The complexation and thermodynamics of neptunium(V) with acetate in aqueous solution

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Supporting Information

Chemicals

Table S 1: Used chemicals, supplier and purity.

Chemical Name	Source	Initial Purity	Purification Method	Final Purity	Analysis Method
Ultrapure Water	Milli-Q academic	18.0 MΩ.cm	-	-	-
^{237}Np(V)	INE, KIT[a]	-	Dovex AG 1X 8 anion echange column, Scevanger precipitation, electrolyses	A(^{237}Np) = A(^{233}Pa); 99.9 % Np(V)	UV/Vis/NIR, ICP-MS, LSC, Alpha
$NaClO_4 \cdot H_2O$, Emsure	Merck KGaA	99.0 - 102.0 %	-	-	-
$HClO_4$, Suprapure	Merck KGaA	70 %	-	69 - 71 %	Metrohm Titroprocessor 686, Metrohm Dosimat 665
NaAc, anhydrous, metals basis	Alfa Aesar	99.997 %	-	-	-
NaOH, 0.1 N, Titripure	Merck KGaA	99.5 – 100.5 %	-	-	-

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Single component spectra of $NpO_2(Ac)_n^{1-n}$ at $I_m(NaClO_4)$ = 4.0 mol kg^{-1} as a function of the temperature.

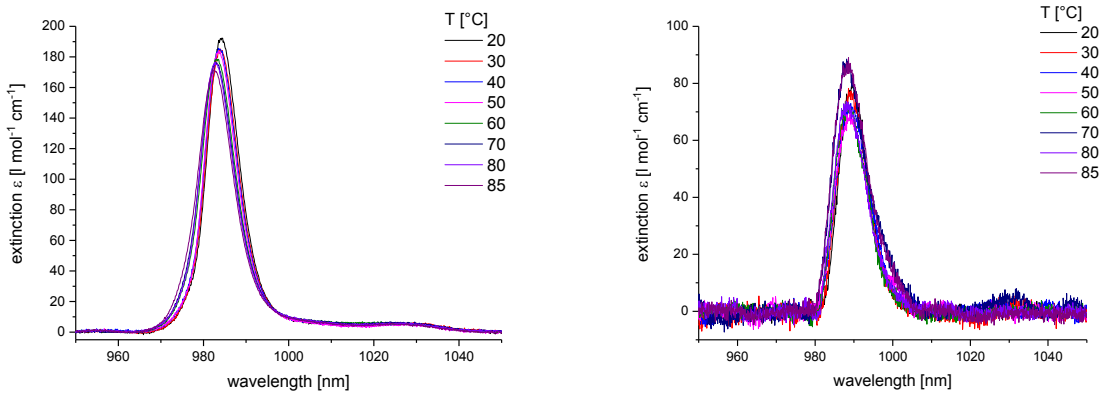

Figure S 1: Absorption spectra of the $NpO_2(Ac)$ (left) and $NpO_2(Ac)_2^-$ (right) complex at $I_m(NaClO_4)$ = 4.0 mol kg^{-1} as a function of the temperature.

Complex stoichiometry

Sample composition and speciation

Table S 2: Collection of the samples used for the determination of the complex stoichiometry.

sample	$m_{tot}(H_2O)$ [mg]	$[NpO_2^+]_{tot}$ [mol kg^{-1}]	$[Ac^-]_{tot}$ [mol kg^{-1}]	$[H^+]_{tot}$ [mol kg^{-1}]	I_m
1	1995.63	6.13E-04	0.00E+00	4.86E-05	4.01
2	1998.98	6.12E-04	1.68E-03	4.85E-05	4.01
3	2002.32	6.11E-04	3.35E-03	4.85E-05	4.01
4	2005.67	6.10E-04	5.02E-03	4.84E-05	4.01
5	2009.01	6.09E-04	6.68E-03	4.83E-05	4.01
6	2012.36	6.08E-04	8.34E-03	4.83E-05	4.01
7	2017.37	6.06E-04	1.08E-02	4.82E-05	4.01
8	2025.74	6.04E-04	1.49E-02	4.81E-05	4.01
9	2037.45	6.00E-04	2.06E-02	4.79E-05	4.01
10	2055.84	5.95E-04	2.94E-02	4.76E-05	4.01
11	2082.60	5.87E-04	4.19E-02	4.71E-05	4.01
12	2141.14	5.71E-04	6.82E-02	4.62E-05	4.01
13	2073.17	1.72E-04	1.17E-01	2.32E-05	4.14
14	2155.67	1.65E-04	2.67E-01	2.29E-05	4.13
15	2247.34	1.58E-04	4.20E-01	2.26E-05	4.13
16	2351.84	1.51E-04	5.81E-01	2.22E-05	4.12
17	2469.18	1.44E-04	7.45E-01	2.19E-05	4.12

Table S 3: Species distribution obtained by principle component analyses for the experimental conditions displayed in Table S 2 at T = 20, 30, 40, 50, 60, 70, 80, and 85 °C.

