 (R(int) = 0.0692)
Reflections with F^2>2	3740
Absorption correction	semi-empirical from equivalents
Max. and min. transmission	0.949 and 0.790
Structure solution	direct methods
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4462 / 55 / 274
Final R indices [F^2>2]	R_1 = 0.0428, wR_2 = 0.1110
R indices (all data)	R_1 = 0.0539, wR_2 = 0.1167
Goodness-of-fit on F^2	1.004
Largest and mean shift/su	0.029 and 0.000
Largest diff. peak and hole	0.780 and -0.935 e/$Å^3$

Results and discussion

Stability constants of Am(III) complexes

Figure 1 shows a representative set of absorption spectra for the titrations of Am(III) with TMOGA. In the titration, two significant changes in the spectra are observed as the concentration of the ligand is increased: 1) the absorption bands of Am(III) are red-shifted, and successively new bands emerge with previous ones disappearing; and 2) the bands split and exhibit more detailed manifolds.

Analysis with the Hyperquad program[19] indicates that the spectral changes during the Am(III)-TMOGA titration can be ascribed to the successive formation of three Am(III) complexes. The deconvoluted spectra of the complexes clearly illustrate the differences in the Am(III) absorption spectra with the successive formation of the complexes. The stability constants, logβ, are calculated to be 3.71 ± 0.012, 5.95 ± 0.021, and 6.93 ± 0.034, respectively for the 1:1, 1:2, and 1:3 Am(III)-TMOGA complexes. The titrations are necessarily performed in 1 M $NaNO_3$ instead of 1 M $NaClO_4$ to avoid the limited solubility of the complexes in 1 M $NaClO_4$. In fitting data from the titrations of Am(III) with TMOGA, reference spectral contributions to account for the Am(III)/nitrate complex, $Am(NO_3)^{2+}$, are included by incorporating the standard spectrum and stability constant of $Am(NO_3)^{2+}$ obtained from a separate set of spectrophotometric titrations of Am(III) with nitrate. The value of the stability constant, logβ, for $Am(NO_3)^{2+}$ was determined to be (0.063 ± 0.05) at I = 1 M and 25°C.[17]

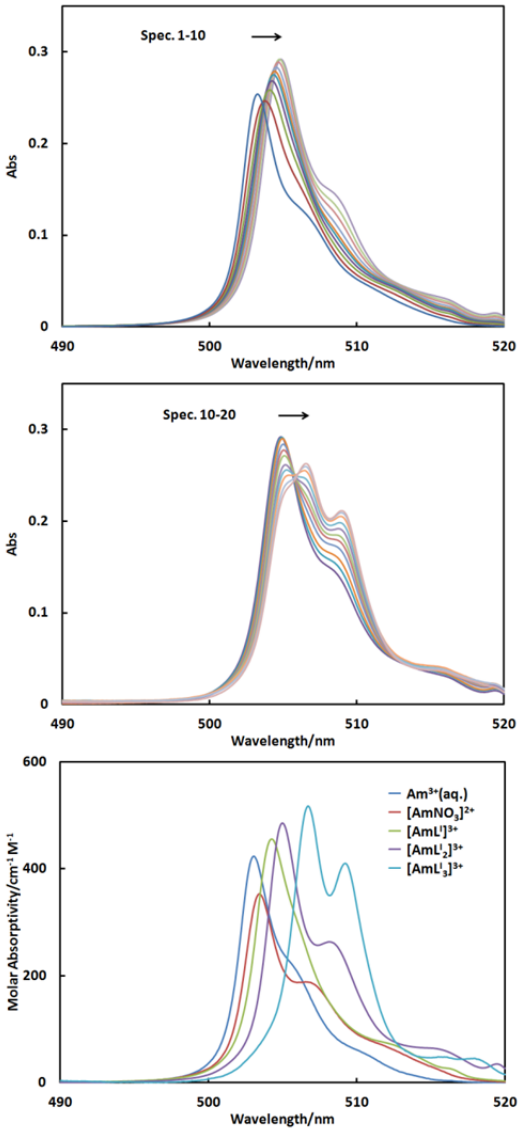


Figure 1 Representative spectrophotometric titration of Am(III)-TMOGA complexation. Upper and middle: Spectra collected during a titration (normalized); Lower: molar absorption spectra from deconvolution. Starting solution: 1 M $NaNO_3$ at pH 3, V = 1.000 mL, $C_{Am(III)}$ = 0.671 mM; titrant solution: 0.200 M TMOGA in 1 M $NaNO_3$ at pH 3.

Crystal structure of $AmL^I_3(ClO_4)_3$

Three Am(III)-L^I complex species, the 1:1, 1:2, and 1:3, have been identified in the solutions by UV-Vis spectrophotometry and the resulting fits. Thus, there have been intensive efforts to prepare a single crystal of each species. However, even starting with a ratio of ligand to metal ion as low as 1:1 in the solutions, only the 1:3 complex with perchlorate has successfully yielded single crystals of sufficient quality for single crystal X-ray diffraction.

