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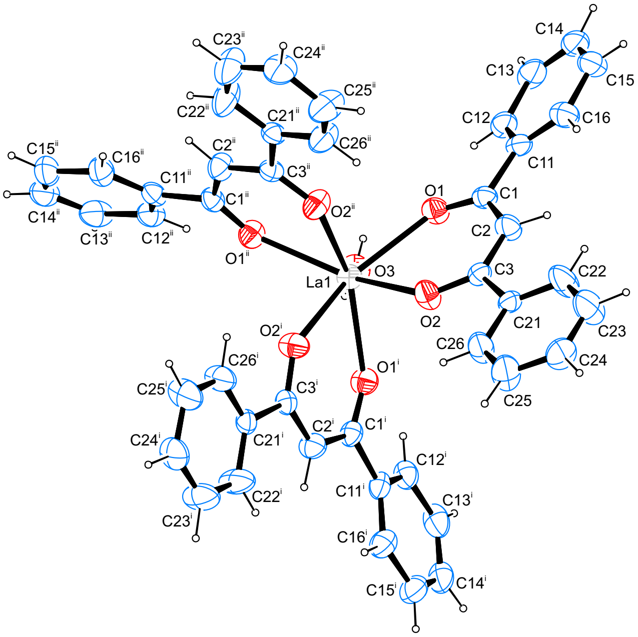
Matthew Hickson, Abigail Sephton, Travis Dugmore, Eric C. Hosten and Richard Betz*

The crystal structure of aqua-tris (1,3-diphenylpropane-1,3-dionato-$\kappa^2 O,O'$)-lanthanum(III), $C_{45}H_{35}LaO_7$



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Abstract
$C_{45}H_{35}LaO_7$, trigonal, $R3$ (no. 146), a = 22.3832(11) Å, c = 6.4522(3) Å, V = 2799.5(3) Å^3, Z = 3, $R_{gt}(F)$ = 0.0192, $wR_{ref}(F^2)$ = 0.0462, T = 200 K.

CCDC no.: 2041291

Table 1: Data collection and handling.

Crystal:	Colourless rod
Size:	0.52 × 0.10 × 0.07 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.20 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14,463, 3097, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3093
$N(param)_{refined}$:	160
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound was prepared upon reacting lanthanum nitrate hexahydrate with three equivalents of dibenzoylmethane and three equivalents of aqueous ammonia solution in water as the solvent. Crystals suitable for the diffraction studies were obtained upon saturation of the reaction mixture with acetone and subsequent storage in a cold room at around 0 °C.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic and vinylic carbon atoms) and were included in the refinement in the riding model approximation, with U(H) set to $1.2U_{eq}$(C).

Both H atoms of the water molecule were located on a difference Fourier map and were included in the refinement in the riding model approximation, with U(H) set to $1.5U_{eq}$(O).

Comment

Lanthanide compounds have attracted considerable attention due to their photoluminescent properties that

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***Corresponding author: Richard Betz**, Department of Chemistry, Nelson Mandela University, Summerstrand Campus (South), University Way, Summerstrand, PO Box 77000, Port Elizabeth, 6031, South Africa, E-mail: Richard.Betz@mandela.ac.za. https://orcid.org/0000-0002-5730-2152
Matthew Hickson, Abigail Sephton, Travis Dugmore and Eric C. Hosten, Department of Chemistry, Nelson Mandela University, Summerstrand Campus (South), University Way, Summerstrand, PO Box 77000, Port Elizabeth, 6031, South Africa. https://orcid.org/0000-0003-4173-2550 (E.C. Hosten)