	20			30			40		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	1.00	0.00	0.00	1.00	0.00	0.00	1.00	0.00	0.00
2	0.94	0.06	0.00	0.95	0.05	0.00	0.95	0.05	0.00
3	0.93	0.07	0.00	0.92	0.08	0.00	0.91	0.09	0.00
4	0.90	0.10	0.00	0.90	0.10	0.00	0.89	0.11	0.00
5	0.87	0.13	0.00	0.85	0.15	0.00	0.85	0.15	0.00
6	0.85	0.15	0.00	0.81	0.19	0.00	0.82	0.18	0.00
7	0.81	0.19	0.00	0.79	0.21	0.00	0.77	0.23	0.00
8	0.74	0.26	0.00	0.72	0.28	0.00	0.70	0.30	0.00
9	0.68	0.32	0.00	0.67	0.33	0.00	0.64	0.36	0.00
10	0.61	0.39	0.00	0.59	0.41	0.00	0.57	0.43	0.00
11	0.53	0.47	0.00	0.51	0.49	0.00	0.48	0.52	0.00
12	0.39	0.61	0.00	0.36	0.64	0.00	0.35	0.65	0.00
13	0.23	0.60	0.17	0.20	0.61	0.20	0.19	0.64	0.17
14	0.09	0.54	0.36	0.09	0.51	0.40	0.07	0.53	0.40
15	0.05	0.46	0.49	0.03	0.44	0.54	0.06	0.44	0.51
16	0.03	0.34	0.63	0.01	0.35	0.64	0.02	0.35	0.63
17	0.02	0.34	0.64	0.01	0.30	0.69	0.01	0.33	0.66

	50			60			70		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	1.00	0.00	0.00	1.00	0.00	0.00	1.00	0.00	0.00
2	0.95	0.05	0.00	0.94	0.06	0.00	0.92	0.08	0.00
3	0.92	0.08	0.00	0.89	0.11	0.00	0.88	0.12	0.00
4	0.88	0.12	0.00	0.85	0.15	0.00	0.83	0.17	0.00
5	0.84	0.16	0.00	0.79	0.21	0.00	0.79	0.21	0.00
6	0.80	0.20	0.00	0.77	0.23	0.00	0.75	0.25	0.00
7	0.77	0.23	0.00	0.72	0.28	0.00	0.70	0.30	0.00
8	0.69	0.31	0.00	0.64	0.36	0.00	0.61	0.39	0.00
9	0.62	0.38	0.00	0.57	0.43	0.00	0.52	0.48	0.00
10	0.55	0.45	0.00	0.50	0.50	0.00	0.47	0.53	0.00
11	0.46	0.54	0.00	0.40	0.60	0.00	0.37	0.63	0.00
12	0.33	0.67	0.00	0.28	0.72	0.00	0.24	0.76	0.00
13	0.16	0.62	0.22	0.14	0.63	0.23	0.12	0.66	0.22
14	0.09	0.49	0.42	0.05	0.53	0.42	0.04	0.57	0.39
15	0.04	0.42	0.55	0.02	0.40	0.58	0.00	0.46	0.54
16	0.01	0.34	0.65	0.02	0.33	0.65	0.00	0.40	0.60
17	0.01	0.30	0.69	0.02	0.27	0.71	0.02	0.31	0.67

	80			85		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	1.00	0.00	0.00	1.00	0.00	0.00
2	0.90	0.10	0.00	0.93	0.07	0.00
3	0.88	0.13	0.00	0.87	0.13	0.00
4	0.83	0.17	0.00	0.82	0.18	0.00
5	0.77	0.23	0.00	0.78	0.22	0.00
6	0.74	0.26	0.00	0.74	0.26	0.00
7	0.69	0.31	0.00	0.69	0.31	0.00
8	0.60	0.40	0.00	0.63	0.37	0.00
9	0.52	0.48	0.00	0.54	0.46	0.00
10	0.47	0.53	0.00	0.46	0.54	0.00
11	0.37	0.63	0.00	0.35	0.65	0.00
12	0.25	0.75	0.00	0.21	0.79	0.00
13	0.10	0.67	0.23	0.08	0.69	0.23
14	0.03	0.53	0.43	0.03	0.54	0.43
15	0.02	0.42	0.56	0.01	0.45	0.54
16	0.01	0.29	0.70	0.00	0.36	0.64
17	0.00	0.30	0.70			

