

Structure of a Neodymium Diglycine Complex, $[\text{Nd}(\text{Gly-Gly})_2(\text{H}_2\text{O})_2](\text{ClO}_4)_3 \cdot 2\text{H}_2\text{O}$

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Abstract. Diaquabis(glycylglycine)neodymium(III) perchlorate dihydrate, $\text{C}_8\text{H}_{20}\text{N}_4\text{NdO}_8 \cdot 3\text{ClO}_4 \cdot 2\text{H}_2\text{O}$, $M_r = 778.89$, monoclinic, $P2_1/n$, $a = 12.082$ (1), $b = 8.965$ (1), $c = 22.888$ (2) Å, $\beta = 91.25$ (1)°, $V = 2478.5$ (3) Å³, $Z = 4$, $D_m = 2.09$, $D_x = 2.0874$ (3) Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.529$ mm⁻¹, $F(000) = 1548$, $R = 0.036$ for 8594 reflections collected at room temperature. The Nd^{3+} cations are nine-coordinated and occur pairwise, bridged by four carboxyl groups. The coordination around each cation is completed by two carbonyl O atoms and two crystal water molecules. One of the two peptide ligands of the asymmetric unit is coordinated to a pair of neodymium ions whereas the other is bonded to three different Nd^{3+} ions, thus interlinking the cation pairs. Accordingly, endless polymeric chains are formed in the crystallographic **b** direction. The perchlorate counterions together with two non-coordinating water molecules fill up the space between the polymeric chains.

Introduction. Rare-earth coordination chemistry often shows pronounced similarity to that of Ca^{2+} (e.g. with respect to size, preference for O donors, variability in coordination number, lack of strong directionality) (Brittain, Richardson & Martin, 1976). Accordingly, studies of lanthanoid complexes with amino acids or peptides may give valuable information about structural characteristics of Ca^{2+} binding sites in complicated biological systems (Martin & Richardson, 1979; De Jersey, Jeffers-Morley & Martin, 1981). The present investigation is part of a research programme concerning the structural features of interactions between trivalent lanthanoid cations and amino acids or peptides in the solid state.

Experimental. The title complex was prepared by dissolving $\text{Nd}(\text{ClO}_4)_3 \cdot 6\text{H}_2\text{O}$ and glycylglycine in

water, as described earlier for related lanthanide-amino acid complexes (Csöreg, Kierkegaard, Legendziewicz & Huskowska, 1987, 1989; Csöreg, Czugler, Kierkegaard, Legendziewicz & Huskowska, 1989). Crystals were grown by slow evaporation at room temperature. The crystal density was measured by the flotation method in a mixture of bromoform and 1,2-dibromoethane.

Intensity data were obtained on a Siemens Stoe/AED2 diffractometer equipped with graphite-monochromated Mo $K\alpha$ radiation ($\theta_{\text{max}} = 40^\circ$, $0 < h < 21$, $0 < k < 16$, $-41 < l < 41$), using the ω - 2θ scan technique. The intensities of a total of 16874 reflections were collected from a violet irregularly shaped single crystal with approximate dimensions $0.30 \times 0.34 \times 0.32$ mm, sealed in a glass capillary. Five reference reflections, measured at intervals of approximately 60 min, showed a decrease in intensity of about 20% during the period of data collection (~11 d). Data reduction included corrections for background, Lorentz, polarization, decay and absorption effects. The empirical absorption corrections were based on ψ scans of five reflections with $81.4 < \chi < 85.5^\circ$ and $18.3 < 2\theta < 29.2^\circ$. The transmission factors varied between 0.45 and 0.55. Of the collected 16874 reflections, 13756 were unique non-zero observations with $R_{\text{int}} = 0.019$. In the refinement of the unit-cell parameters the θ values of 50 well centred reflections with $29 < 2\theta < 49^\circ$ were used.

The Nd^{3+} position, derived from a Patterson synthesis, served as a starting point for solution of the structure by conventional difference electron density calculations (Sheldrick, 1976). The model was refined by minimization of the $\sum w\Delta F^2$ function, using the full-matrix least-squares method (Sheldrick, 1976). The perchlorate anions showed rotational disorder, which could be partly resolved into major disorder sites. Eight disorder sites were refined for the four O atoms of Cl(1); twelve and seven O sites were included in the structural model of the perchlorate groups of Cl(2) and Cl(3), respectively (Table 1). The