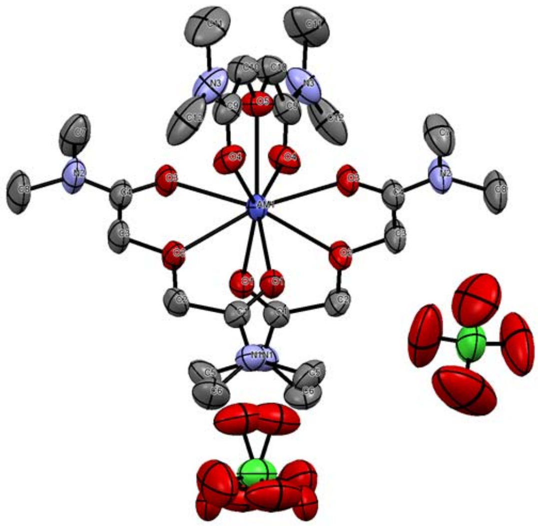


Figure 2 ORTEP drawing (50% probability ellipsoids) of $AmL^{I}{}_3{}^{3+}$ viewed down the c*-axis. Am-blue, oxygen-red, nitrogen-light blue, carbon-gray, chlorine-green, for clarity hydrogen atoms are not shown.

The 1:3 perchlorate salt complex, $AmL^{I}{}_3(ClO_4)_3$, crystallizes in the monoclinic space group C2/c. There are four molecules of the $[AmL^{I}{}_3]^{3+}$ complex and twelve perchlorate counterions in the C-centered unit cell. Similar to the coordination of Ln(III) or An(IV) in the 1:3 complexes with TROGA ligands, the nine-coordinated Am(III) has a distorted tricapped trigonal prism geometry with six carboxyl oxygen atoms at the corners and three ether oxygen atoms capped on the three faces.[13-15] The two triangular faces are slightly rotated relative to each other. The Am(III) center and the three capping ether oxygen atoms are coplanar.

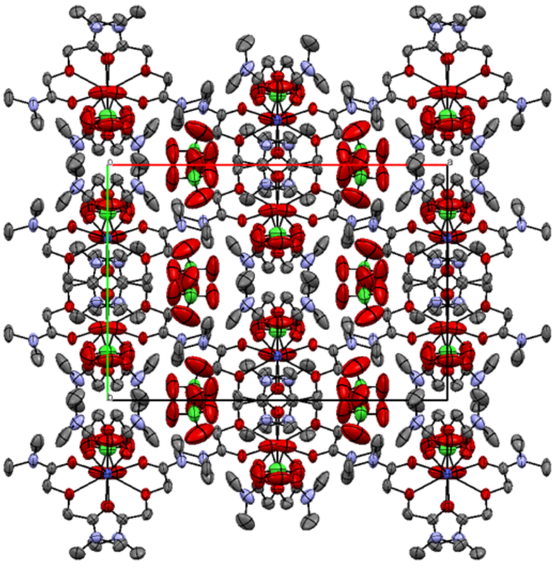


Figure 3. Packing structure of $AmL^{I}{}_3(ClO_4)_3$ viewed down the c-axis, showing the isolated nature of the complex and disordered perchlorates. Hydrogen atoms have been omitted for the sake of clarity. Oxygens-colored red, carbons-gray, americiums-blue, and nitrogens-light blue.

Because there are neither hydrogen bond donors, in the $[AmL^{I}{}_3]^{3+}$ complex or perchlorate ion, nor π interactions between the ion pair, the crystal structure is essentially comprised of isolated $[AmL^{I}{}_3]^{3+}$ complexes surrounded by perchlorate counterions that sit in the space among the complexes. As a result, the perchlorate laying on the 2-fold axis is found to be disordered about the axis. The chlorine is located just on-axis, while one oxygen atom is set off-axis over two positions and the three remaining oxygen atoms are disordered over six positions across the 2-fold axis. The perchlorate that is located in a general position shows no disorder. A packing structure of $AmL^{I}{}_3(ClO_4)_3$ viewed down the c-axis is shown in Figure 5.

Table 2. Selected bond lengths of $Am(L^{I})_3(ClO_4)_3$ in comparison with Nd(III) complex.