Calculation of the free ligand concentration $[Ac^-]_{eq}$

The concentration of deprotonated acetate $[Ac^-]_{eq}$ is calculated as performed in our studies on the complexation of Cm(III) with acetate and propionate.[1, 2] The complexation of Na^+ and acetate is assumed to be negligible and $[Ac^-]_{eq}$ is calculated using the Henderson-Hasselbalch equation (eqn. (a)):

$$[Ac^-]_{eq} = \frac{-[H^+]_{tot} + [Ac^-]_{tot} - K'_{HAc}(T) + \sqrt{\left([H^+]_{tot} - [Ac^-]_{tot} + K'_{HAc}(T)\right)^2 + 4 \cdot K'_{HAc}(T) \cdot [Ac^-]_{tot}}}{2} \quad \text{(a)}$$

$$[Ac^-]_{tot} = [Ac^-]_{eq} + [HAc]_{eq} \quad \text{(b)}$$

$$[H^+]_{tot} = [H^+]_{eq} + [HAc]_{eq} \quad \text{(c)}$$

$[H^+]_{tot}$ and $[Ac^-]_{tot}$ are the total concentrations of protons and acetate (eqn. (b)+(c)). $K'_{HAc}(T)$ is the conditional protonation constant of Ac^- at a given temperature and ionic strength. $K'_{HAc}(T)$ is calculated according to the SIT which is recommended in the NEA-TDB.[3] Within the SIT approach, ion-ion interaction coefficients are needed for calculating the activity coefficients of the reactants. Therefore, the following parameters given by the NEA-TDB:[3] $\varepsilon(Na^+, Ac^-) = 0.08 \pm 0.01$, $\varepsilon(H^+, ClO_4^-) = 0.14 \pm 0.02$, and $\varepsilon(H^+, Cl^-) = 0.12 \pm 0.01$ are used. Within the temperature range studied in the present work, it is appropriate to assume that these values are temperature independent. The temperature dependence of the dissociation of HAc is calculated using the given values of $\log K^0_{HAc}(25\ °C) = -4.757$ and $\Delta_r H^0_m = -0.41$ kJ mol^{-1} (both vales are given in the NIST database 46.6) and the integrated Van´t Hoff equation.[4]

Results of the slope analyses for the formation of $NpO_2(Ac)_n^{1-n}$

Table S 4: Results of the slope analyses according to equation (1) at $I_m(NaClO_4)$ = 4.0 mol kg^{-1} and various temperatures.

T [°C]	$NpO_2(Ac)$		$NpO_2(Ac)_2^-$	
	slope	R^2 (COD)	slope	R^2 (COD)
20	1.05 ± 0.02	0.9967	1.08 ± 0.08	0.9805
30	0.96 ± 0.03	0.9928	1.06 ± 0.01	0.9995
40	0.96 ± 0.01	0.9979	1.11 ± 0.05	0.9905
50	0.98 ± 0.02	0.9979	1.02 ± 0.01	0.9992
60	0.97 ± 0.01	0.9983	1.08 ± 0.03	0.9966
70	0.97 ± 0.02	0.9955	0.98 ± 0.04	0.9922
80	0.98 ± 0.02	0.9969	1.09 ± 0.10	0.9685
85	1.03 ± 0.03	0.9944	1.03 ± 0.03	0.9973

Ionic strength dependence

Sample composition and speciation for the $NaClO_4$ medium

Table S 5: Collection of the samples used to study the ionic strength dependence of the complexation reaction in $NaClO_4$ medium.

