

A Hydrolytically Stable Complexant for Minor An Separation from Ln in Process Relevant Diluents

Supporting Information

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Experimental Section

Chemicals. All reagents were purchased from U.S. chemical suppliers, stored according to published protocols, and used as received unless indicated otherwise. All aqueous solutions were prepared with Milli-Q type 1 water system.

Small-Scale Dissolution. A small amount of mass, generally between 10 and 20 mg, was accurately weighed out into a sample tube. Ten microliters of the appropriate diluent (Exxal-8 or kerosene) were added to the sample vial and sonicated for five to thirty minutes. This step was repeated until complete dissolution of the complexant was observed. The sample was capped, parafilm, and left for 24 hours to ensure the complexant did not crash out of solution.

Solvent Extraction. Organic phase. An appropriate mass of **1** was transferred from a sample vial to a 5.0 mL Class A volumetric flask to make a concentrated stock solution. Weighing by difference was used on the original sample vial to calculate the actual mass of **1** dissolved. The solvent was added to the flask and manually agitated to facilitate dissolution. Generally, the complexant was fully dissolved after 5 minutes of sonication. Various concentrations of **1** were prepared from the concentrated stock solution in 1.0 or 2.0 mL Class A volumetric glassware.

Aqueous phase. Eight concentrated stock solutions of $^{241}\text{Am}^{3+}$ were prepared in various concentrations of HNO_3 (0.25–5.0 M). These $^{241}\text{Am}^{3+}$ stock solutions were used throughout the entirety of this study to spike the working aqueous phase of the matching $[\text{HNO}_3]$. Initial counts of the aqueous phase were generally 3400–4000 cpm / 200 μL . This method was repeated for $^{154}\text{Eu}^{3+}$. **Extraction.** Equal volumes (0.6 mL) of the organic and active aqueous phase, in that order, were added to a 1.5 mL microcentrifuge tube. The centrifuge tubes were capped and wrapped in parafilm then placed on a pulsing vortex shaker at 1700 rpm for the appropriate time. After the allotted time on the vortex shaker, the samples were centrifuged for five minutes to achieve phase separation. The organic and aqueous phases were transferred to separate 1.5 mL centrifuge tubes with a disposable fine-tip transfer pipette. A 200 μL aliquot of each phase was transferred to a counting tube with sampling performed in duplicate. **Gamma Spectroscopy.** A 2480 Wizard² automatic gamma counter with 3 inch NaI(Tl) was used for the determination of $^{241}\text{Am}^{3+}$ and $^{154}\text{Eu}^{3+}$ activity of samples. Samples were counted for 10 minutes, or until total counts reached 10000, and the instrument software relayed the results as counts per minute (CPM). The distribution ratios of a given metal ion (D_M) were calculated according to Equation 1 where $[M]$ is the net CPM of either the organic phase (numerator) or aqueous phase (denominator). Separation factors (SF) were calculated using Equation 2. Values are reported in figures as an average of duplicate samples with error bars representing uncertainties calculated at 2σ from the counting error. The supporting information for this work provides additional detail.

$$D_M = \frac{[M]_{org}}{[M]_{aq}} \quad \text{Equation (1)}$$

$$SF_{Am(III)/Eu(III)} = \frac{D_{Am(III)}}{D_{Eu(III)}} \quad \text{Equation (2)}$$

UV-vis Titration. UV-visible spectrophotometric titrations were performed in a 3 mL, 1.00 cm path length quartz cuvette, using a Cary 5000 UV-vis spectrophotometer. For each titration, an appropriate volume of a 1.5×10^{-3} M $\text{Ln}(\text{NO}_3)_3$ ($\text{Ln} = \text{Nd}, \text{Eu}$) solution in 99% acetonitrile/1% water (v/v) was added to 2.0 mL of a 1.5×10^{-5} M solution of **1** in the same solvent system. The

total volume of $\text{Ln}(\text{NO}_3)_3$ added was 60 μL . A wavelength scan from 225 to 450 nm at a scan rate of 100 nm per minute and a 5 data point average was taken of the free (uncomplexed) complexant and after each titration. The sample was manually agitated for three minutes after each titration to ensure complete complexation. The titration was complete when the variation in absorbance became negligible. Raw spectra were baseline corrected with OriginPro software and imported into HypSpec software for determination of stability constants by nonlinear regression curve fitting.

