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Tetramethylammonium hexanitratolanthanate(III) methanol solvate

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(O–C) = 0.012 Å
R factor = 0.055
wR factor = 0.145
Data-to-parameter ratio = 19.1

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

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The title compound, $[(CH_3)_4N]_3[La(NO_3)_6]\cdot MeOH$, contains an anionic hexanitratolanthanate(III) complex with the nitrate ligands octahedrally disposed around the metal. The incorporated methanol solvent is involved in hydrogen bonding to one nitrate ligand.

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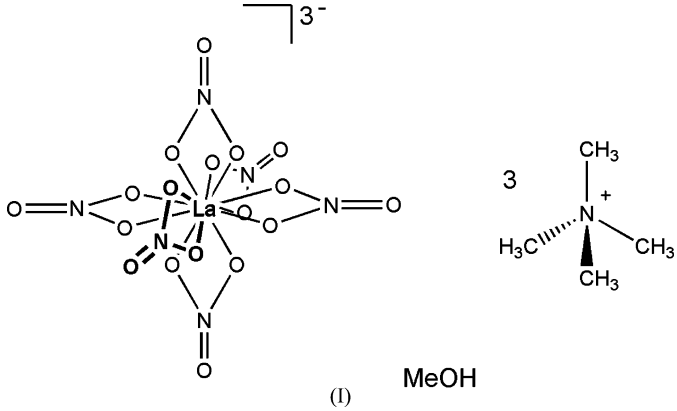
Comment

During investigations into the synthesis of rare-earth pseudohalide complexes, the use of nitrate reagents has instead yielded new rare-earth nitrato complexes. The hexanitratolanthanate(III) species is highly symmetrical, based around a 12-coordinate lanthanum centre, although this is not reflected in the overall symmetry of the structure. The chelating nitrate ligands are octahedrally disposed around the central lanthanum. All Ln—O bonds lie within a narrow length range and the O—Ln—O bite angles are all similar (see Table 1). The nitrate ligands arrange themselves so as to be mutually perpendicular around the metal centre. This arrangement of the ligands is seen in other examples of hexanitratolanthante(III) complexes, for example in $[La(NO_3)_6]^{3-}$-containing structures with terpyridinium (Drew *et al.*, 2000) and a methylpyridinium-based counter-cation (Jing *et al.*, 1994). Less regular nitrate arrangements have also been observed, such as in the tri-*n*-butylammonium structure (Yan *et al.*, 1995). One methanol molecule is incorporated into the structure and is involved in only a single interaction with a nitrate ligand.



(I)

Experimental

Tetramethylammonium nitrosodicyanamide (50 mg, 0.30 mmol), copper(II) nitrate (36 mg, 0.19 mmol) and lanthanum(III) nitrate hexahydrate (46 mg, 0.11 mmol) were dissolved in methanol (5 ml).

Colourless crystals of the title compound grew over the course of two weeks at room temperature.

Crystal data

$(C_4H_{12}N)[La(NO_3)_6]\cdot CH_4O$
$M_r = 765.45$
Monoclinic, $P2_1/c$
$a = 9.7169\ (2)$ Å
$b = 19.0510\ (4)$ Å
$c = 17.6572\ (5)$ Å
$\beta = 93.379\ (10)^\circ$
$V = 3262.96\ (13)$ Å^3
$Z = 4$
$D_x = 1.558$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.39$ mm^{-1}
$T = 123\ (1)$ K
Needle, colourless
$0.18 \times 0.08 \times 0.03$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.788$, $T_{max} = 0.959$
28586 measured reflections
7499 independent reflections
5216 reflections with $I > 2\sigma(I)$
$R_{int} = 0.096$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.145$
$S = 1.04$
7499 reflections
393 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.43$ e Å^{-3}
$\Delta\rho_{min} = -1.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

La1—O1	2.649 (3)	La1—O10	2.642 (3)
La1—O3	2.655 (3)	La1—O12	2.647 (3)
La1—O4	2.612 (4)	La1—O13	2.660 (3)
La1—O6	2.672 (3)	La1—O15	2.663 (3)
La1—O7	2.677 (3)	La1—O16	2.629 (3)
La1—O9	2.649 (3)	La1—O18	2.661 (3)
O1—La1—O3	48.2 (1)	O10—La1—O12	48.2 (1)
O4—La1—O6	48.4 (1)	O13—La1—O15	48.0 (1)
O7—La1—O9	48.2 (1)	O16—La1—O18	48.3 (1)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O19—H14···O7	0.84	2.26	3.024 (6)	152

All H atoms were placed in calculated positions and refined as riding [C—H = 0.98 Å, O—H = 0.84 Å, and $U_{iso}(H) = 1.2U_{eq}(O)$ and $1.5U_{eq}(C)$]. Two of the tetramethylammonium cations show minor signs of disorder although the small extent of this precluded satisfactory modelling. The largest residual density peak and hole are located 1.10 and 0.87 Å, respectively, from the lanthanum atom.

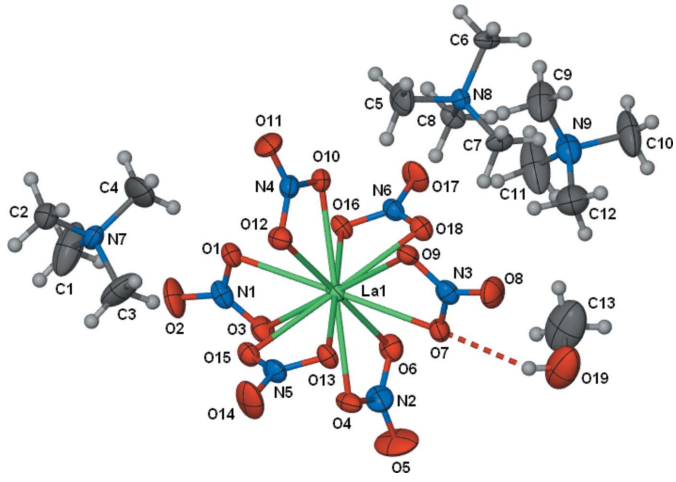


Figure 1
The asymmetric unit of $[La(NO_3)_6][Me_4N]_3\cdot MeOH$, showing relevant atomic labelling; displacement ellipsoids are shown at 50% probability.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 1999); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

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