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Structure of Potassium Triaqua(ethylenediaminetetraacetato)neodymate(III) Pentahydrate

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Abstract. $K[Nd(C_{10}H_{12}N_2O_8)(H_2O)_3].5H_2O$, M_r = 615·67, orthorhombic, *Fdd*2, $a = 19{\cdot}944\,(9)$, $b = 36{\cdot}035\,(9)$, $c = 12{\cdot}276\,(8)$ Å, $V = 8823\,(7)$ Å^3, $Z = 16$, $D_x = 1{\cdot}854$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 26{\cdot}24$ cm^{-1}, $F(000) = 4944$, $T = 293$ K, $R = 0{\cdot}029$ for 3187 observed reflections. Distances from Nd to the coordinating atoms fit well in the pattern of decreasing distances in the series La^{3+}–Lu^{3+} as a result of lanthanide contraction: Nd—O(edta) 2·411 (4)–2·472 (2), Nd—O(water) 2·520 (4)–2·574 (4), Nd—N 2·709 (4) and 2·715 (4) Å.

Introduction. Crystal structures of several lanthanide edta complexes have been reported: $K[La(edta)(H_2O)_3].5H_2O$ (Hoard, Lee & Lind, 1965), $Na[Ln(edta)(H_2O)_3].5H_2O$, Ln = Tb (Lee, 1967), Dy (Nassimbeni, Wright, van Niekerk & McCallum, 1979), Pr, Gd, Sm (Templeton, Templeton, Zalkin & Ruben, 1982), Sm (Engel, Takusagawa & Koetzle, 1984), Ho (Templeton, Templeton & Zalkin, 1985), and $Cs[Yb(edta)(H_2O)_2].3H_2O$ (Nassimbeni, Wright, van Niekerk & McCallum, 1979). It was suggested that there should be a change from a nine-coordinated to an eight-coordinated complex in the lanthanide series La^{3+} to Lu^{3+} as a result of the decreasing ionic radii. Only the Yb complex has so far been found to be eight-coordinated. All nine-coordinated complexes have very similar structures and most of them crystallize in the orthorhombic space group *Fdd*2. Ho and Dy complexes have been found to crystallize in the monoclinic space group *Fd*11 which involves ordering of the occupancy of water molecule sites that were described as disordered or with high temperature factors in the other structures. The crystal structure of the title compound was determined to obtain more information on the Nd–ligand distances.

Experimental. Purple crystals of $K[Nd(edta)(H_2O)_3]$.-$5H_2O$ were obtained from an aqueous solution of $K_2Nd(OH)(edta).4H_2O$ (Djordjević & Vuletić, 1980) in an attempt to grow single crystals of the hydroxo complex. Single crystal (longest dimensions along the *a*, *b* and *c* crystallographic axes 0·37 × 0·62 × 0·55 mm) was used for X-ray analysis. Intensities were measured on a Philips PW 1100 diffractometer (Mo $K\alpha$ radiation, graphite monochromator) in the range $4 < 2\theta < 60°$ ($0 \le h \le 28$, $0 \le k \le 50$, $0 \le l \le 17$). Unit-cell parameters were determined by least-squares refinement of 16 reflections, $12 < 2\theta < 16°$. Intensity data were collected in the $\theta/2\theta$ mode, scan speed 0·04° s^{-1}, scan width 1·2°. The intensity variation of standard reflections (391, 0,12,0, 602) measured every 2 h showed

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no significant change. Three reflections suffering from extinction were omitted. Out of 3257 measured, 3187 unique data with $I > 3\sigma(I)$ were used in the refinement. Intensities were corrected for the Lp effects but not for absorption. Coordinates of all atoms, except those of the crystal water molecules, were taken from the Pr complex (Templeton, Templeton, Zalkin & Ruben, 1982) as initial values in the full-matrix least-squares refinement on F. Crystal water oxygen atoms were located in a difference Fourier map. Two positions were found for *W*7 and were given a site occupation factor of 0·5. Positions of H atoms on the edta ligand were calculated geometrically (riding model, C—H 1·08 Å). Refinement with anisotropic temperature factors for non-H atoms (except for isotropic refinement of *W*7*A* and *W*7*B*) and single overall isotropic temperature factor for H atoms (270 parameters) resulted in final $R = 0{\cdot}029$, $wR = 0{\cdot}030$, $w = 1/[\sigma^2(F) + 0{\cdot}002269F^2]$. Refinement with the signs of if'' reversed gave $R = 0{\cdot}031$ and $wR = 0{\cdot}034$. The value of $w\mathscr{R} = wR^-/wR^+ = 1{\cdot}13$ confirms the correct assignment of absolute configuration (Rogers, 1981). Maximum shift/e.s.d. in the final cycle was 0·34. Maximum and minimum $\Delta\rho$ values in the final difference Fourier map were 1·10 (0·9 Å from Nd) and $-2{\cdot}04$ e Å^{-3}. Scattering factors and corrections for anomalous dispersion were from *International Tables for X-ray Crystallography* (1974). Calculations were performed on a UNIVAC 1110 computer using *SHELX*76 (Sheldrick, 1976).

