

Tetrakis(μ -*N*-acetyl-*N*-phenylglycinato- κ^2 O, O')-bis[*N*-acetyl-*N*-phenylglycinato- κ^3 O, O,O,O]-(*1,10*-phenanthroline- κ^2 N, N')cerium(III)] dihydrateAi-Yun Fu,^{a,b*} Shi-Zhou Fu^b and Tao Yu^a^aDepartment of Chemistry, Dezhou University, Shandong Dezhou 253023, People's Republic of China, and ^bDepartment of Chemistry, Liaocheng University, Shandong Liaocheng 252059, People's Republic of ChinaCorrespondence e-mail:
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Key indicators

Single-crystal X-ray study
T = 571 K
Mean σ (C–C) = 0.012 Å
R factor = 0.047
wR factor = 0.123
Data-to-parameter ratio = 13.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title complex, $[\text{Ce}_2(\text{C}_{10}\text{H}_{10}\text{NO}_3)_6(\text{C}_{12}\text{H}_8\text{N}_2)_2]\cdot 2\text{H}_2\text{O}$, each Ce atom shows a distorted tricapped trigonal prismatic coordination, comprising two N-atom donors from a *1,10*-phenanthroline ligand and seven O atoms of the *N*-acetyl-*N*-phenylglycine (*L2*) molecules. Two Ce atoms are bridged by two terdentate and two bidentate carboxylate groups of *L2*, to give a centrosymmetric dimer. The crystal structure is stabilized by intermolecular O–H...O hydrogen bonds.

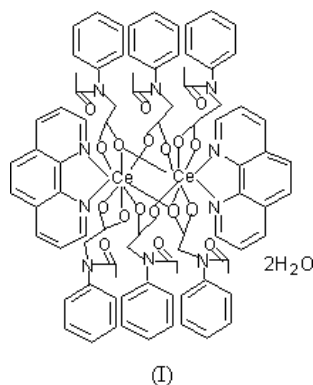
Received 29 November 2004

Accepted 21 December 2004

Online 8 January 2005

Comment

The title complex, (I) (Fig. 1), contains centrosymmetric dinuclear cerium/phenanthroline/*N*-acetyl-*N*-phenylglycinate complexes and uncoordinated water molecules. Each cerium(III) ion is nine-coordinated by one *1,10*-phenanthroline (*L1*) ligand *via* atoms N4 and N5, one chelating bidentate carboxylate group of an *N*-acetyl-*N*-phenylglycine (*L2*) ligand *via* O4 and O5, two bridging bidentate carboxylate groups from two *L2* ligands *via* O8ⁱ (see Table 1 for symmetry code) and O7, and one bridging terdentate carboxylate group of *L2* *via* O1ⁱ and chelating terdentate carboxylate groups of *L2* *via* O1 and O2.



The coordination geometry around Ce is that of a distorted tricapped trigonal prism, with the capping positions occupied by atoms N5 of *L1* and O1 and O4 of two *L2* ligands. The two Ce ions are connected by four *L2* ligands *via* two bidentate and two terdentate carboxylate bridges, with a Ce...Ce distance of 3.9972 (19) Å. The average length of the bridging bidentate Ce–O bonds (2.448 Å) is slightly less than that of the bridging terdentate Ce–O1ⁱ bond [2.461 (4) Å] which, in turn, is less than the average for the chelating bidentate Ce–O bonds [2.533 (5) Å]. The chelating terdentate Ce–O bond is the longest of all. All of these are shorter than the Ce–N bonds. The other bond lengths and angles in (I) are unexceptional.

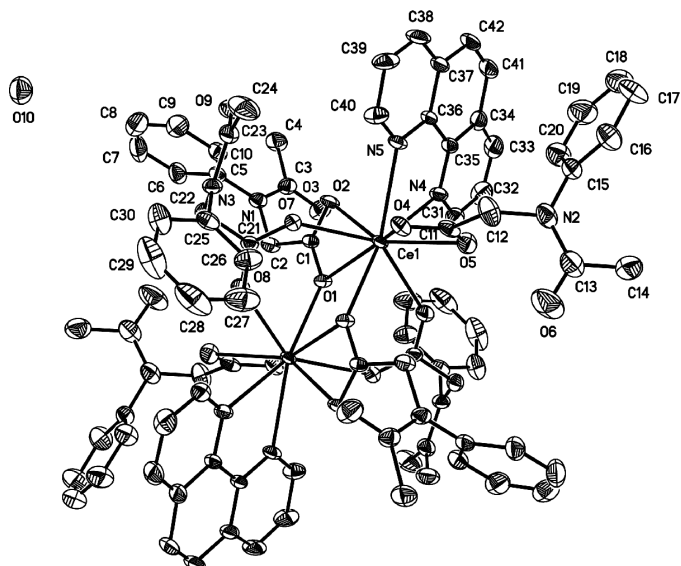


Figure 1
The structure of the title compound, (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted.

