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Aqua[2,2′-(naphthalene-2,3-dioxy)bis(*N*,*N*-diethylacetamide)]trinitratocerium(III) acetonitrile solvate

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In the title compound, $[Ce(NO_3)_3(C_{22}H_{30}N_2O_4)(H_2O)]\cdot CH_3CN$, the Ce^{III} atom is coordinated by 11 O atoms in a distorted monocapped pentagonal antiprismatic geometry. The Ce—O(carbonyl) bond distances [average 2.415 (3) Å] are significantly shorter than the other Ce—O bonds [2.495 (3)–2.931 (3) Å] in the complex.

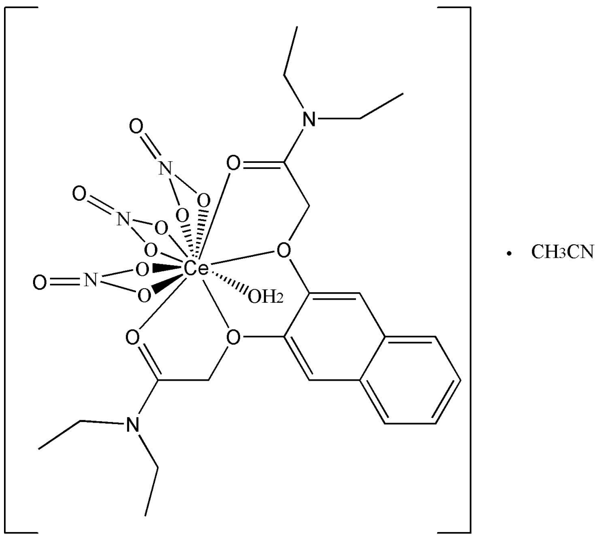
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.091
Data-to-parameter ratio = 13.1

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

Comment

The luminescent property of lanthanide complexes incorporating open-chain crown ethers has attracted our attention. As 2,2′-(naphthalene-2,3-dioxy)bis(*N*,*N*-diethylacetamide) (*L*) has the proper conjugate absorption group, it has been used as a chelating ligand to prepare the title Ce^{III} complex, (I), in our laboratory.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The Ce^{III} atom is coordinated by 11 O atoms, four from the tetradentate *L* ligand, six from chelating nitrate anions and one from the water molecule, with a distorted monocapped pentagonal antiprismatic geometry. The bond distances between atom Ce1 and the carbonyl O atoms (O1 and O3) are significantly shorter than the other Ce1—O bond distances (Table 1). The four O atoms of *L* are coplanar, but atom Ce1 lies 0.475 (3) Å out of the mean plane formed by the four O atoms.

The coordinated water molecule is hydrogen bonded with a nitrate group of a neighboring complex. Weak C—H···N hydrogen bonding occurs between the Ce^{III} complex and the acetonitrile solvent molecule (Table 2 and Fig. 1).

Experimental

Ligand *L* was prepared according to the method described by Zhang & Liu (2003). An ethyl acetate solution of $Ce(NO_3)_3{\cdot}6H_2O$ (0.1 mmol) was added dropwise to an ethyl acetate solution (20 ml) of *L* (0.1 mmol). The mixture was stirred for 4 h and a white precipitate formed. The precipitate was separated and washed three times with ethyl acetate (yield 75%). Single crystals of (I) were obtained by recrystallization from a CH_3CN solution. Analysis calculated for $C_{24}H_{35}CeN_6O_{14}$: C 37.21, H 4.22, N 10.80%; found: C 37.38, H 4.54, N 10.87%.

