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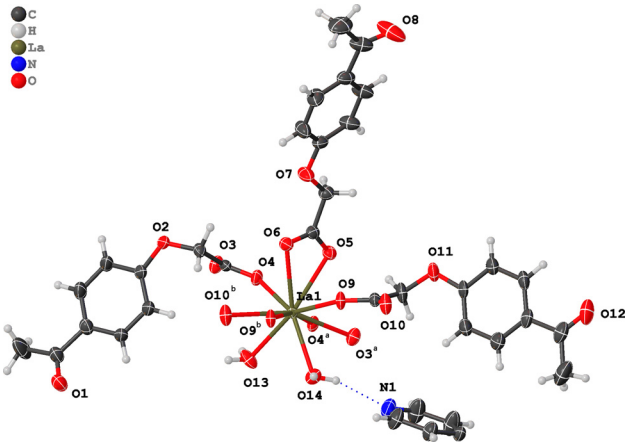
The crystal structure of *catena*-[diaqua-(4-acetylphenoxyacetato-κ^2O,O)-bis(4-acetylphenoxyacetato-$\kappa^3O,O:O$)-dihydrate-lanthanum(III)]–4,4′-bipyridine (2/1), $C_{35}H_{35}NO_{14}La$



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Abstract

$C_{35}H_{35}NO_{14}La$, triclinic, $P\overline{1}$ (no. 2), a = 9.06760(10) Å, b = 13.6139(2) Å, c = 15.5964(2) Å, α = 115.2310(10)°, β = 97.4010(10)°, γ = 95.4830(10)°, V = 1702.65(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0431, $wR_{ref}(F^2)$ = 0.1059, T = 296.13(10) K.

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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.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	10.3 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	66.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20,535, 6027, 0.086
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5689
$N(param)_{refined}$:	466
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

1 Source of materials

The synthesis of La(III) complex proceeds as following method: 0.1942 g 4-acetylphenoxyacetic acid (1.0 mmol), 0.1767 g $LaCl_3{\cdot}6H_2O$ (0.50 mmol), 0.0781 g 4,4′-bipyridine (0.50 mmol), and 0.040 g NaOH (1.0 mmol) were added to 25 mL of ethanol-water solution (v:v = 3:2) with stirring. The mixture immediately became cloudy, and continued to react at 75 °C for 5 h. After cooling to room temperature, it is filtered, and the filtrate remains still placed. The colourless block crystals of the title La(III) complex were obtained in 22 days.

2 Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.97 Å, O–H = 0.85–0.86 Å). Their U_{iso} values were set to 1.2U_{eq} or 1.5U_{eq} of the parent atoms.

3 Comment

The synthesis and properties of lanthanum(III) complexes have been of chemical interest, since they show excellent properties in antibacterial activity [5], catalysis for cycloaddition of di-substituted epoxides and CO_2 [6],