Counting Statistics

A critical limit (L_C , Equation S1) was calculated for every counting (solvent extraction) experiment (Tables S1, S2, S4, S5, S7, and S8) to determine if the net count was statistically significant at the 95% confidence level.

$$L_C = 1.645\sqrt{2B} \quad \text{Equation S1}$$

When a net CPM was found to be below the calculated L_C a footnote was added to the data table (Tables S5 and S8) to indicate that the activity is not detected and an upper limit (L_U , Equation S2) at the 95% confidence is reported. The calculated values were left in the raw data tables with a footnote, but these values were not used to report a distribution value in the associated summary table (Tables S6 and S9) or figure (Figure 2).

$$L_U = \text{cpm}_{\text{net}} + 1.645\sqrt{(\text{cpm}_{\text{net}} + 2B)} \quad \text{Equation S2}$$

The uncertainty for each distribution value (D_M , where $M = {}^{241}\text{Am}^{3+}$ or ${}^{154}\text{Eu}^{3+}$) was calculated from the counting error (2σ) according to Equation S3:

$$\sigma_{D_M} = D_M \times \sqrt{\left(\frac{\left(\sqrt{\sigma_{\text{org}}^2 + \sigma_{\text{blank}}^2}\right)^2}{\text{Net cpm}_{\text{org}}}\right)^2 + \left(\frac{\left(\sqrt{\sigma_{\text{aq}}^2 + \sigma_{\text{blank}}^2}\right)^2}{\text{Net cpm}_{\text{aq}}}\right)^2} \quad \text{Equation S3}$$

The uncertainty for the average D_M was calculated according to Equation S4 and reported in the summary tables (Tables S3, S6, and S9).

$$\sigma_{\text{Avg}(D_M)} = \frac{\sqrt{\sigma_{D_1}^2 + \sigma_{D_2}^2}}{2} \quad \text{Equation S4}$$

D_1 and D_2 refer to the individual D_M values from duplicate samples taken for each data point.

Table S1. Raw Data to Accompany Figure 1(a). $^{241}\text{Am}^{3+}$ Distribution (D)-values, at 1M $\text{HNO}_{3(\text{aq})}$ as $f(t)$ in Kerosene/Exxal (50v/50v)

Time (min.)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Am} (org./aq.)	D_{Am} Avg.
5	org. - 1	502	1.28	481	101.0	0.1553	0.1542
	org. - 2	491	1.26	470	99.8	0.1532	
	aq. - 3	3119	1.00	3098			
	aq. - 4	3089	1.00	3068			
10	org. - 5	863	1.00	842	100.6	0.3089	0.3309
	org. - 6	933	1.00	912	98.6	0.3529	
	aq. - 7	2747	1.00	2726			
	aq. - 8	2605	1.00	2584			
20	org. - 9	1509	1.00	1488	98.1	0.7485	0.7627
	org.- 10	1560	1.00	1539	99.3	0.7769	
	aq. - 11	2009	1.00	1988			
	aq. - 12	2002	1.00	1981			
30	org. -13	1813	1.00	1792	96.0	1.112	1.134
	org. -14	1911	1.00	1890	99.4	1.156	
	aq. - 15	1633	1.00	1612			
	aq. - 16	1656	1.00	1635			
45	org. -17	2128	1.00	2107	97.5	1.563	1.590
	org. -18	2206	1.00	2185	99.8	1.616	
	aq. - 19	1369	1.00	1348			
	aq. - 20	1373	1.00	1352			
60	org. -21	2337	1.00	2316	98.1	1.995	2.024
	org.-22	2403	1.00	2382	99.9	2.053	
	aq. - 23	1182	1.00	1161			
	aq. - 24	1181	1.00	1160			
90	org. -25	2341	1.00	2320	97.5	2.042	2.030
	org. -26	2357	1.00	2336	98.6	2.017	
	aq. - 27	1157	1.00	1136			
	aq. - 28	1179	1.00	1158			
120	org. -29	2345	1.00	2324	97.0	2.084	2.104
	org. -30	2470	1.00	2449	101.6	2.124	
	aq. - 31	1136	1.00	1115			
	aq. - 32	1174	1.00	1153			
A ₀	A ₀ - 33	3565	1.00	3544			
A ₀	A ₀ - 34	3567	1.00	3546			
A ₀	Avg.	3566	1.00	3545			
Blank	Avg.	21	6.87				