sample	$m_{tot}(H_2O)$ [mg]	$[NpO_2^+]_{tot}$ [mol kg^{-1}]	$[Ac^-]_{tot}$ [mol kg^{-1}]	$[H^+]_{tot}$ [mol kg^{-1}]	I_m
1	2016.71	2.49E-04	1.09E-01	2.16E-05	0.58
2	2043.98	2.46E-04	1.09E-01	2.15E-05	0.76
3	2072.44	2.42E-04	1.08E-01	2.14E-05	0.95
4	2103.26	2.39E-04	1.08E-01	2.13E-05	1.14
5	2137.65	2.35E-04	1.08E-01	2.12E-05	1.35
6	2174.41	2.31E-04	1.08E-01	2.11E-05	1.57
7	2214.72	2.27E-04	1.08E-01	2.10E-05	1.80
8	2259.78	2.22E-04	1.07E-01	2.09E-05	2.04
9	1980.85	1.80E-04	5.60E-01	1.81E-05	1.09
10	2041.25	1.74E-04	5.43E-01	1.80E-05	1.47
11	2101.65	1.69E-04	5.28E-01	1.79E-05	1.83
12	2162.06	1.65E-04	5.13E-01	1.79E-05	2.17
13	2222.46	1.60E-04	4.99E-01	1.78E-05	2.49

Table S 6: Species distribution obtained by principle component analyses for the experimental conditions displayed in Table S 5 at T = 20, 30, 40, 50, 60, 70, 80, and 85 °C.

	20			30			40		
sample	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$
1	0.48	0.52	0.00	0.45	0.55	0.00	0.43	0.57	0.00
2	0.47	0.53	0.00	0.47	0.53	0.00	0.44	0.56	0.00
3	0.47	0.53	0.00	0.46	0.54	0.00	0.44	0.56	0.00
4	0.48	0.52	0.00	0.47	0.53	0.00	0.44	0.56	0.00
5	0.49	0.51	0.00	0.45	0.55	0.00	0.43	0.57	0.00
6	0.48	0.52	0.00	0.45	0.55	0.00	0.42	0.58	0.00
7	0.47	0.53	0.00	0.43	0.57	0.00	0.41	0.59	0.00
8	0.45	0.55	0.00	0.41	0.59	0.00	0.39	0.61	0.00
9	0.06	0.54	0.40	0.05	0.52	0.44	0.04	0.52	0.44
10	0.06	0.53	0.41	0.05	0.51	0.44	0.04	0.51	0.45

11	0.06	0.51	0.43	0.04	0.49	0.47	0.04	0.50	0.47
12	0.06	0.50	0.44	0.04	0.48	0.48	0.04	0.48	0.48
13	0.05	0.50	0.45	0.04	0.46	0.49	0.03	0.47	0.50

	50			**60**			**70**		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	0.40	0.60	0.00	0.34	0.66	0.00	0.30	0.70	0.00
2	0.42	0.58	0.00	0.34	0.66	0.00	0.31	0.69	0.00
3	0.41	0.59	0.00	0.33	0.67	0.00	0.30	0.70	0.00
4	0.40	0.60	0.00	0.34	0.66	0.00	0.31	0.69	0.00
5	0.39	0.61	0.00	0.33	0.67	0.00	0.31	0.69	0.00
6	0.39	0.61	0.00	0.32	0.68	0.00	0.30	0.70	0.00
7	0.37	0.63	0.00	0.31	0.69	0.00	0.28	0.72	0.00
8	0.36	0.64	0.00	0.28	0.72	0.00	0.27	0.73	0.00
9	0.03	0.51	0.45	0.02	0.51	0.47	0.01	0.51	0.47
10	0.04	0.51	0.46	0.02	0.50	0.48	0.01	0.51	0.49
11	0.04	0.49	0.48	0.01	0.49	0.50	0.01	0.49	0.50
12	0.04	0.47	0.49	0.01	0.47	0.51	0.01	0.48	0.51
13	0.04	0.46	0.50	0.01	0.46	0.53	0.01	0.47	0.52

	80			**85**		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	0.28	0.72	0.00	0.23	0.77	0.00
2	0.29	0.71	0.00	0.25	0.75	0.00
3	0.29	0.71	0.00	0.24	0.76	0.00
4	0.30	0.70	0.00	0.24	0.76	0.00
5	0.29	0.71	0.00	0.23	0.77	0.00
6	0.28	0.72	0.00	0.23	0.77	0.00
7	0.27	0.73	0.00	0.22	0.78	0.00
8	0.25	0.75	0.00	0.21	0.79	0.00
9	0.01	0.51	0.48	0.00	0.51	0.49
10	0.01	0.50	0.49	0.00	0.50	0.50
11	0.01	0.49	0.50	0.00	0.49	0.51
12	0.01	0.47	0.52	0.00	0.48	0.52
13	0.01	0.46	0.53	0.00	0.46	0.54

Extrapolation of log K´(T) according to the SIT

The thermodynamic stability constants log K^0_n(T) are derived from eqn (4).

$$\log K'(T) - \Delta z^2 D(T) = log\ K^0(T) + \Delta\varepsilon(T) I_m \qquad (4)$$

Thus, plotting $\log K'(T) - \Delta z^2 D$ versus I_m yields log K^0(T) from the intercept of the ordinate.

