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Tetrakis(μ-*N*-acetyl-*N*-phenylglycinato)-bis[(*N*-acetyl-*N*-phenylglycinato)(1,10-phenanthroline-κ^2N,N')lanthanum(III)] dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.025
wR factor = 0.070
Data-to-parameter ratio = 13.4

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

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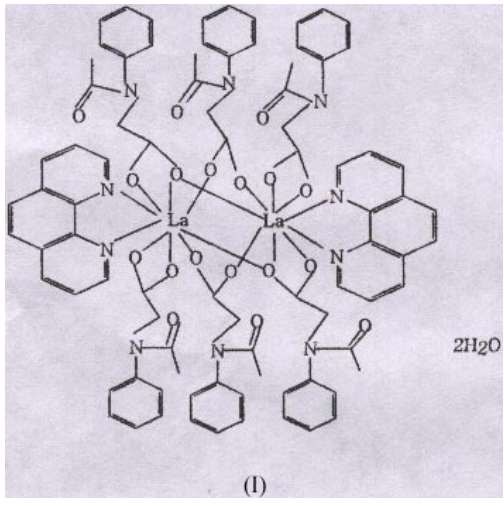
In the title complex, $[La_2(C_{10}H_{10}NO_3)_6(C_{12}H_8N_2)_2]\cdot 2H_2O$, the La^{III} atoms are bridged by two terdentate and two bidentate carboxylate groups with an inversion centre between the two La^{III} ions. Each La atom is nine-coordinated by two N atoms of 1,10-phenanthroline and seven O atoms belonging to *N*-acetyl-*N*-phenylglycine molecules, and exhibits distorted tricapped trigonal prismatic geometry. The crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

Comment

The title complex, (I) (Fig. 1), contains one dinuclear lanthanum/phenanthroline/*N*-acetyl-*N*-phenylglycinate complex and two uncoordinated water molecules. Each lanthanum ion is coordinated by one 1,10-phenanthroline (L_1) ligand *via* atoms N4 and N5 (Table 1), one chelating bidentate carboxylate group of an *N*-phenyl-*N*-acetylglycine (L_2) ligand *via* O7 and O8, two bridging bidentate carboxylate groups of L_2 *via* O5^i (see Table 1 for symmetry code) and O4, and one bridging terdentate carboxylate group of L_2 *via* O1^i and chelating terdentate carboxytate groups of L_2 *via* O1 and O2.



(I)

Overall, the coordination geometry around La is that of a distorted tricapped trigonal prism, with the capping positions occupied by atoms N5 of L_1 and O1 and O7 of two L_2 ligands. The two La ions are connected by four L_2 ligands *via* two bidentate and two terdentate carboxylate bridges with an inversion centre between the two La ions. The average of the bridging bidentate La—O bonds (2.452 Å) is slightly shorter than that of the bridging terdentate La—O bonds (2.467 Å), which in turn is shorter than the average of the chelating terdentate La—O bonds (2.584 Å).