Table S2. Raw Data to Accompany Figure 1(a). $^{152}\text{Eu}^{3+}$ Distribution (D)-values, at 1M $\text{HNO}_{3(\text{aq})}$ as $f(t)$ in Kerosene/Exxal (50v/50v)

Time (min.)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Eu} (org./aq.)	D_{Eu} (Avg.)
5	org. - 1	197	2.22	46	101.7	0.0125	0.0128
	org. - 2	199	2.22	48	100.5	0.0132	
	aq. - 3	3838	1.00	3687			
	aq. - 4	3794	1.00	3643			
10	org. - 5	199	2.29	48	99.2	0.0134	0.0127
	org. - 6	195	2.22	44	100.4	0.0121	
	aq. - 7	3746	0.99	3595			
	aq. - 8	3794	1.00	3643			
20	org. - 9	200	2.26	49	100.6	0.0134	0.0126
	org. -10	194	2.24	43	100.5	0.0118	
	aq. - 11	3795	1.00	3644			
	aq. - 12	3799	1.00	3648			
30	org. -13	195	2.26	44	100.6	0.0121	0.0131
	org. -14	202	2.28	51	99.5	0.0142	
	aq. - 15	3801	1.00	3650			
	aq. - 16	3752	1.00	3601			
45	org. -17	207	2.25	56	98.4	0.0157	0.0136
	org. -18	193	2.28	42	100.4	0.0115	
	aq. - 19	3707	1.00	3556			
	aq. - 20	3796	1.00	3645			
60	org. -21	200	2.26	49	98.8	0.0137	0.0138
	org-22	201	2.27	50	99.0	0.0139	
	aq. - 23	3730	1.00	3579			
	aq. - 24	3738	1.00	3587			
90	org. -25	193	2.26	42	98.3	0.0118	0.0125
	org. -26	200	2.28	49	102.6	0.0132	
	aq. - 27	3720	1.00	3569			
	aq. - 28	3869	1.00	3718			
120	org. -25	202	2.25	51	98.7	0.0143	0.0141
	org. -26	202	2.34	51	100.5	0.0140	
	aq. - 27	3723	1.00	3572			
	aq. - 28	3792	1.00	3641			
A ₀	A ₀ - 33	3876	1.00	3725			
A ₀	A ₀ - 34	3770	1.00	3619			
A ₀	Avg.	3823	1.00	3672			
Blank	Avg.	151	2.61				

Table S3. Summary D_M for Tables S1 and S2

Time (min)	D_{Am} Avg.	D_{Am} Uncertainty (2σ)	D_{Eu} Avg.	D_{Eu} Uncertainty (2σ)	SF (Avg.) (D_{Am} / D_{Eu})	SF Uncertainty (2σ)
5	0.1542	0.004	0.0128	0.002	12.0	2.2
10	0.3309	0.007	0.0127	0.002	26.0	4.8
20	0.7627	0.015	0.0126	0.002	60.4	11.1
30	1.134	0.023	0.0131	0.002	86.5	15.6
45	1.590	0.032	0.0136	0.002	117	20.4
60	2.024	0.041	0.0138	0.002	147	25.4
90	2.030	0.041	0.0125	0.002	163	30.4
120	2.104	0.043	0.0141	0.002	149	25.4

