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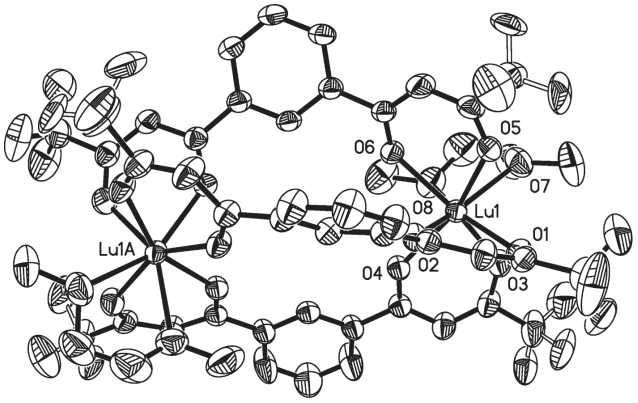
The crystal structure of tris(μ_2-1,3-bis(4,4,4-trifluoro-3-oxido-1-(oxo)but-2-en-1-yl)phenyl-$\kappa^4O,O':O'',O'''$)-bis(1,2-dimethoxyethane-κ^2O,O') dilutetium(III), $C_{50}H_{38}F_{18}Lu_2O_{16}$



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Abstract

$C_{50}H_{38}F_{18}Lu_2O_{16}$, monoclinic, $C2/c$ (no. 15), $a = 15.7024(12)$ Å, $b = 12.6169(10)$ Å, $c = 29.715(2)$ Å, $\beta = 91.919(10)°$, $V = 5883.7(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0273$, $wR_{ref}(F^2) = 0.066$, $T = 273$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound is easily synthesized by means of reported procedures [3]. To a methanol solution of 1,3-bis(4,4,4-trifluoro-1,3-dioxobutyl)phenyl (1 g, 2.8 mmol), NaOH (0.2 g, 5.6 mmol) was added, and the mixture was allowed to stir for 5 min. To this methanol solution, $LuCl_3 \cdot 6H_2O$ (1.86 mmol)