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
La1	0.666667	0.333333	0.67076 (2)	0.02874 (7)
O1	0.61972 (12)	0.40750 (11)	0.7561 (3)	0.0357 (4)
O2	0.57855 (12)	0.31842 (13)	0.4182 (3)	0.0390 (5)
O3	0.666667	0.333333	1.0621 (5)	0.0381 (8)
H3A[a]	0.640070	0.338242	1.143943	0.057*
H3B[a]	0.691164	0.323583	1.137802	0.057*
C1	0.57855 (15)	0.42455 (14)	0.6797 (4)	0.0297 (6)
C2	0.53751 (17)	0.39387 (16)	0.5044 (5)	0.0362 (6)
H2	0.506380	0.408978	0.464301	0.043*
C3	0.53902 (14)	0.34292 (14)	0.3849 (4)	0.0276 (5)
C11	0.57458 (15)	0.48210 (14)	0.7856 (4)	0.0313 (6)
C12	0.60185 (16)	0.50134 (17)	0.9837 (5)	0.0370 (6)
H12	0.619639	0.476030	1.052617	0.044*
C13	0.60331 (17)	0.5572 (2)	1.0820 (6)	0.0484 (9)
H13	0.621890	0.569906	1.217706	0.058*
C14	0.57781 (18)	0.59443 (19)	0.9827 (7)	0.0527 (10)
H14	0.580579	0.633768	1.048075	0.063*
C15	0.5485 (2)	0.57482 (18)	0.7905 (6)	0.0482 (8)
H15	0.529393	0.599442	0.724954	0.058*
C16	0.54685 (18)	0.51887 (17)	0.6911 (5)	0.0405 (7)
H16	0.526598	0.505555	0.557454	0.049*
C21	0.49276 (14)	0.31523 (14)	0.1992 (4)	0.0290 (5)
C22	0.4510 (2)	0.3403 (2)	0.1316 (7)	0.0568 (11)
H22	0.449582	0.375902	0.207513	0.068*
C23	0.4111 (2)	0.3143 (2)	−0.0456 (7)	0.0623 (12)
H23	0.382853	0.332489	−0.089486	0.075*
C24	0.41179 (19)	0.2634 (2)	−0.1568 (6)	0.0501 (9)
H24	0.382422	0.244102	−0.273596	0.060*
C25	0.4554 (2)	0.2403 (2)	−0.0988 (7)	0.0597 (11)
H25	0.458673	0.206814	−0.181034	0.072*
C26	0.49482 (19)	0.2653 (2)	0.0797 (6)	0.0485 (9)
H26	0.523847	0.247681	0.120231	0.058*

[a] Occupancy: 0.3333.

already sees their grand-scale application in the manufacture of screens for television sets and computers [7]. For several decades, coordination compounds of lanthanides have also found ample application in medicine for diagnostic and therapeutic purposes [8]. At the start of a research project aimed at elucidating the nature of the ligand as well as the coordination number on the physicochemical properties of selected lanthanide coordination compounds, the title compound was synthesized and its crystal and molecular structure secured on grounds of diffraction experiments based on single crystals. For the crystal structure of a multinuclear analogue of the title compound, see [9]. Analogous mononuclear coordination compounds have also been reported for praseodymium [10], neodymium [11], samarium [10], europium [12, 13], holmium [14] and erbium [10].

The title compound is a coordination compound of lanthanum demonstrating a coordination number of seven for the central atom. The ligand sphere is comprised of three chelating dibenzoylmethanido ligands as well as one molecule of water. The asymmetric unit consists of one third of a molecule with the cation and water O atom sitting on special positions. Lanthanum-oxygen distances to the chelating ligand are measured at 2.431(2) and 2.449(2) Å while the La–O distance to the water molecule was found at 2.525(3) Å. The coordination polyhedron around the central atom is far removed from an ideal pentagonal bipyramid with O–La–O angles covering a range of only 115.02(4)°–149.07(8)°. The aromatic systems of the chelate rings are nearly co-planar – the least-squares planes as defined by the respective non-hydrogen atoms of the phenyl groups intersect at an angle of only 16.29(19)°. A conformational analysis of the chelate rings according to Cremer & Pople [15] is precluded by the low puckering amplitude.

In the crystal, three possible hydrogen bonds supported by the coordinated water molecule as donor and lanthanum-coordinated oxygen atoms as acceptors are identified. Furthermore, a C–H⋯π interaction is apparent that is supported by one of hydrogen atoms in *para* position to the moiety featuring the delocalized negative charge on one of the aromatic systems and its symmetry-generated equivalent as acceptor. In terms of graph-set analysis [16, 17], the descriptor for the classical hydrogen bonds is $C_1^1(4)$ on the unary level. In total, the molecules are connected to infinite chains along the crystallographic *c* axis by the classical hydrogen bonds. The shortest distance between two centers of gravity was measured at 4.889(2) Å.

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