Table S4. Raw Data to Accompany Figure 2(b). $^{241}\text{Am}^{3+}$ Distribution (D)-values, $f(\text{HNO}_3(\text{aq}))$ with Initial Concentration in Kerosene/Exxal (50v/50v) of 25 mM

[HNO ₃] (M)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Am} (org./aq.)	D_{Am} Avg.
0.25	org. - 1	1175	1.00	1153	100.7	0.4358	0.4503
	org. - 2	1217	1.00	1195	99.9	0.4648	
	aq. - 3	2668	1.00	2646			
	aq. - 4	2593	1.00	2571			
0.5	org. - 5	1687	1.00	1665	97.7	0.9224	0.9431
	org. - 6	1775	1.00	1753	100.6	0.9637	
	aq. - 7	1827	1.00	1805			
	aq. - 8	1841	1.00	1819			
0.75	org. - 9	2399	1.00	2377	101.2	1.799	1.850
	org. -10	2509	1.00	2487	103.9	1.901	
	aq. - 11	1343	1.00	1321			
	aq. - 12	1330	1.00	1308			
1.0	org. -13	2877	1.00	2855	98.3	2.648	2.695
	org. -14	3037	1.00	3015	102.8	2.741	
	aq. - 15	1100	1.00	1078			
	aq. - 16	1122	1.00	1100			
2.0	org. -17	3118	1.00	3096	97.2	10.12	10.25
	org. -18	3220	1.00	3198	100.1	10.38	
	aq. - 19	328	1.75	306			
	aq. - 20	330	1.74	308			
3.0	org. -21	3474	1.00	3452	96.4	13.18	13.45
	org.-22	3673	1.00	3651	101.6	13.73	
	aq. - 23	284	1.88	262			
	aq. - 24	288	1.86	266			
4.0	org. -25	2954	1.00	2932	95.0	8.778	9.006
	org. -26	3152	1.00	3130	100.9	9.233	
	aq. - 27	356	1.68	334			
	aq. - 28	361	1.66	339			
5.0	org. -25	3055	1.00	3033	97.9	9.721	9.699
	org. -26	3109	1.00	3087	99.7	9.677	
	aq. - 27	334	1.73	312			
	aq. - 28	341	1.71	319			
0.25	A ₀ - 33	3793	1.00	3771			
0.50	A ₀ - 34	3572	1.00	3550			
0.75	A ₀ - 35	3674	1.00	3652			
1.0	A ₀ - 36	4024	1.00	4002			
2.0	A ₀ - 37	3523	1.00	3501			
3.0	A ₀ - 38	3875	1.00	3853			
4.0	A ₀ - 39	3460	1.00	3438			

5.0	$A_0 - 40$	3438	1.00	3416
Blank	Avg.	22	6.88	

Table S5. Raw Data to Accompany Figure 2(b). $^{154}\text{Eu}^{3+}$ Distribution (D)-values, $f(\text{HNO}_3(\text{aq}))$ with Initial Concentration in Kerosene/Exxal (50v/50v) of 25 mM