Discussion. An *ORTEP* drawing (Johnson, 1965) of the complex anion is shown in Fig. 1. The atom-numbering scheme is the same as that used by Engel, Takusagawa & Koetzle (1984). Atomic parameters are listed in Table 1.* Selected interatomic distances and angles are given in Table 2.

The crystal structure is very similar to those of other nine-coordinated edta complexes. The Nd^{3+} ion is surrounded by four oxygen atoms (O1–O4) and two nitrogen atoms (N1, N2) from the edta group and three water oxygen atoms (*W*1–*W*3). Because of lanthanide-ion contraction these distances are on the average 0·009 Å shorter than in the Pr complex, and 0·028 Å longer than in the Sm complex (Templeton, Templeton, Zalkin & Ruben, 1982). Nd is displaced 0·568 (10) Å from the mean plane through the four O atoms, which is 0·043 Å more than in the Sm and 0·003 Å less than in the Pr complex.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances within the edta moiety and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44501 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2(U_{12}aba^*b^*\cos\gamma + U_{13}aca^*c^*\cos\beta + U_{23}bcb^*c^*\cos\alpha)].$$

	x	y	z	U_{eq} or U†
Nd	0·08726 (1)	0·15428 (1)	0	0·01844 (4)
K	0·13298 (9)	0·13195 (4)	0·33409 (12)	0·0565 (3)
O1	−0·0209 (2)	0·1709 (1)	0·0728 (3)	0·0327 (7)
O2	0·1019 (2)	0·1829 (1)	0·1790 (3)	0·0318 (7)
O3	0·2077 (1)	0·1678 (1)	0·0263 (2)	0·0284 (7)
O4	0·0211 (2)	0·1653 (1)	−0·1618 (3)	0·0258 (6)
O5	−0·0910 (2)	0·2116 (2)	0·1488 (6)	0·0622 (13)
O6	0·1084 (4)	0·2311 (1)	0·2899 (4)	0·0778 (19)
O7	0·3111 (2)	0·1744 (1)	−0·0377 (3)	0·0410 (9)
O8	0·0090 (2)	0·1757 (1)	−0·3405 (3)	0·0379 (8)
N1	0·0630 (2)	0·2284 (1)	0·0061 (4)	0·0292 (6)
N2	0·1506 (2)	0·1921 (1)	−0·1606 (3)	0·0252 (6)
C1	0·0845 (2)	0·2467 (1)	−0·0956 (5)	0·0308 (10)
C2	0·1517 (2)	0·2326 (1)	−0·1329 (4)	0·0317 (9)
C3	−0·0096 (3)	0·2341 (1)	0·0246 (5)	0·0412 (11)
C4	−0·0419 (2)	0·2033 (1)	0·0878 (4)	0·0318 (8)
C5	0·1008 (4)	0·2433 (2)	0·1011 (5)	0·0434 (13)
C6	0·1026 (3)	0·2171 (1)	0·1973 (4)	0·0357 (10)
C7	0·2211 (2)	0·1798 (1)	−0·1648 (4)	0·0289 (9)
C8	0·2493 (2)	0·1736 (1)	−0·0498 (4)	0·0261 (8)
C9	0·1172 (2)	0·1853 (2)	−0·2663 (3)	0·0319 (10)
C10	0·0427 (2)	0·1751 (1)	−0·2555 (4)	0·0258 (7)
*W*1	0·1385 (2)	0·1084 (1)	−0·1307 (3)	0·0342 (7)
*W*2	0·1373 (2)	0·1036 (1)	0·1161 (3)	0·0317 (7)
*W*3	0·0086 (2)	0·0980 (1)	−0·0185 (3)	0·0298 (7)
*W*4	0·0024 (6)	0·1063 (4)	0·2841 (18)	0·187 (6)
*W*5	0·0658 (3)	0·0400 (1)	0·0983 (4)	0·049 (1)
*W*6	0·1423 (5)	0·0958 (1)	0·5414 (7)	0·078 (2)
*W*7*A*	0·1107 (12)	0·1862 (17)	0·4654 (21)	0·124 (6)†
*W*7*B*	0·0480 (9)	0·1747 (5)	0·4441 (17)	0·101 (5)†
*W*8	0·0327 (6)	0·0342 (4)	0·3240 (9)	0·128 (3)