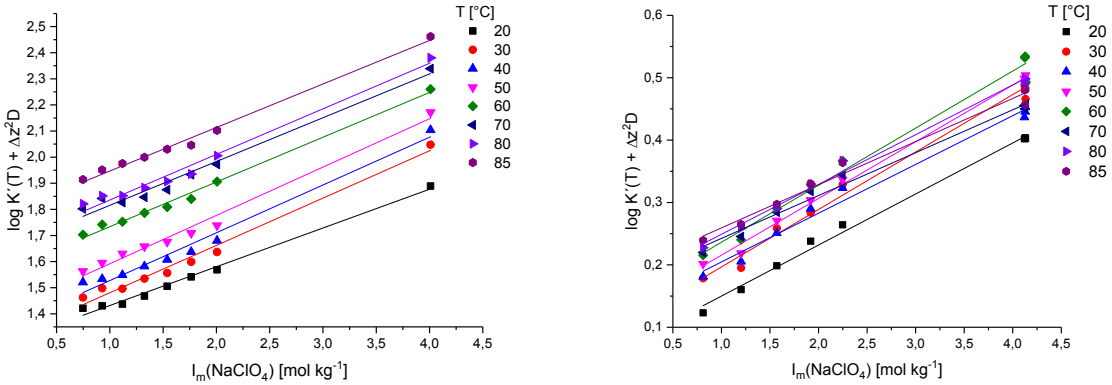

Figure S 2: Extrapolation of log $K`_n(T)$ to $I_m = 0$ according to the SIT at various temperatures in $NaClO_4$ medium. (left) $NpO_2(Ac)$, (right) $NpO_2(Ac)_2^-$

Sample composition and speciation for the NaCl medium

Table S 7: Collection of the samples used to study the ionic strength dependence of the complexation reaction in NaCl medium.

sample	$m_{tot}(H_2O)$ [mg]	$[NpO_2^+]_{tot}$ [mol kg^{-1}]	$[Ac^-]_{tot}$ [mol kg^{-1}]	$[H^+]_{tot}$ [mol kg^{-1}]	I_m
1	1782.00	2.31E-04	5.17E-02	3.40E-05	0.53
2	1782.00	2.31E-04	5.17E-02	3.40E-05	1.10
3	1782.00	2.31E-04	5.17E-02	3.40E-05	1.62
4	1782.00	2.31E-04	5.17E-02	3.40E-05	2.17
5	1782.00	2.31E-04	5.17E-02	3.40E-05	2.70
6	1782.00	2.31E-04	5.17E-02	3.40E-05	3.25
7	1782.00	2.31E-04	5.17E-02	3.40E-05	3.78
8	1930.00	1.79E-04	5.69E-01	2.34E-05	1.45
9	1930.00	1.79E-04	5.69E-01	2.34E-05	2.31
10	1930.00	1.79E-04	5.69E-01	2.34E-05	3.16
11	1930.00	1.79E-04	5.69E-01	2.34E-05	4.07
12	1930.00	1.79E-04	5.69E-01	2.34E-05	4.94

Table S 8: Species distribution obtained by principle component analyses for the experimental conditions displayed in Table S 7 at T = 20, 30, 40, 50, 60, 70, 80, and 85 °C.

	20			**30**			**40**		
sample	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$	NpO_2^+	$NpO_2(Ac)$	$NpO_2(Ac)_2^-$
1	0.78	0.55	0.00	0.58	0.42	0.00	0.56	0.44	0.00
2	0.80	0.50	0.00	0.60	0.40	0.00	0.57	0.43	0.00
3	0.76	0.52	0.00	0.57	0.43	0.00	0.55	0.45	0.00
4	0.74	0.52	0.00	0.56	0.44	0.00	0.53	0.47	0.00
5	0.70	0.57	0.00	0.53	0.47	0.00	0.50	0.50	0.00
6	0.66	0.60	0.00	0.47	0.53	0.00	0.45	0.55	0.00
7	0.60	0.70	0.00	0.43	0.57	0.00	0.39	0.61	0.00
8	0.06	0.53	0.41	0.04	0.52	0.44	0.05	0.50	0.45
9	0.05	0.50	0.46	0.03	0.48	0.49	0.03	0.47	0.50
10	0.03	0.44	0.52	0.02	0.41	0.57	0.02	0.43	0.55