[HNO ₃] (M)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Eu} (org./aq.)	D_{Eu} Avg.
0.25	org. - 1	179	2.36	31	103.1	0.0077	0.0076
	org. - 2	178	2.37	30	102.3	0.0075	
	aq. - 3	4208	1.00	4060			
	aq. - 4	4178	1.00	4030			
0.5	org. - 5	194	2.27	46	102.4	0.0122	0.0139
	org. - 6	208	2.19	60	104.0	0.0156	
	aq. - 7	3955	1.00	3807			
	aq. - 8	4001	1.00	3853			
0.75	org. - 9	277	1.90	129	101.1	0.0253	0.0253
	org. -10	276	1.90	128	100.4	0.0253	
	aq. - 11	5251	1.00	5103			
	aq. - 12	5213	0.99	5065			
1.0	org.- 13	332	1.74	184	100.5	0.0319	0.0326
	org.- 14	341	1.71	193	100.8	0.0334	
	aq. - 15	5927	0.99	5779			
	aq. - 16	5938	0.99	5790			
2.0	org. -17	351	1.69	203	100.7	0.0645	0.0660
	org. -18	363	1.66	215	102.2	0.0675	
	aq. - 19	3298	1.00	3150			
	aq. - 20	3335	1.00	3187			
3.0	org. -21	458	1.48	310	101.2	0.0848	0.0885
	org.-22	482	1.44	334	100.8	0.0923	
	aq. - 23	3808	1.00	3660			
	aq. - 24	3770	1.00	3622			
4.0	org. -25	381	1.62	233	105.9	0.0624	0.0618
	org.- 26	371	1.64	223	103.1	0.0613	
	aq. - 27	3889	1.00	3741			
	aq. - 28	3793	1.00	3645			
5.0	org.- 29	413	1.56	265	102.0	0.0720	0.0724
	org.- 30	418	1.55	270	103.0	0.0727	
	aq. - 31	3832	1.00	3684			
	aq. - 32	3863	1.00	3715			
0.25	A ₀ - 33	4118	1.00	3970			
0.50	A ₀ - 34	3911	1.00	3763			
0.75	A ₀ - 35	5321	1.00	5173			
1.0	A ₀ - 36	6083	1.00	5935			
2.0	A ₀ - 37	3477	1.00	3329			
3.0	A ₀ - 38	4072	1.00	3924			
4.0	A ₀ - 39	3901	1.00	3753			

5.0	A ₀ - 40	4018	1.00	3870
Blank	Avg.	148	2.60	

Table S6. Summary D_M for Tables S4 and S5

[HNO₃] (M)	D_{Am} Avg.	D_{Am} Uncertainty (2σ)	D_{Eu} Avg.	D_{Eu} Uncertainty (2σ)	SF (Avg.) (D_{Am} / D_{Eu})	SF Uncertainty (2σ)
0.25	0.4503	0.009	0.0076	0.002	59.2	15.6
0.50	0.9431	0.019	0.0139	0.002	67.8	10.8
0.75	1.850	0.038	0.0253	0.002	73.1	5.5
1.0	2.695	0.055	0.0326	0.002	82.6	4.8
2.0	10.25	0.316	0.0660	0.003	155	9.2
3.0	13.45	0.444	0.0885	0.003	152	7.6
4.0	9.006	0.267	0.0618	0.003	146	8.1
5.0	9.699	0.295	0.0724	0.003	134	7.0

Table S7. Raw Data to Accompany Figure 2(c). Distribution of $^{241}\text{Am}^{3+}$ as a Function of Complexant Concentration in Kerosene/Exxal (50v/50v) Log-Log plot

[Ligand] (mM)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Am} (org./aq.)	D_{Am} Avg.
6.25	org. - 1	702	1.19	681	101.3	0.2378	0.2424
	org. - 2	741	1.16	720	103.9	0.2470	
	aq. - 3	2885	1.00	2864			
	aq. - 4	2936	1.00	2915			
12.5	org. - 5	2102	1.00	2081	97.5	1.565	1.593
	org. - 6	2165	1.00	2144	99.1	1.621	
	aq. - 7	1351	1.00	1330			
	aq. - 8	1344	1.00	1323			
25.0	org. - 9	2992	1.00	2971	94.4	8.922	9.276
	org. -10	3093	1.00	3072	96.9	9.630	
	aq. -11	354	1.68	333			
	aq. -12	340	1.71	319			
37.5	org. -13	3138	1.00	3117	92.9	23.26	24.06
	org. -14	3327	1.00	3306	98.3	24.86	
	aq. - 15	155	2.54	134			
	aq. - 16	154	2.55	133			
50.0	org. -17	3222	1.00	3201	93.4	48.50	47.27
	org. -18	3336	1.00	3315	96.8	46.04	
	aq. -19	87	3.40	66			
	aq. - 20	93	3.28	72			
62.5	org. -21	3300	1.00	3279	95.0	71.28	81.64
	org.-22	3425	1.00	3404	98.3	92.00	
	aq. - 23	67	3.87	46			
	aq. - 24	58	4.16	37			
75.0	org. -25	3342	1.00	3321	95.8	110.7	110.9
	org. -26	3354	1.00	3333	96.1	111.1	
	aq. - 27	51	4.45	30			
	aq. - 28	51	4.43	30			
A ₀	A ₀ - 29	3540	1.00	3519			
A ₀	A ₀ - 30	3501	1.00	3480			
A ₀	Avg.	3520.5	1.00	3499.5			
Blank	Avg.	21	6.84				