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disorder sites were refined together with either their displacement parameters or their site-occupancy factors in consecutive calculations, without any further constraints. The sum of the site-occupancy factors, as refined in the calculation previous to the final refinement (*cf.* Table 1), are 4.01 for the group of Cl(1), and 4.19 and 3.94 for the two other ClO₄⁻ anions. Besides the non-H atomic positions and the O disorder sites, the O- and N-bonded H-atom sites were also located from $\Delta\rho$ maps, and were kept riding on their parent atoms during the subsequent calculations. The C-bonded H atoms of the glycylglycine moieties, however, were assigned geometrically predicted positions with C—H = 1.00 Å, which were recalculated after each cycle of the refinement. In the final structural model the non-H atoms with full site occupancy were allowed to vibrate anisotropically, whereas the O disorder sites were treated isotropically. Isotropic displacement parameters were refined also for the H-atom positions. Nevertheless, for satisfactory convergence the common U_{iso} parameter of the hydrate H atoms had to be fixed in the final calculation, where refinement of 346 parameters converged to $R = 0.036$ and $wR = 0.053$ for 8594 reflections with $I > 5\sigma(I)$. The maximum Δ/σ ratio became less than 0.56. Weights of the structure factors were calculated as $[\sigma^2(F) + 0.00025F^2]^{-1}$ and the scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The wR_{tot} value, calculated for the refined model using all the 13756 unique non-zero reflections, became 0.059. The maximum and minimum values of $\Delta\rho$ in the final difference Fourier map were 0.43 and $-0.42 \text{ e } \text{Å}^{-3}$, respectively. The refined fractional atomic coordinates of the non-H atoms are listed in Table 1.* E.s.d.'s of the perchlorate disorder sites are taken from the next-to-last refinement calculation.

Discussion. As seen in Figs. 1 and 2, the Nd³⁺ ion is nine-coordinated. The Nd—O coordination bond lengths vary from 2.397 (3) to 2.751 (4) Å (Table 2) with an arithmetic mean value of 2.50 Å, in

* Intramolecular bond distances and bond angles within the glycylglycine moieties involving the non-hydrogen atoms (Tables 3 and 4), fractional atomic coordinates and isotropic displacement parameters of the H atoms (Table 5), bond lengths and bond angles involving the H atoms (Tables 6 and 7), intermolecular contact distances less than 3.3 Å and geometry of possible hydrogen bonds (Table 8), anisotropic displacement parameters of the non-hydrogen atoms with full site occupancy (Table 9), a stereo packing diagram of the crystal structure (Fig. 3) and a list of the observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54920 (63 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0012]

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters of the non-H atoms with full site occupancy, and fractional atomic coordinates, site occupancy factors and isotropic displacement parameters for the observed disorder sites of the perchlorate O atoms; e.s.d.'s are given in parentheses*