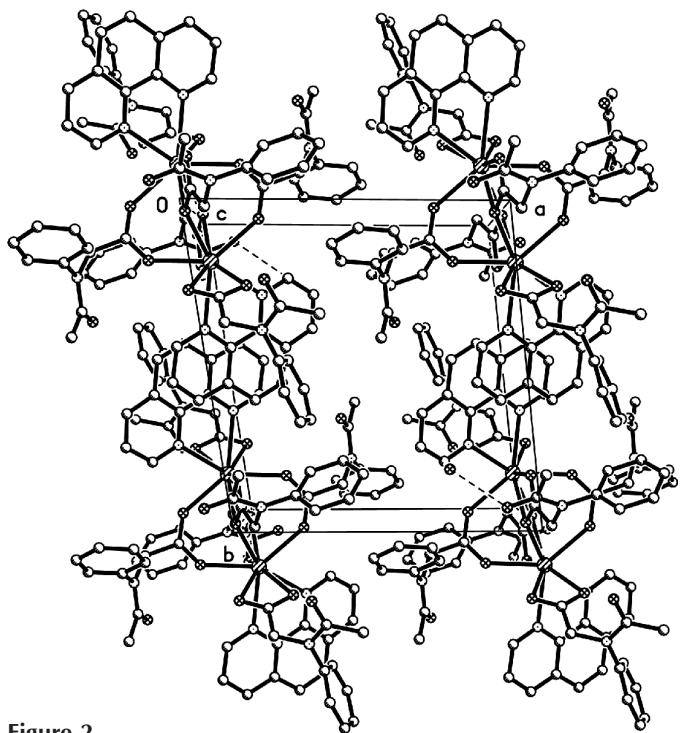


Figure 2
The crystal packing of (I), showing the hydrogen-bonding interactions as dashed lines. H atoms have been omitted.

The Ce—O bonds in (I) are shorter than the equivalent La—O bonds in the corresponding lanthanum compound (Fu *et al.*, 2004), where the average La—O bonds for bridging bidentate, bridging terdentate, chelating bidentate, chelating terdentate ligands and the average La—N bond distances are 2.452, 2.467, 2.543, 2.625 and 2.666 Å, respectively. The water O atom in (I) does not coordinate to Ce, but participates in intermolecular O—H...O hydrogen bonds (Table 2), which stabilize the crystal packing (Fig. 2).

Experimental

Ce(NO₃)₃·*n*H₂O (1 mmol) and L1 (1 mmol) were dissolved in anhydrous ethanol (20 ml). To this solution, an aqueous solution (30 ml) of L2 (2 mmol) and NaOH (2 mmol) was added dropwise at 313 K. The mixture was stirred for 3 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 30 d. Large yellow block-like crystals were obtained. Elemental analysis found: C 55.08, H 4.33, N 7.56%; calculated for C₈₄H₈₀Ce₂N₁₀O₂₀: C 55.14, H 4.41, N 7.65%.

Crystal data

[Ce₂(C₁₀H₁₀NO₃)₆·
(C₁₂H₈N₂)₂·2H₂O]
M_r = 1829.82
Triclinic, *P* $\bar{1}$
a = 11.750 (6) Å
b = 13.512 (7) Å
c = 14.068 (7) Å
 α = 65.430 (6)°
 β = 86.264 (7)°
 γ = 81.663 (7)°
V = 2009.7 (18) Å³

Z = 1
D_x = 1.512 Mg m⁻³
Mo *K*α radiation
Cell parameters from 3651
reflections
 θ = 2.3–25.0°
 μ = 1.20 mm⁻¹
T = 571 (2) K
Block, yellow
0.41 × 0.32 × 0.19 mm