Table S8. Raw Data to Accompany Figure 2(c). Distribution of $^{154}\text{Eu}^{3+}$ as a Function of Complexant Concentration in Kerosene/Exxal (50v/50v) Log-Log plot

[Ligand] (mM)	Sample ID	CPM	Error (%)	Net CPM	Recovery (%)	D_{Eu} (org./aq.)	D_{Eu} Avg.
6.25	org. - 1	147	2.60	-3 ^a	102.6	-0.0009	-0.0001
	org. - 2	152	2.57	2 ^b	103.0	0.0006	
	aq. - 3	3657	1.00	3507			
	aq. - 4	3665	1.00	3515			
12.5	org. - 5	198	2.25	48	103.3	0.0138	0.0125
	org. - 6	189	2.30	39	103.3	0.0112	
	aq. - 7	3629	1.00	3479			
	aq. - 8	3639	1.00	3489			
25.0	org. - 9	382	1.62	232	101.9	0.0714	0.0719
	org. -10	389	1.60	239	103.7	0.0724	
	aq. - 11	3399	1.00	3249			
	aq. - 12	3452	1.00	3302			
37.5	org. -13	712	1.19	562	102.0	0.1924	0.1899
	org. -14	705	1.19	555	103.0	0.1873	
	aq. -15	3071	1.00	2921			
	aq. -16	3113	1.00	2963			
50.0	org. -17	1072	1.00	922	100.6	0.3666	0.3680
	org. -18	1084	1.00	934	101.4	0.3693	
	aq. - 19	2665	1.00	2515			
	aq. - 20	2679	1.00	2529			
62.5	org. -21	1441	1.00	1291	99.1	0.6168	0.6307
	org.-22	1485	1.00	1335	99.7	0.6446	
	aq. - 23	2243	1.00	2093			
	aq. - 24	2221	1.00	2071			
75.0	org. -25	1785	1.00	1635	98.8	0.9407	0.9457
	org. -26	1806	1.00	1656	99.5	0.9506	
	aq. - 27	1888	1.00	1738			
	aq. - 28	1892	1.00	1742			
A ₀	A ₀ - 29	3408	1.00	3257.5			
A ₀	A ₀ - 30	3425	1.00	3274.5			
A ₀	Avg.	3416.5	1.00	3266			
Blank	Avg.	150.5	2.58				

^{a,b}Net CPM is below the critical limit (L_C) and therefore, reported as not detected, $L_C = 29$ (95% confidence level). Upper limits (L_U) at the 95% confidence level: ^a $L_U = \text{NA}$, ^b $L_U = 31$.

Table S9. Summary D_M for Tables S7 and S8

[Ligand] (mM)	D_{Am} Avg.	D_{Am} Uncertainty (2σ)	D_{Eu} Avg.	D_{Eu} Uncertainty (2σ)	SF (Avg.) (D_{Am} / D_{Eu})	SF Uncertainty (2σ)
6.25	0.2424	0.005	--	--	--	25
12.5	1.593	0.032	0.0125	0.002	128	7.1
25.0	9.276	0.278	0.0719	0.003	129	6.9
37.5	24.06	1.12	0.1899	0.005	127	9.5
50.0	47.27	3.31	0.3680	0.009	128	14
62.5	81.64	8.44	0.6307	0.014	129	15
75.0	110.9	14.1	0.9457	0.021	117	25