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1419 (5)	0.0841 (4)	0.6624 (3)	0.0348 (10)
C2	0.4679 (6)	0.4837 (4)	0.0347 (3)	0.0348 (10)
C3	−0.0142 (5)	0.2351 (4)	0.6426 (3)	0.0332 (10)
H3	0.035286	0.260429	0.605409	0.040*
C4	−0.0382 (5)	0.3086 (4)	0.7318 (3)	0.0283 (9)
C5	−0.1217 (6)	0.2705 (4)	0.7835 (4)	0.0427 (12)
H5	−0.141810	0.319931	0.842141	0.051*
C6	−0.1746 (6)	0.1596 (4)	0.7481 (4)	0.0427 (12)
H6	−0.233106	0.135119	0.782157	0.051*
C7	−0.1735 (6)	−0.0364 (4)	0.6306 (4)	0.0380 (10)
C8	−0.2797 (8)	−0.0798 (5)	0.6772 (6)	0.073 (2)
H8A	−0.303688	−0.158576	0.641933	0.109*
H8B	−0.370546	−0.048920	0.676891	0.109*
H8C	−0.233076	−0.059750	0.742493	0.109*
C9	0.0941 (5)	0.4635 (3)	0.7237 (3)	0.0273 (8)
H9A	0.162746	0.415548	0.691842	0.033*
H9B	0.153561	0.534735	0.767867	0.033*
C10	−0.0147 (4)	0.4769 (3)	0.6486 (3)	0.0236 (8)
C11	0.3149 (4)	0.7031 (3)	0.6507 (3)	0.0255 (8)
C12	0.3619 (5)	0.8198 (4)	0.7278 (3)	0.0315 (9)
H12A	0.404309	0.865586	0.700465	0.038*
H12B	0.274546	0.848268	0.752905	0.038*
C13	0.5544 (5)	0.9232 (4)	0.8672 (3)	0.0354 (10)
C14	0.6763 (7)	0.9180 (4)	0.9272 (4)	0.0494 (13)
H14	0.694867	0.850324	0.923149	0.059*
C15	0.7706 (6)	1.0141 (5)	0.9933 (4)	0.0483 (12)
H15	0.851530	1.010169	1.034292	0.058*
C16	0.7469 (5)	1.1165 (4)	0.9999 (3)	0.0392 (11)
C17	0.6228 (6)	1.1189 (4)	0.9387 (4)	0.0449 (12)
H17	0.604634	1.186203	0.941519	0.054*
C18	0.5257 (6)	1.0237 (4)	0.8739 (4)	0.0427 (12)
H18	0.441600	1.027378	0.835137	0.051*
C19	0.9689 (7)	1.2234 (6)	1.1442 (4)	0.0652 (17)
H19A	1.038169	1.291600	1.170830	0.098*
H19B	1.022169	1.163024	1.116797	0.098*
H19C	0.923193	1.217278	1.194315	0.098*
C20	0.8482 (6)	1.2208 (5)	1.0669 (4)	0.0505 (14)
C21	0.5401 (4)	0.6472 (3)	0.4444 (3)	0.0243 (8)
C22	0.4375 (5)	0.7158 (4)	0.4193 (4)	0.0362 (10)
H22A	0.389577	0.675613	0.351858	0.043*
H22B	0.358821	0.727068	0.457526	0.043*
C23	0.5870 (5)	0.8301 (4)	0.3677 (3)	0.0316 (10)
C24	0.6590 (5)	0.9359 (4)	0.3927 (4)	0.0374 (10)
H24	0.654183	0.993298	0.451763	0.045*
C25	0.7376 (5)	0.9563 (4)	0.3305 (4)	0.0377 (10)
H25	0.785780	1.027308	0.347961	0.045*
C26	0.7454 (5)	0.8707 (4)	0.2412 (3)	0.0382 (11)
C27	0.5931 (6)	0.7442 (4)	0.2798 (4)	0.0409 (11)
H27	0.545767	0.673054	0.262820	0.049*
C28	0.6716 (6)	0.7664 (5)	0.2173 (4)	0.0438 (12)
H28	0.674481	0.709240	0.157623	0.053*
C29	0.8324 (6)	0.8961 (5)	0.1766 (4)	0.0470 (13)
C30	0.8447 (8)	0.8033 (7)	0.0832 (5)	0.0706 (19)
H30A	0.746886	0.774606	0.042242	0.106*
H30B	0.882915	0.746109	0.095250	0.106*
H30C	0.912125	0.829514	0.051957	0.106*
C31	0.2725 (7)	0.4665 (6)	0.1193 (5)	0.0627 (17)
H31	0.176248	0.478198	0.131182	0.075*
C32	0.3258 (7)	0.4992 (6)	0.0548 (5)	0.0613 (17)
H32	0.265793	0.531583	0.024849	0.074*
C33	−0.0641 (5)	0.1240 (4)	0.6092 (3)	0.0329 (10)
H33	−0.045413	0.074645	0.550041	0.039*
C34	0.5486 (6)	0.4340 (5)	0.0823 (4)	0.0437 (11)
H34	0.645012	0.421078	0.071762	0.052*
C35	0.4857 (6)	0.4038 (5)	0.1454 (4)	0.0516 (13)
H35	0.542294	0.370254	0.175865	0.062*
La1	0.24448 (2)	0.47768 (2)	0.48636 (2)	0.01943 (12)
N1	0.3492 (6)	0.4198 (4)	0.1654 (3)	0.0498 (11)
O1	−0.1120 (4)	−0.0992 (3)	0.5698 (3)	0.0456 (8)
O2	0.0200 (4)	0.4187 (2)	0.7774 (2)	0.0334 (7)
O3	−0.1517 (3)	0.4673 (3)	0.6492 (2)	0.0326 (7)
O4	0.0399 (3)	0.5028 (3)	0.5891 (2)	0.0299 (6)
O5	0.2229 (3)	0.6875 (2)	0.5763 (2)	0.0293 (6)
O6	0.3696 (3)	0.6257 (2)	0.6599 (2)	0.0290 (6)
O7	0.4704 (4)	0.8237 (3)	0.8035 (2)	0.0445 (8)
O8	0.8368 (6)	1.3057 (4)	1.0591 (4)	0.0827 (16)
O9	0.4850 (3)	0.5850 (2)	0.4778 (2)	0.0298 (6)
O10	0.6731 (3)	0.6523 (3)	0.4340 (3)	0.0376 (7)
O11	0.5147 (4)	0.8203 (3)	0.4358 (3)	0.0390 (7)
O12	0.8896 (5)	0.9897 (4)	0.1976 (3)	0.0627 (11)
O13	0.0700 (3)	0.2935 (2)	0.4161 (3)	0.0396 (7)
H13A	−0.015205	0.302944	0.433903	0.059*
H13B	0.104598	0.253082	0.440932	0.059*
O14	0.3182 (3)	0.3605 (3)	0.3231 (2)	0.0341 (7)
H14A	0.412365	0.358901	0.331834	0.051*
H14B	0.306277	0.392443	0.286804	0.051*