Data collection

Bruker SMART CCD area-detector
diffractometer
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 1997)
T_{min} = 0.640, *T_{max}* = 0.805
10237 measured reflections

6953 independent reflections
5257 reflections with *I* > 2σ(*I*)
R_{int} = 0.036
 θ_{max} = 25.0°
h = -13 → 13
k = -15 → 16
l = -16 → 16

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.047
wR (*F*²) = 0.123
S = 1.01
6953 reflections
532 parameters

H atoms treated by a mixture of
independent and constrained
refinement
w = 1/[σ²(*F_o*²) + (0.0636*P*)²]
where *P* = (*F_o*² + 2*F_c*²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 1.66 e Å⁻³
Δρ_{min} = -1.10 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|--------------------------------------|-------------|-------------------------|-------------|
| Ce1—O7 | 2.429 (4) | Ce1—O1 | 2.571 (3) |
| Ce1—O1 ⁱ | 2.463 (4) | Ce1—N5 | 2.651 (5) |
| Ce1—O8 ⁱ | 2.466 (4) | Ce1—N4 | 2.660 (5) |
| Ce1—O5 | 2.504 (4) | Ce1—O2 | 2.667 (4) |
| Ce1—O4 | 2.561 (4) | | |
| O7—Ce1—O1 ⁱ | 72.11 (12) | O5—Ce1—N5 | 77.82 (14) |
| O7—Ce1—O8 ⁱ | 137.67 (13) | O4—Ce1—N5 | 70.59 (14) |
| O1 ⁱ —Ce1—O8 ⁱ | 73.43 (13) | O1—Ce1—N5 | 127.88 (13) |
| O7—Ce1—O5 | 131.98 (14) | O7—Ce1—N4 | 131.22 (14) |
| O1 ⁱ —Ce1—O5 | 95.97 (13) | O1 ⁱ —Ce1—N4 | 153.45 (14) |
| O8 ⁱ —Ce1—O5 | 75.27 (14) | O8 ⁱ —Ce1—N4 | 80.02 (14) |
| O7—Ce1—O4 | 80.92 (13) | O5—Ce1—N4 | 77.18 (15) |
| O1 ⁱ —Ce1—O4 | 76.88 (12) | O4—Ce1—N4 | 115.24 (14) |
| O8 ⁱ —Ce1—O4 | 114.08 (14) | O1—Ce1—N4 | 97.05 (13) |
| O5—Ce1—O4 | 51.12 (14) | N5—Ce1—N4 | 61.75 (15) |
| O7—Ce1—O1 | 75.56 (12) | O7—Ce1—O2 | 71.73 (12) |
| O1 ⁱ —Ce1—O1 | 74.90 (13) | O1 ⁱ —Ce1—O2 | 118.90 (11) |
| O8 ⁱ —Ce1—O1 | 72.33 (12) | O8 ⁱ —Ce1—O2 | 105.35 (13) |
| O5—Ce1—O1 | 147.60 (13) | O5—Ce1—O2 | 144.16 (14) |
| O4—Ce1—O1 | 147.61 (12) | O4—Ce1—O2 | 140.45 (14) |
| O7—Ce1—N5 | 84.77 (14) | O1—Ce1—O2 | 49.32 (11) |
| O1 ⁱ —Ce1—N5 | 142.58 (14) | N5—Ce1—O2 | 78.81 (13) |
| O8 ⁱ —Ce1—N5 | 137.13 (14) | N4—Ce1—O2 | 67.90 (13) |

Symmetry code: (i) -*x*, 2 - *y*, 1 - *z*.

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| O10–H2 \cdots O3 ⁱⁱ | 0.82 (2) | 2.06 (2) | 2.858 (7) | 165 (6) |
| O10–H1 \cdots O6 ⁱⁱⁱ | 0.84 (3) | 2.10 (3) | 2.824 (8) | 145 (5) |

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

The water O–H distances were restrained to 0.85 (3) Å and the H \cdots H distance to 1.38 (3) Å; their $U_{\text{iso}}(\text{H})$ 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 and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The highest peak in the final difference map is 1.5 Å from C36 and the deepest hole is 1.0 Å from the Ce atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors thank the Science and Technology Office of Dezhou City, Shandong Province, People's Republic of China, for research grant No. 030701.

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