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Bis(μ-*N*-acetyl-*N*-phenylglycinato-κ^2O:O')-bis[triaqua(1,10-phenanthroline-κ^2N,N')-lanthanum(III)] bis(*N*-acetyl-*N*-phenylglycinate) dinitrate dihydrate

Ai-Yun Fu,[a]* Da-Qi Wang,[b] Qing-Jun Shen[a] and Cun-Lan Zhang[a]

[a]Department of Chemistry, Dezhou University, Shandong Dezhou 253023, People's Republic of China, and [b]Department of Chemistry, Liaocheng University, Shandong Liaocheng 252059, People's Republic of China

Correspondence e-mail:
aiyunfu@yahoo.com.cn

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In the title complex, $[La_2(C_{10}H_{10}NO_3)_2(C_{12}H_8N_2)_4(H_2O)_6]$-$(C_{10}H_{10}NO_3)_2(NO_3)_2\cdot2H_2O$, each La^{III} ion is nine-coordinated by four N atoms from two bidentate 1,10-phenanthroline ligands and by five O atoms (two from *N*-acetyl-*N*-phenylglycinate ligands and three from water molecules). The La^{III} cations, which exhibit distorted tricapped trigonal prismatic coordination, are bridged by two *N*-acetyl-*N*-phenylglycinate ligands into a dimeric structure, generated by inversion symmetry. The crystal structure is stabilized by a three-dimensional hydrogen-bond network.

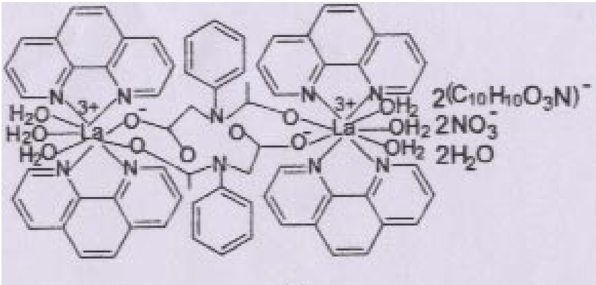
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.032
wR factor = 0.083
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The formula unit of the title compound, (I) (Fig. 1), consists of an $[La_2(C_{10}H_{10}NO_3)_2(C_{12}H_8N_2)_4(H_2O)_6]^{4+}$ cation, two $C_{10}H_{10}O_3N^-$ anions, two NO_3^- anions and two uncoordinated water molecules. Each La^{III} ion is nine-coordinated by four N atoms (N2, N3, N4 and N5) from two 1,10-phenanthroline (L_1) ligands (Table 1), atom O1 from the carboxylate group of an *N*-acetyl-*N*-phenylglycinate (L_2) ligand, atom O3^{i} (see Table 1 for symmetry code) from the acetyl group of another L_2 ligand and atoms O4, O5 and O6 from three water molecules. The coordination sphere around La is distorted tricapped trigonal prismatic, with the capping positions occupied by atom N3 of L_1, N4 of another L_1 and O5 (water). The coordinated L_2 ligand bonds to one La *via* a carboxylate group O atom and to another La atom *via* an acetyl O atom, thus acting as a bridge. The overall complex cation has inversion symmetry.



(I)

The La—O bond lengths in (I) are in the range 2.415 (2)–2.553 (2) Å, with a carboxylate O atom making the shortest bond. The average La—N bond length in (I) of 2.766 Å is longer than that seen (2.666 Å) in bis[tris(*N*-phenyl-*N*-acetylglycine)(1,10-phenanthroline)lanthanum] (Fu *et al.*, 2004)

The crystal packing in (I) is stabilized by O—H⋯O hydrogen bonds (Table 2). Notable among these are the O4—

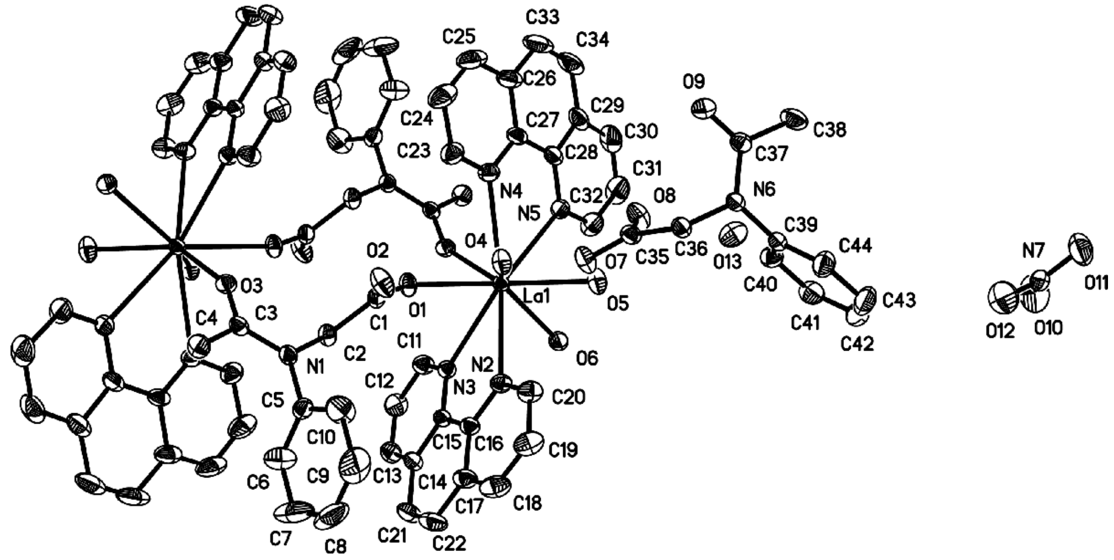


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

H2···O7 and O5—H3···O8 bonds from coordinated water molecules to uncoordinated L_2 molecules. Overall, a three-dimensional network results.