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

Coordination polyhedron of the Nd and the K atom			
Nd–O1	2·411 (4)	K–O1^{i}	2·706 (4)
Nd–O2	2·445 (4)	K–O2	2·717 (4)
Nd–O3	2·472 (2)	K–*W*2	2·866 (4)
Nd–O4	2·417 (4)	K–*W*3^{i}	2·896 (4)
Nd–N1	2·715 (4)	K–*W*4	2·831 (13)
Nd–N2	2·709 (4)	K–*W*6	2·865 (8)
Nd–*W*1	2·520 (4)	K–*W*7*A*	2·57 (3)
Nd–*W*2	2·522 (4)	K–*W*7*B*	2·66 (2)
Nd–*W*3	2·574 (4)		
Mean distances in the edta ligand			
⟨C–C⟩	1·52 (2)	⟨C–C–N⟩	113 (1)
⟨C–O(–Nd)⟩	1·26 (1)	⟨C–N–C⟩	110 (2)
⟨C–O⟩	1·25 (1)	⟨C–C–O⟩	117·7 (6)
⟨C–N⟩	1·48 (1)	⟨O–C–O⟩	124·6 (6)
Hydrogen bonds			
*W*1–O7^{ii}	2·738 (5)	*W*5–O6^{iii}	2·70 (1)
*W*1–O8^{i}	2·744 (6)	*W*5–*W*8	2·86 (1)
*W*2–O4^{i}	2·775 (6)	*W*6–O5^{i}	2·73 (1)
*W*2–*W*5	2·708 (6)	*W*6–O7^{iv}	2·75 (1)
*W*3–O3^{ii}	2·775 (5)	*W*7*A*–O6	2·70 (3)
*W*3–*W*5	2·779 (6)	*W*7*B*–O8^{v}	2·76 (2)
*W*4–*W*6^{ii}	2·80 (2)	*W*8–O5^{i}	2·69 (1)
*W*4–*W*8	2·71 (2)	*W*8–*W*8^{vi}	2·79 (2)

Symmetry code: (i) $\frac{1}{4} + x, \frac{1}{4} - y, \frac{1}{4} + z$; (ii) $x - \frac{1}{4}, \frac{1}{4} - y, z - \frac{1}{4}$; (iii) $\frac{1}{4} - x, y - \frac{1}{4}, z - \frac{1}{4}$; (iv) $x - \frac{1}{4}, \frac{1}{4} - y, \frac{3}{4} + z$; (v) $x, y, 1 + z$; (vi) $-x, -y, z$.

Standard deviations of mean values were calculated according to the expression $\sigma = [\sum(d - \bar{d})^2/(N - 1)]^{1/2}$.

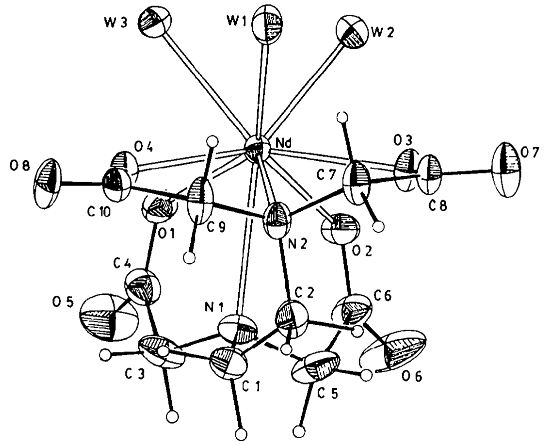

Fig. 1. *ORTEP* drawing of the complex anion with the atom-numbering scheme.

Crystal water oxygen atoms *W*4 and *W*8 were not found to have disordered positions as in the Sm salt solved by neutron diffraction (Engel *et al.*, 1984) nor does *W*8 have such a high temperature factor as in the other structures. Instead, disordered positions of *W*7 were found (*W*7*A* and *W*7*B*). The same hydrogen-bonding network as in the neutron diffraction study of the Sm complex was found for the Nd salt, except for the bond involving *W*7*B*. The differences in the positions of the crystal water molecules are influenced by the larger K^+ ion, which is surrounded by seven O atoms at distances ranging from 2·57 (3) or 2·66 (3) to 2·895 (4) Å. Bond distances and angles in the edta moiety are normal.

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Structure of *trans*-Dichloro-*cis*-dichlorobis(1-propylimidazole)platinum(IV)

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Abstract. $[PtCl_4(C_6H_{10}N_2)_2]$, $M_r = 557·2$, monoclinic, $P2_1/a$, $a = 10·015$ (4), $b = 16·134$ (6), $c = 12·175$ (5) Å, $\beta = 108·12$ (5)°, $V = 1869·7$ (9) Å^3, $Z = 4$, $D_m = 2·00$ (5), $D_x = 1·98$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 8·46$ mm^{-1}, $F(000) = 1064$, $T = 294$ (2) K, $R = 0·038$ for 2663 unique observed reflections. The complex consists of monomeric $PtCl_4$-(1-propylimidazole)$_2$ units. The coordination geometry is octahedral. The two 1-propylimidazole ligands are *cis*-coordinated to Pt, the Pt—N distances are 2·061 (9) and 2·045 (8) Å. The Pt—Cl distances ranging from 2·310 (3) to 2·322 (3) Å are normal for complexes of Pt^{IV}.

Introduction. In contrast to neutral Pt^{II} complexes, the Pt^{IV} analogues are rather poorly investigated though their biological and therapeutical properties seem to be equally interesting. Therefore we now present the crystal structure of tetrachlorobis(1-propylimidazole)-platinum(IV) which we obtained by a modification of the method of Braddock, Connors, Jones, Khokhar, Melzack & Tobe (1975).

Experimental. 0·48 g (0·001 mol) of *cis*-dichlorobis-(1-propylimidazole)platinum(II) was suspended in 20 ml of 2*M* HCl and 4 ml of 30% H_2O_2. The mixture was boiled until a yellow solution was obtained, then it

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