mull-responsive chemsensor for detection of different pollutants [7], nematicidal activity [8], gas adsorption property [9], corrosion inhibiting property [10], etc. Our research group has also synthesized and structurally characterized some lanthanum(III) complexes [11–13].

Figure 1 shows the coordination environment of the title complex. The asymmetric unit of the title complex is composed of one lanthanum(III) ion, three 4-acetylphenoxyacetate ligands, two coordinated water molecules, and half uncoordinated 4,4′-bipyridine. As shown in Figure 1, the lanthanum(III) ion is ten-coordinated and surrounded by eight carboxylate oxygen atoms (O3^a, O4^a, O4, O5, O6, O9, O9^b, O10^b) from three 4-acetylphenoxyacetate ligands, and two oxygen atoms (O13, O14) of two coordinated water molecules, respectively. The 4-acetylphenoxyacetate ligand adopt bidentate or tridentate coordination modes: one is that the two oxygen atoms of the

carboxyl group are coordinated with the same La(III) ion, the other is that the two oxygen atoms of the carboxyl group are coordinated with the same La(III) ion, and then one oxygen atom is coordinated with a different La(III) ion, which is different from ref. [14]. The bond lengths of La–O are 2.539(3)–2.778(3) Å, and the coordination angles around La(III) ion are in the range of 47.49(8)–166.88(10)°. The complex molecules form 1D infinite structure by the oxygen atoms of the tridentate carboxyl groups with a La···La distance of 4.581 Å [15]. Although 4,4-bipyridine does not involved in coordination, however, it plays an important role in stabilizing the structure of the complex and in the formation of O–H···N hydrogen bonds.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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