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.32 × 0.32 × 0.30 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.46 mm^{-1}
Diffractometer, scan mode:	Bruker APEX2, φ and ω-scans
$2\theta_{max}$, completeness:	28.3°, 97.1%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	23077, 7247, 0.016
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6388
$N(param)_{refined}$:	445
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Lu1	0.08988(2)	0.22420(2)	0.37582(2)	0.03603(5)
O1	0.23313(15)	0.20960(19)	0.38860(8)	0.0475(6)
O2	0.14349(14)	0.16603(18)	0.31086(7)	0.0422(5)
O3	0.13601(17)	0.37220(19)	0.41200(8)	0.0513(6)
O4	0.06174(15)	0.35881(16)	0.32652(7)	0.0411(5)
O5	0.11593(15)	0.05166(19)	0.39545(9)	0.0497(6)
O6	−0.01857(14)	0.12852(17)	0.34312(8)	0.0429(5)
O7	0.0638(2)	0.2006(2)	0.45440(9)	0.0645(7)
O8	−0.04543(16)	0.3006(2)	0.39911(10)	0.0627(7)
F1	0.42648(19)	0.0788(4)	0.38025(12)	0.1242(14)
F2	0.40274(19)	0.2310(3)	0.40830(13)	0.1016(11)
F3	0.3543(2)	0.0952(3)	0.43786(11)	0.1065(11)
F4A[a]	0.2372(7)	0.6149(6)	0.4150(4)	0.126(4)
F5A[a]	0.1420(5)	0.5741(8)	0.4619(3)	0.166(4)
F6A[a]	0.2512(6)	0.4819(6)	0.4586(3)	0.125(3)
F5B[b]	0.1850(8)	0.5127(9)	0.4741(3)	0.106(3)
F4B[b]	0.2094(8)	0.6302(7)	0.4255(4)	0.083(3)
F6B[b]	0.2869(5)	0.4924(8)	0.4308(3)	0.097(3)
F7A[c]	0.0901(7)	−0.2211(6)	0.3951(4)	0.134(5)
F8A[c]	0.2026(4)	−0.1277(7)	0.4064(4)	0.122(3)
F9A[c]	0.1109(7)	−0.1266(8)	0.4526(3)	0.156(3)
F7B[d]	0.1719(5)	−0.1777(6)	0.3695(2)	0.096(2)
F8B[d]	0.1862(6)	−0.1127(6)	0.4338(3)	0.089(3)
F9B[d]	0.0786(5)	−0.2047(7)	0.4190(3)	0.078(3)
C1	0.3688(2)	0.1382(4)	0.39841(14)	0.0607(10)
C2	0.2862(2)	0.1503(3)	0.37035(12)	0.0447(7)
C3	0.2786(2)	0.0970(3)	0.33010(12)	0.0476(8)
H3	0.3233	0.0545	0.3211	0.057*
C4	0.2044(2)	0.1056(2)	0.30213(11)	0.0394(6)
C5	0.19455(19)	0.0414(3)	0.26000(11)	0.0397(7)
C6	0.2453(2)	−0.0468(3)	0.25176(13)	0.0513(8)
H6	0.2898	−0.0643	0.2719	0.062*
C7	0.2299(2)	−0.1083(3)	0.21387(14)	0.0583(10)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H7	0.2641	−0.1668	0.2085	0.070*
C8	0.1640(2)	−0.0831(3)	0.18399(13)	0.0511(8)
H8	0.1538	−0.1250	0.1586	0.061*
C9	0.11228(19)	0.0048(3)	0.19141(11)	0.0405(7)
C10	0.12899(19)	0.0671(3)	0.22923(11)	0.0401(7)
H10	0.0959	0.1269	0.2341	0.048*
C11	0.2006(3)	0.5310(3)	0.43326(17)	0.0874(18)
C12	0.1489(2)	0.4647(3)	0.39897(12)	0.0471(8)
C13	0.1231(2)	0.5116(3)	0.35913(13)	0.0527(9)
H13	0.1379	0.5820	0.3542	0.063*
C14	0.0751(2)	0.4577(2)	0.32538(10)	0.0391(6)
C15	0.0372(2)	0.5177(3)	0.28643(11)	0.0449(7)
C16	0.0000	0.4641(3)	0.2500	0.0394(9)
H16	0.0000	0.3904	0.2500	0.047*
C17	0.0371(4)	0.6280(3)	0.28584(14)	0.0843(17)
H17	0.0622	0.6651	0.3098	0.101*
C18	0.0000	0.6831(6)	0.2500	0.0842(19)
H18	0.0000	0.7568	0.2500	0.101*
C19	0.1251(3)	−0.1312(4)	0.40545(19)	0.0801(16)
C20	0.0797(2)	−0.0335(3)	0.38638(12)	0.0466(8)
C21	0.0070(2)	−0.0508(3)	0.36027(15)	0.0570(10)
H21	−0.0128	−0.1198	0.3564	0.068*
C22	−0.03807(19)	0.0325(3)	0.33928(11)	0.0396(7)
C23	0.1333(4)	0.1736(6)	0.48481(16)	0.1012(19)
H23A	0.1660	0.2360	0.4919	0.152*
H23B	0.1690	0.1222	0.4709	0.152*
H23C	0.1114	0.1445	0.5119	0.152*
C24	0.0054(4)	0.2744(5)	0.47369(17)	0.0876(16)
H24A	0.0310	0.3443	0.4759	0.105*
H24B	−0.0092	0.2516	0.5036	0.105*
C25	−0.0715(3)	0.2772(5)	0.4437(2)	0.0909(17)
H25A	−0.1004	0.2093	0.4442	0.109*
H25B	−0.1105	0.3312	0.4538	0.109*
C26	−0.1165(3)	0.3157(5)	0.3684(2)	0.0860(15)
H26A	−0.1503	0.2523	0.3671	0.129*
H26B	−0.0962	0.3312	0.3390	0.129*
H26C	−0.1506	0.3736	0.3785	0.129*

Occupancies: [a] = 0.59; [b] = 0.41; [c] = 0.55, [d] = 0.45

was added, and the mixture was allowed to stir for 24 h at ambient temperature. Water was then added to this mixture, and the precipitate thus formed was filtered, washed with water, and dried in air to give the title compound in a yield of 62.1%. Crystals were obtained in about two weeks by recrystallization from DME/hexane.

Experimental details

All H atoms on C atoms were placed in idealized positions [C—H = 0.96 (methyl), 0.97 (methylene) and 0.93 Å (aromatic)] and included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.5U_{eq}$(methyl C) and $1.2U_{eq}$(methylene and aromatic C). The F4, F5 and F6 atoms are disordered (*cf.* table 2). The non-hydrogen atoms in the structure of the title complex were refined with anisotropic displacement parameters.

Discussion

Lutetium(III) complexes have wide range applications in catalysis in the past few years [4–6]. Some directly related structures of the title structure have been reported [3, 7–8]. In the title crystal structure, the bond distances and bond angles are all in normal range [9]. The crystal structure features weak intermolecular C—H···F and C—H···O hydrogen bonds.

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