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	S.o.f.	$U_{\text{eq}}/U_{\text{iso}}(\text{Å}^2)$
Nd	-0.00210 (1)	0.09932 (2)	0.07811 (1)		0.0228 (1)
O(W1)	-0.0536 (3)	0.0184 (3)	0.1802 (1)		0.041 (1)
O(W2)	0.1677 (3)	0.0612 (4)	0.1398 (2)		0.056 (1)
O(1)	0.0549 (3)	-0.2758 (3)	-0.0010 (1)		0.033 (1)
O(2)	0.0282 (3)	-0.1730 (3)	0.0871 (1)		0.034 (1)
Cl(1)	0.0532 (3)	-0.2785 (3)	0.0538 (2)		0.027 (1)
Cl(2)	0.0868 (4)	-0.4264 (4)	0.0804 (2)		0.039 (1)
N(3)	0.0667 (3)	-0.4391 (3)	0.1426 (2)		0.034 (1)
C(4)	0.0347 (3)	-0.5652 (4)	0.1656 (2)		0.027 (1)
O(4)	0.0132 (3)	-0.6788 (3)	0.1361 (1)		0.038 (1)
C(5)	0.0221 (4)	-0.5732 (4)	0.2312 (2)		0.040 (1)
N(6)	0.0446 (3)	-0.7261 (4)	0.2514 (2)		0.041 (1)
O(1')	0.1805 (3)	0.2375 (4)	0.0330 (2)		0.052 (1)
O(2')	0.1177 (2)	0.0257 (3)	-0.0030 (1)		0.029 (1)
C(1')	0.1849 (3)	0.1369 (4)	-0.0041 (2)		0.033 (1)
C(2')	0.2626 (4)	0.1472 (5)	-0.0543 (3)		0.049 (2)
N(3')	0.3302 (3)	0.0157 (4)	-0.0632 (2)		0.043 (1)
C(4')	0.2970 (4)	-0.1129 (5)	-0.0861 (2)		0.038 (1)
O(4')	0.1991 (3)	-0.1459 (4)	-0.0987 (2)		0.041 (1)
C(5')	0.3895 (4)	-0.2226 (7)	-0.0963 (3)		0.061 (2)
N(6')	0.3465 (3)	-0.3590 (5)	-0.1245 (2)		0.058 (2)
Cl(1)	-0.2536 (1)	0.2171 (2)	0.2823 (1)		0.0491 (3)
O(11a)	-0.2232 (5)	0.3630 (6)	0.3044 (3)	0.86 (1)	0.073 (2)
O(12a)	-0.1655 (8)	0.1239 (10)	0.3017 (4)	0.71 (1)	0.103 (3)
O(12b)	-0.2396 (19)	0.0860 (22)	0.3104 (10)	0.23 (1)	0.078 (6)
O(13a)	-0.3523 (9)	0.1844 (13)	0.3089 (5)	0.57 (2)	0.098 (3)
O(13b)	-0.3740 (10)	0.2243 (15)	0.2696 (6)	0.36 (1)	0.078 (4)
O(13c)	-0.3180 (17)	0.1278 (23)	0.3264 (9)	0.18 (1)	0.048 (4)
O(14a)	-0.2151 (7)	0.2204 (9)	0.2239 (4)	0.65 (1)	0.083 (2)
O(14b)	-0.2879 (11)	0.1880 (15)	0.2232 (6)	0.45 (2)	0.103 (4)
Cl(2)	0.1379 (1)	0.8473 (2)	0.2883 (1)		0.0498 (3)
O(21a)	0.0576 (7)	0.9512 (10)	0.3062 (4)	0.49 (1)	0.052 (2)
O(21b)	0.0260 (23)	0.8797 (30)	0.2939 (12)	0.20 (1)	0.088 (8)
O(21c)	0.1089 (23)	0.9991 (30)	0.3023 (11)	0.21 (1)	0.082 (7)
O(21d)	0.0842 (45)	0.8921 (53)	0.3382 (23)	0.13 (2)	0.112 (15)
O(22a)	0.1138 (7)	0.7100 (9)	0.3237 (4)	0.63 (1)	0.087 (2)
O(23a)	0.1295 (9)	0.8113 (11)	0.2268 (5)	0.44 (3)	0.062 (3)
O(23b)	0.0883 (11)	0.7934 (14)	0.2341 (5)	0.37 (3)	0.058 (3)
O(23c)	0.1879 (18)	0.7662 (25)	0.2472 (10)	0.20 (1)	0.069 (6)
O(24a)	0.2480 (10)	0.8865 (13)	0.2929 (5)	0.56 (1)	0.105 (3)
O(24b)	0.1897 (13)	1.0130 (18)	0.2751 (7)	0.32 (1)	0.086 (4)
O(24c)	0.1873 (18)	0.8119 (25)	0.3431 (9)	0.46 (2)	0.176 (7)
O(24d)	0.2425 (25)	0.7781 (35)	0.3014 (14)	0.18 (1)	0.081 (8)
Cl(3)	0.1051 (2)	0.3148 (2)	0.4096 (1)		0.0705 (5)
O(31a)	0.0866 (7)	0.4632 (10)	0.4314 (4)	0.74 (1)	0.107 (3)
O(31b)	0.0519 (13)	0.3717 (18)	0.4579 (7)	0.31 (1)	0.076 (4)
O(32a)	0.1598 (8)	0.2259 (11)	0.4539 (4)	0.79 (2)	0.131 (3)
O(32b)	0.2132 (23)	0.2824 (32)	0.4243 (12)	0.18 (1)	0.087 (8)
O(33a)	0.0125 (7)	0.2358 (9)	0.3858 (4)	0.89 (1)	0.127 (3)
O(34a)	0.1839 (6)	0.3139 (9)	0.3632 (3)	0.75 (1)	0.094 (2)
O(34b)	0.0888 (27)	0.4208 (33)	0.3652 (14)	0.28 (2)	0.153 (11)
O(W3)	0.7950 (5)	0.4914 (5)	0.0478 (2)		0.086 (2)
O(W4)	0.6031 (13)	0.5833 (12)	-0.0479 (5)		0.264 (9)

agreement with the earlier observations in related Nd complexes (Legendziewicz, Huskowska, Waškowska & Argay, 1984; Głowiak, Legendziewicz, Dao & Huskowska, 1987). The cations occur pairwise, bridged by two bidentate and two tridentate carboxyl groups. The coordination around each Nd³⁺ ion is completed by four more O atoms, two

carbonyl O atoms and two water O atoms in the present case. Similar structural motifs have been observed *e.g.* in Nd₂(Gly)₆(ClO₄)₆·9H₂O (Legendziewicz, Huskowska, Waškowska & Argay, 1984), in lanthanide glutamate perchlorate hydrates with Ln³⁺ = Pr³⁺, Ho³⁺ and Dy³⁺ (Csöreg, Kierkegaard, Legendziewicz & Huskowska, 1987; Csöreg, Czugler, Kierkegaard, Legendziewicz & Huskowska, 1989), and in Ln(Gly)₃(H₂O)₃(ClO₄)₃ with holmium as cation (Legendziewicz, Huskowska, Argay &