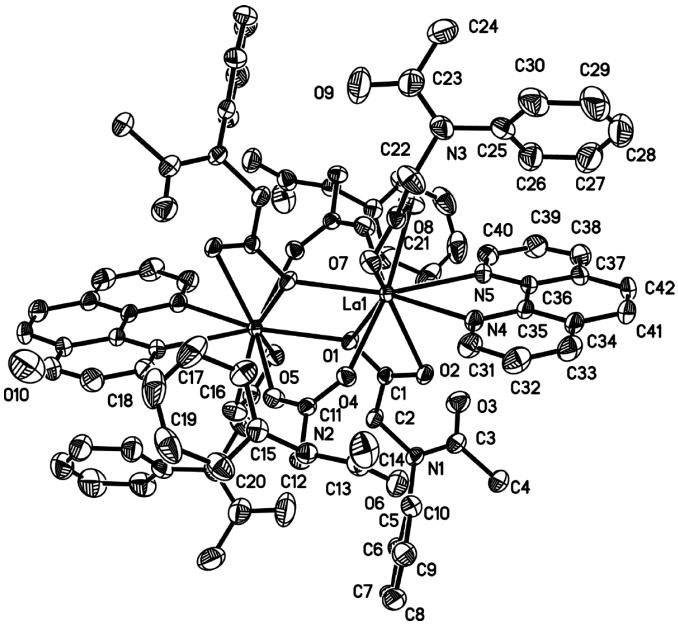


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

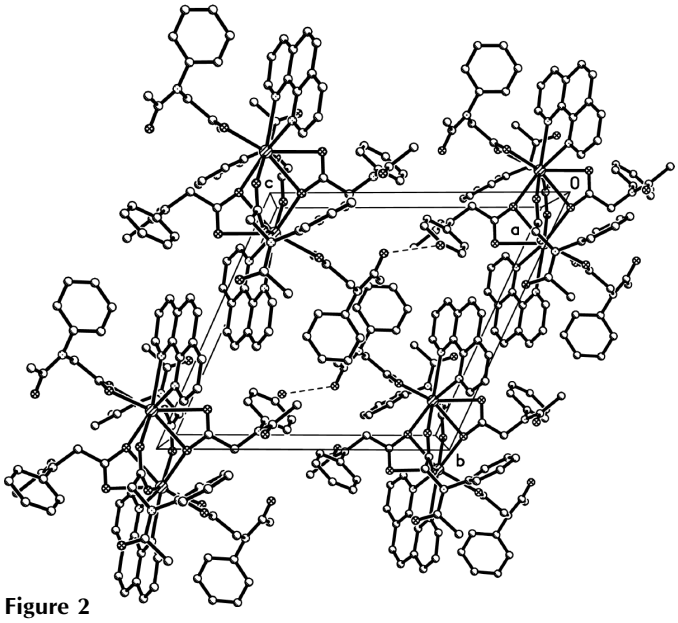


Figure 2
The crystal packing of (I), showing the O···O hydrogen-bonded interactions as dashed lines (all H atoms and the water molecules have been omitted for clarity).

The La—O bonds in (I) are shorter than the equivalent bonds in the related compound bis(1,10-phenanthroline)-tris(*trans*-2,3-dimethylacrylato)lanthanum(III) (Lu *et al.*, 2001), where the corresponding La—O bridging bidentate, La—O bridging terdentate, and La—O chelating terdentate distances are 2.473, 2.474 and 2.661 Å, respectively. The other bond lengths and angles in (I) are unexceptional.

The water O atom in (I) does not coordinate to La but participates in intermolecular O—H···O hydrogen bonds (Table 2) which stabilize the crystal packing of (I) (Fig. 2).

Experimental

$La(NO_3)_3{\cdot}nH_2O$ (1 mmol) and L_1 (1 mmol) were dissolved in anhydrous ethanol (20 ml). To this solution, an aqueous mixture (30 ml) of L_2 (2 mmol) and NaOH (2 mmol) was added dropwise at 313 K. The mixture was stirred for 4 h and about half of the solvent was evaporated in a rotary vacuum evaporator at the same temperature. The resulting solution was filtered and left to stand in air for about 20 d. Large yellow block-shaped crystals of (I) were obtained (m.p. 531.5 K). Elemental analysis found: C 55.13, H 4.32, N 7.52%; calculated for $C_{84}H_{80}La_2N_{10}O_{20}$: C 55.21, H 4.41, N 7.66%.

Crystal data

$[La_2(C_{10}H_{10}NO_3)_6(C_{12}H_8N_2)_2]\cdot 2H_2O$
M_r = 1827.40
Triclinic, $P\overline{1}$
a = 11.777 (3) Å
b = 13.574 (3) Å
c = 14.114 (3) Å
α = 65.372 (2)°
β = 86.194 (3)°
γ = 81.600 (3)°
V = 2029.0 (8) Å^3
Z = 1
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7895 reflections
θ = 2.3–28.3°
μ = 1.12 mm^{-1}
T = 298 (2) K
Block, yellow
0.45 × 0.32 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.633, T_{max} = 0.824
10710 measured reflections
7083 independent reflections
6303 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.0°
$h = -11 \rightarrow 14$
$k = -15 \rightarrow 16$
$l = -13 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.070
S = 1.00
7083 reflections
529 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 1.0763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 1.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected bond distances (Å).

La1—O4	2.431 (2)	La1—O1	2.5762 (19)
La1—O1^{i}	2.4672 (19)	La1—N4	2.658 (2)
La1—O5^{i}	2.474 (2)	La1—N5	2.674 (2)
La1—O8	2.513 (2)	La1—O2	2.675 (2)
La1—O7	2.573 (2)	La1···La1^{i}	4.0167 (9)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O10—H1···O3^{ii}	0.912 (10)	1.960 (14)	2.858 (5)	168 (3)
O10—H2···O9^{iii}	0.910 (10)	1.927 (12)	2.836 (5)	178 (4)

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

The water H atoms were located in a difference map 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 posi-

tions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak in the difference map is 1.57 Å from atom C35.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); 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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