Bond	Bond length/Å	
	Am	Nd
Am/Nd-O (corner)	2.401(4)	2.402(3)
	2.432(4)	2.422(3)
	2.451(5)	2.449(3)
	2.401(4)	2.402(3)
	2.432(4)	2.422(3)
	2.451(5)	2.449(3)
Average	2.428(4)	2.424(3)
Am/Nd-O (capping)	2.508(6)	2.526(4)
	2.525(4)	2.533(3)
	2.525(4)	2.533(3)
Average	2.519(5)	2.531(6)

Am(III), although of comparable ionic size, forms slightly more stable complexes than Nd(III) with TMOGA in aqueous solution. As listed in Table 2, the solid $AmL^{I}{}_3(ClO_4)_3$ perchlorate salts have an average $Am\text{-}O_{(C=O)}$ bond length that is slightly longer than the $Nd\text{-}O_{(C=O)}$, whereas the $Am\text{-}O_{(C\text{-}O\text{-}C)}$ bond length is slightly shorter than the $Nd\text{-}O_{(C\text{-}O\text{-}C)}$ length. However, because the differences are small and actually within experimental error, the structural differences between the Am(III) and Nd(III) complexes cannot be attributed to the very small difference in complex stabilities.[14]

Absorption/Reflectance Spectra of Am(III) Complexes

The solid $AmL^{I}{}_3(ClO_4)_3$ compound is analyzed by diffuse reflectance UV-Vis spectroscopy for comparison to the 1:3 complex in solution, thereby providing useful information about the absorption spectra of Am(III) and the structure of the complexes. The shape of diffuse reflectance spectrum of the solid $AmL^{I}{}_3(ClO_4)_3$ compound is nearly identical to the absorption spectrum of the 1:3 complex in solution, as shown in Figure 3. This indicates the structures of the 1:3 complexes are nearly the same in the solid state as in solution.

To further understand the extraction of Ln(III)/An(III) with TROGA extractants, an Am(III) complex with DMDOOGA (L^{II}) is prepared by extraction and UV-Vis absorption spectrum is recorded. The comparable shape of the resultant DMDOOGA spectra with both the spectra of $AmL^{I}{}_3{}^{3+}$ in solution and in the solid state (as shown in Figure 3), suggests that the coordination geometry of Am(III) in the extracted complex with DMDOOGA is similar to that in $AmL^{I}{}_3{}^{3+}$. This spectral similarity indicates that nitrate might not directly bond to Ln(III)/An(III) centers in the solvent extraction system but rather acts as counterion in the form of ion pair to balance the positive charge of the 1:3 complexes.

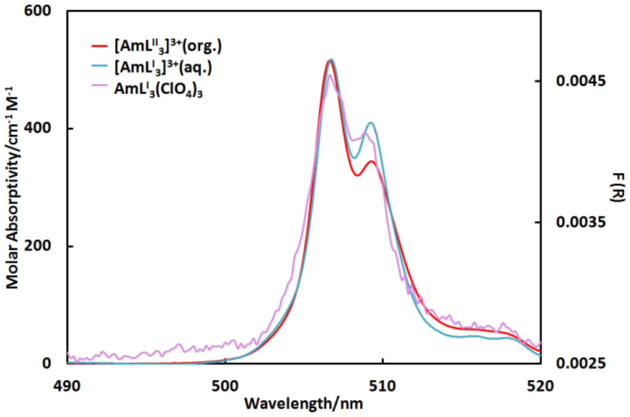


Figure 4 Diffuse reflectance spectra of solid $\mathrm{AmL^{I}_3(ClO_4)_3}$ compound (purple, right axis) in comparison with the absorption spectra of $[\mathrm{AmL^{I}_3}]^{3+}$ in aqueous solution (blue, left axis), and of extracted $[\mathrm{AmL^{II}_3}]^{3+}$ in organic phase(red, left axis).

Summary

In conclusion, three Am(III) complexes with TMOGA have been identified and the stability constants have been determined by spectrophotometry in 1 M $NaNO_3$ at 25 °C. Single crystals of the $\mathrm{AmL_3(ClO_4)_3}$ compound were grown from aqueous solution by slow evaporation and analyzed by X-ray diffraction. In the solid compound, the nine-coordinated Am(III) has a distorted tricapped trigonal prism geometry with six carboxyl oxygen atoms at the corners and three ether oxygen atoms capped on the three faces. The similarity of UV-Vis spectra suggests the 1:3 Am(III)-TMOGA complex exists in solution with a nearly identical structure as in the solid $\mathrm{AmL_3(ClO_4)_3}$ compound, and the extracted complex of Am(III) with DMDOOGA might possess a similar coordination geometry. These results provide fundamental information on the complexation of TROGA-class ligands with Ln(III)/Am(III) in solvent extraction. Further studies are needed to systematically investigate the complexation of other An(III), especially Cm(III) and Ln(III) with water-soluble TROGA ligands to reveal the mechanism that results in the preferential separation of Am(III) from Cm(III) while not affecting the separation of An(III) from Ln(III) during the separation process.

Acknowledgement

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Notes and references

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† Electronic Supplementary Information (ESI) available: Cif file for $\mathrm{AmL^{I}_3(ClO_4)_3}$, CCDC 1023259. See DOI: 10.1039/b000000x/

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Table of Content:

The UV/Vis spectra and crystal structure of $AmL^I{}_3{}^{3+}$ (L^I = TMOGA) illustrate that Am(III) forms similar extracted complexes with TROGA extractants.