Table 2. *Geometry of the coordination around the Nd^{III} ion; Nd—O distances (Å) and O—Nd—O angles (°) with e.s.d.'s in parentheses*

Nd—O(W1)	2.538 (3)	Nd—O(W2)	2.489 (4)
Nd—O(2)	2.477 (3)	Nd—O(1')	2.751 (4)
Nd—O(2')	2.469 (3)	Nd—O(1)	2.444 (3)
Nd—O(4) ^a	2.397 (3)	Nd—O(2) ^a	2.461 (3)
Nd—O(4') ^a	2.472 (3)		
O(1')—Nd—O(2')	49.4 (1)	O(2)—Nd—O(2')	73.2 (1)
O(2)—Nd—O(1')	110.9 (1)	O(W2)—Nd—O(2')	84.3 (1)
O(W2)—Nd—O(1')	67.5 (1)	O(W2)—Nd—O(2)	72.5 (1)
O(W1)—Nd—O(2')	140.9 (1)	O(W1)—Nd—O(1')	133.6 (1)
O(W1)—Nd—O(2)	71.4 (1)	O(W1)—Nd—O(W2)	69.7 (1)
O(W1)—Nd—O(1) ^a	141.6 (1)	O(W1)—Nd—O(4) ^b	75.2 (1)
O(W1)—Nd—O(2) ^a	111.5 (1)	O(W1)—Nd—O(4') ^b	67.4 (1)
O(W2)—Nd—O(1) ^a	134.2 (1)	O(W2)—Nd—O(4) ^b	75.3 (1)
O(W2)—Nd—O(2) ^a	141.5 (1)	O(W2)—Nd—O(4') ^b	134.4 (1)
O(1) ^a —Nd—O(2)	137.2 (1)	O(1) ^a —Nd—O(4) ^b	83.0 (1)
O(1) ^a —Nd—O(1')	68.7 (1)	O(1) ^a —Nd—O(2')	77.2 (1)
O(1) ^a —Nd—O(4') ^b	78.0 (1)	O(2)—Nd—O(4) ^b	139.7 (1)
O(2)—Nd—O(2') ^a	71.9 (1)	O(2)—Nd—O(4') ^b	107.0 (1)
O(4) ^b —Nd—O(1')	77.4 (1)	O(4) ^b —Nd—O(2')	126.8 (1)
O(4) ^b —Nd—O(2) ^a	143.2 (1)	O(4) ^b —Nd—O(4') ^b	79.5 (1)
O(1')—Nd—O(2) ^a	113.0 (1)	O(1')—Nd—O(4') ^b	141.2 (1)
O(2')—Nd—O(2) ^a	71.7 (1)	O(2')—Nd—O(4) ^b	140.5 (1)
O(2') ^a —Nd—O(4') ^b	71.1 (1)	O(1) ^a —Nd—O(2) ^a	69.8 (1)

Symmetry code: (a) $-x, -y, -z$; (b) $x, 1+y, z$.

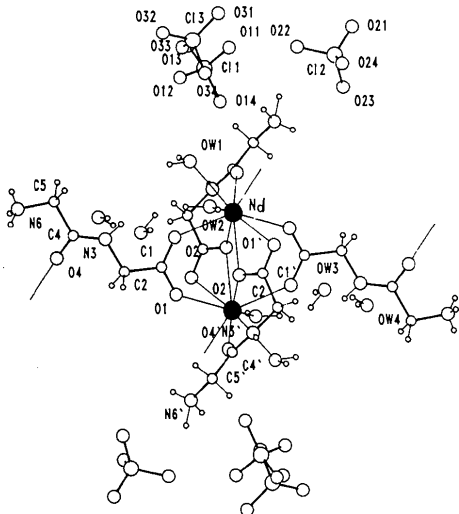


Fig. 1. Perspective view of two formula units of the title complex. Atoms of the crystallographic asymmetric unit are labelled as in the text. The disorder sites, excepting four major O sites for each perchlorate group, are excluded for clarity.