Crystal data

$[Ce(NO_3)_3(C_{22}H_{30}N_2O_4)$-$(H_2O)]{\cdot}C_2H_3N$
$M_r = 771.70$
Triclinic, $P\overline{1}$
$a = 8.457$ (7) Å
$b = 13.732$ (11) Å
$c = 15.621$ (13) Å
$\alpha = 115.53$ (1)°
$\beta = 90.89$ (1)°
$\gamma = 102.366$ (11)°
$V = 1587$ (2) $Å^3$
$Z = 2$
$D_x = 1.615$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6489 reflections
$\theta = 3.0$–25.0°
$\mu = 1.51$ mm^{-1}
$T = 295$ (2) K
Block, colorless
0.47 × 0.42 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.537$, $T_{max} = 0.620$
8010 measured reflections
5429 independent reflections
4841 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$
$h = -10 \rightarrow 9$
$k = -10 \rightarrow 16$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.08$
5429 reflections
416 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 2.3465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.29$ e $Å^{-3}$
$\Delta\rho_{min} = -1.05$ e $Å^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.126 (3)

Table 1
Selected interatomic distances (Å).

Ce1—O1	2.415 (3)	Ce1—O8	2.628 (4)
Ce1—O2	2.898 (4)	Ce1—O9	2.648 (4)
Ce1—O3	2.414 (3)	Ce1—O11	2.607 (4)
Ce1—O4	2.931 (3)	Ce1—O12	2.672 (4)
Ce1—O5	2.697 (4)	Ce1—O14	2.495 (3)
Ce1—O6	2.590 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O14—H1···O5^{i}	0.83	2.17	2.841 (5)	138
O14—H2···O10^{ii}	0.86	1.95	2.811 (6)	178
C14—H14···N6	0.93	2.60	3.505 (9)	164

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

H atoms on the water molecule were located in a difference Fourier map and refined riding in their as-found positions relative to atom O14, with a fixed isotropic displacement parameter of 0.05 $Å^2$. The methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and their torsion angles were refined based on electron density, with $U_{iso}(H) = 1.5U_{eq}$(carrier). H atoms bonded to atom C3 were placed in calculated positions (C—H = 0.96–0.97 Å) and included in structure factors calculations with fixed positional parameters and a $U_{iso}(H)$ value of 0.05 $Å^2$. Other H atoms were placed in calculated positions, with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene), and included in the final cycles of refinement in a riding model with $U_{iso}(H) = 1.2U_{eq}$(carrier). The hightest peak and deepest hole are 1.01 and 0.83 Å from atom Ce1. The C4-methyl group is disordered over to two positions (C4a and C4b). Two occupancies were initially refined and converged to 0.632–(4) and 0.368–(4); these were fixed as 0.63 and 0.37 in final cycles of refinement.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

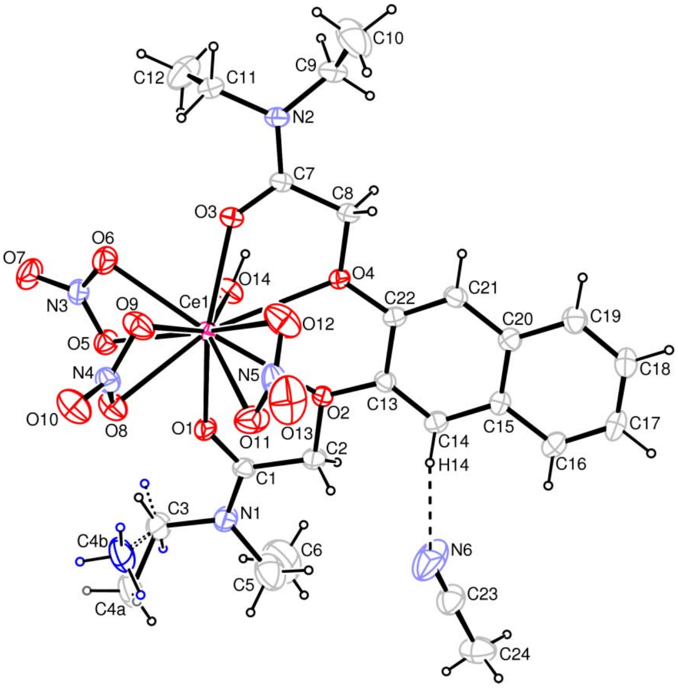


Figure 1
The structure of (I), with 30% probability displacement ellipsoids. The dashed line indicates weak C—H···N hydrogen bonding. Both disorder components are shown.

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