11	0.03	0.39	0.58	0.01	0.35	0.64	0.00	0.36	0.64
12	0.00	0.36	0.64	0.00	0.30	0.70	0.00	0.31	0.69

	50			**60**			**70**		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	0.52	0.48	0.00	0.48	0.52	0.00	0.45	0.55	0.00
2	0.55	0.45	0.00	0.47	0.53	0.00	0.44	0.56	0.00
3	0.51	0.49	0.00	0.44	0.56	0.00	0.41	0.59	0.00
4	0.50	0.50	0.00	0.42	0.58	0.00	0.37	0.63	0.00
5	0.45	0.55	0.00	0.39	0.61	0.00	0.35	0.65	0.00
6	0.43	0.57	0.00	0.35	0.65	0.00	0.32	0.68	0.00
7	0.37	0.63	0.00	0.31	0.69	0.00	0.26	0.74	0.00
8	0.04	0.49	0.47	0.01	0.49	0.50	0.01	0.48	0.51
9	0.03	0.45	0.52	0.01	0.46	0.54	0.00	0.46	0.54
10	0.02	0.38	0.60	0.00	0.41	0.59	0.00	0.39	0.61
11	-	-	-	0.00	0.32	0.68	0.00	0.34	0.66
12	0.01	0.27	0.72	0.00	0.29	0.71	0.00	0.29	0.71

	80			**85**		
sample	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**	**NpO_2^+**	**$NpO_2(Ac)$**	**$NpO_2(Ac)_2^-$**
1	0.44	0.56	0.00	0.38	0.62	0.00
2	0.44	0.56	0.00	0.37	0.63	0.00
3	0.40	0.60	0.00	0.35	0.65	0.00
4	0.37	0.63	0.00	0.33	0.67	0.00
5	0.34	0.66	0.00	0.30	0.70	0.00
6	0.30	0.70	0.00	0.25	0.75	0.00
7	0.27	0.73	0.00	0.21	0.79	0.00
8	0.01	0.47	0.52	0.00	0.47	0.53
9	0.00	0.43	0.57	0.00	0.44	0.56
10	0.00	0.39	0.61	0.00	0.38	0.62
11	0.00	0.34	0.66	0.00	0.35	0.65
12	0.00	0.28	0.72	0.00	0.29	0.71

Extrapolation of log K´(T) according to the SIT

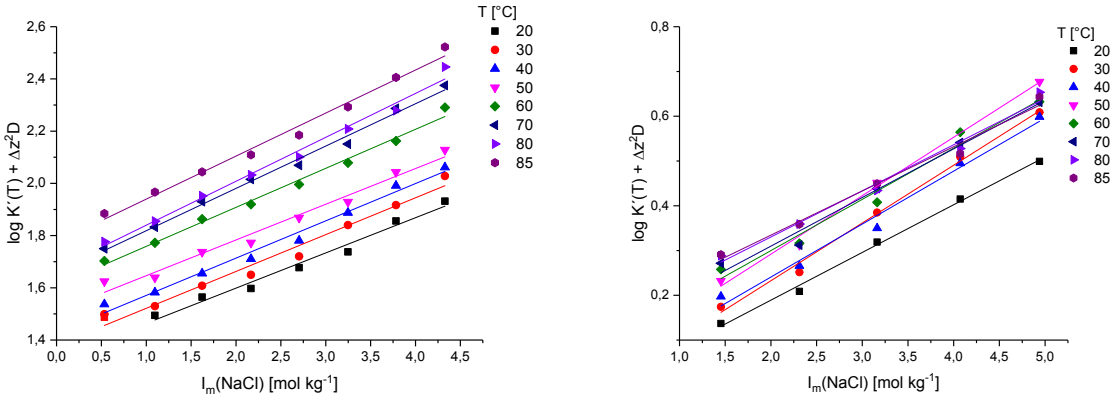

Figure S 3: Extrapolation of log K`$_n$(T) to I_m = 0 according to the SIT at various temperatures in NaCl medium. (left) $NpO_2(Ac)$, (right) $NpO_2(Ac)_2^-$

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