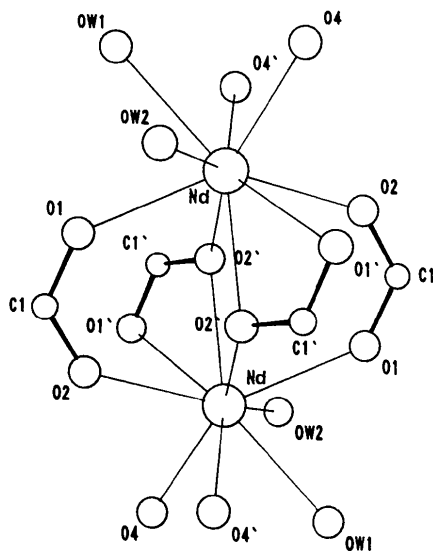


Fig. 2. Perspective view of the coordination around the pair of trivalent neodymium cations.

Waškowska, 1989). Nevertheless, the distance of 3.9958 (4) Å between the paired neodymium ions in the present complex is considerably shorter than the related inter-cationic distances of 4.248 (1), 5.171 (1) and 5.271 (1) Å observed in Nd₂(Gly)₆(ClO₄)₃·9H₂O and also somewhat shorter than the Ho³⁺...Ho³⁺ distance of 4.196 (0) Å in Ho(Gly)₃(H₂O)₃(ClO₄)₃. It is comparable, however, with the distances of 4.116 (0) for Pr³⁺...Pr³⁺, 3.899 (1) and 3.897 (1) for Ho³⁺...Ho³⁺ and 3.914 (1) and 3.909 (1) Å for Dy³⁺...Dy³⁺, found in the glutamate complexes. The coordination (Fig. 2) is irregular, differing significantly from the capped square antiprism or from the tricapped trigonal prism, the two ideal polyhedra for nine-coordination (Drew, 1977). It most closely resembles the environment of the holmium and dysprosium ions in their glutamate perchlorate hydrates (Csöreg, Czugler, Kierkegaard, Legendziewicz & Huskowska, 1989).

The bond distances and the bond angles within the two peptide molecules, occurring in their zwitterion form, agree well with each other and generally conform to the expected values. Each glycylglycine ligand coordinates through its three O atoms, but the two molecules in the crystallographic asymmetric unit behave differently. The primed peptide, having the tridentate carboxyl group, is coordinated to a pair of Nd³⁺ ions, whereas the three O atoms of the unprimed ligand molecule bind to three different cations, thus interlinking the bridged cation pairs in the crystallographic *b* direction (Fig. 3, deposited). Accordingly, endless polymeric chains are formed.

The perchlorate counter ions, well shielded from the neodymium cations, fill up the voids between the polymeric chains together with two non-coordinating crystal water molecules. The observed Cl—O bond lengths in the disordered ClO_4^- groups range from 1.34 to 1.64 Å, with a calculated average distance of 1.43 [6] Å (the root-mean-square deviation is given in square brackets), which is comparable with the commonly accepted mean value of 1.44 Å for such bonds (Ondik & Smith, 1968).

The numerous examples of intermolecular contact distances shorter than 3.3 Å between the non-H atoms (Table 8, deposited) indicate that besides the possible hydrogen bonds (Table 8) the crystal structure seems to be stabilized by an electrostatically favourable packing.

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Structure of the Crystalline Dimer of α -Hydroxyisobutyrophenone: (*R,S*)-3,3,6,6-Tetramethyl-1,4-diphenyl-2,5,7-trioxabicyclo[2.2.1]heptane

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Abstract. $\text{C}_{20}\text{H}_{22}\text{O}_3$, $M_r = 310.39$, monoclinic, $P2_1/c$, $a = 16.58$ (3), $b = 8.34$ (1), $c = 24.35$ (3) Å, $\beta = 91.2$ (2)°, $V = 3367$ (8) Å³, $Z = 8$, $D_x = 1.224$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.76$ cm⁻¹, $F(000) = 1328$, $T = 296$ K, $R = 0.077$, 1712 unique observed reflections. The crystalline compound slowly formed from α -hydroxyisobutyrophenone and assigned various structures over the past 100 years, but

none unequivocally, is shown to be racemic 3,3,6,6-tetramethyl-1,4-diphenyl-2,5,7-trioxabicyclo-[2.2.1]heptane. The *R* and *S* enantiomers in the asymmetric unit are related to each other by a pseudocenter of inversion.

Introduction. While it has long been recognized that α -hydroxy ketones dimerize into crystalline or glassy solids, the chemical structure of these products has remained controversial. Four types of dimeric structures have generally been proposed, *viz*: bis- β -keto ethers (I) (Favorskii & Mandryka, 1912; Favorskii,

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