Experimental

$La(NO_3)_3 \cdot nH_2O$ (1 mmol) and L_1 (2 mmol) were dissolved in distilled water (20 ml). To this solution, an aqueous mixture (30 ml) of HL_2 (1 mmol) and NaOH (1 mmol) was added dropwise at 313 K. The resulting mixture was stirred for 6 h and part of the solvent was evaporated in a rotary vacuum evaporator at the same temperature. The resulting solution was filtered and the filtrate left in air for about six weeks, after which large yellow block-shaped crystals of (I) were obtained. Elemental analysis found: C 51.93, H 4.36, N 9.64%; calculated for $C_{88}H_{88}La_2N_{14}O_{26}$: C 51.88, H 4.29, N 9.55%.

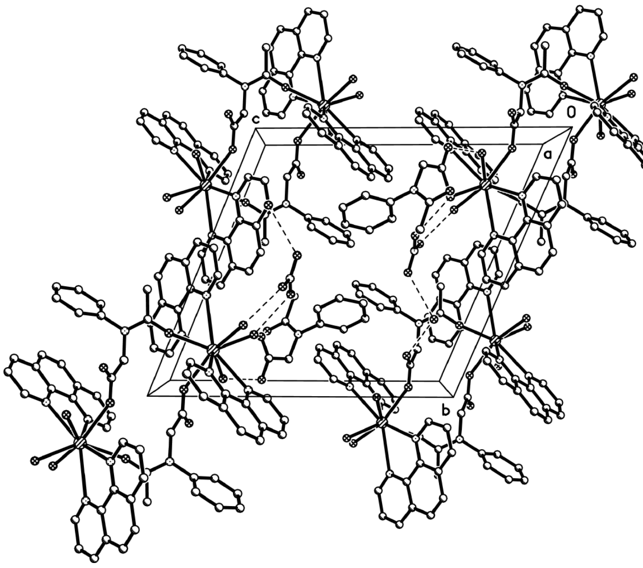


Figure 2
The crystal packing of (I), showing the the O···O hydrogen-bonded interactions as dashed lines (H atoms have been omitted for clarity).

Crystal data

$[La_2(C_{10}H_{10}NO_3)_2(C_{12}H_8N_2)_4(H_2O)_6](C_{10}H_{10}NO_3)_2(NO_3)_2 \cdot 2H_2O$
M_r = 2035.54
Triclinic, $P\overline{1}$
a = 11.4722 (19) Å
b = 14.271 (2) Å
c = 14.986 (2) Å
α = 66.966 (2)°
β = 86.448 (2)°
γ = 79.303 (2)°
V = 2218.6 (6) Å^3
Z = 1
D_x = 1.524 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6354 reflections
θ = 2.3–27.8°
μ = 1.04 mm^{-1}
T = 298 (2) K
Block, yellow
0.45 × 0.37 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.653, T_{max} = 0.828
11765 measured reflections
7742 independent reflections
6642 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -14 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.01
7742 reflections
610 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 1.4707P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Selected bond distances (Å).

La1—O1	2.415 (2)	La1—N2	2.726 (3)
La1—O5	2.483 (2)	La1—N4	2.745 (3)
La1—O3^i	2.503 (2)	La1—N3	2.755 (3)
La1—O4	2.542 (2)	La1—N5	2.838 (3)
La1—O6	2.553 (2)		

Symmetry code: (i) $2 - x, -y, -z$.

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Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O4—H1⋯O2	0.893 (10)	2.002 (15)	2.855 (3)	159 (3)
O4—H2⋯O7	0.898 (10)	1.720 (11)	2.616 (3)	175 (4)
O5—H3⋯O8	0.903 (10)	1.706 (14)	2.598 (4)	169 (4)
O5—H4⋯O10^{i}	0.896 (10)	1.888 (16)	2.760 (4)	164 (3)
O6—H5⋯O11^{i}	0.897 (10)	1.879 (17)	2.744 (4)	161 (4)
O6—H14⋯O9^{ii}	0.896 (10)	1.843 (15)	2.719 (3)	165 (4)
O13—H15⋯O12^{i}	0.902 (10)	2.015 (12)	2.910 (5)	172 (3)
O13—H16⋯O2^{iii}	0.901 (10)	1.948 (15)	2.825 (4)	164 (4)

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $1 + x, y, z$; (iii) $x, 1 + y, z$.

Water H atoms were found in difference maps and the O—H distances were restrained to 0.90 (1) Å; the U_{iso}(H) values were allowed to refine. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å, depending on hybridization, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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