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Crystal structure of 2, 3-bis((4-methylbenzoyl)oxy) succinic acid–*N, N*-dimethylformamide (1/1), $C_{23}H_{25}NO_9$

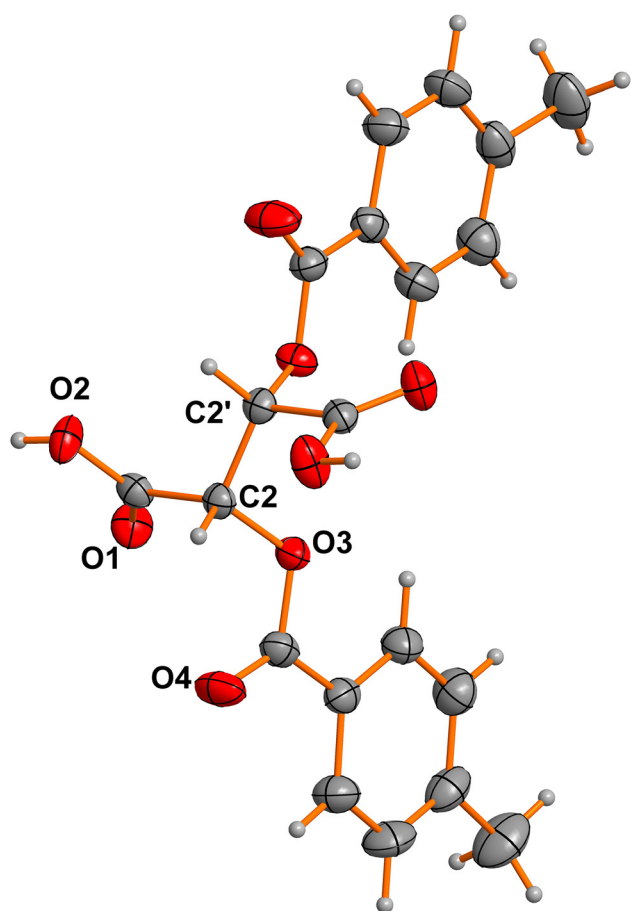


Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.20 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18108, 4085, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3477
$N(\text{param})_{\text{refined}}$:	304
Programs:	Bruker [1], SHELX [2–4]

Abstract

$C_{23}H_{25}NO_9$, orthorhombic, $P2_12_12$ (no. 18), $a = 15.8641(6)$ Å, $b = 11.1230(4)$ Å, $c = 13.0925(5)$ Å, $V = 2310.26(15)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0372$, $wR_{\text{ref}}(F^2) = 0.0883$, $T = 298$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were of analytical reagent grade and used without further purification. $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.06 mmol) was dissolved in the mixture of *N,N*-dimethylformamide (DMF) (2 mL) and water (2 mL), and (2*S*, 3*S*)-2,3-bis((4-methylbenzoyl)oxy) succinic acid (0.09 mmol) was dissolved in water (2 mL), then mixed together adjusting the pH of the solution to 5 with KOH (1 M), then stayed the solution at 298 K in a tube. Colorless prismatic crystals of the title compound were obtained after a few weeks in 20% yield. Surprisingly the La ions are not included.

Experimental details

Coordinates of hydrogen atoms were constrained using a riding model. Their U_{iso} values were set to $1.2U_{\text{eq}}$ of the parent atoms.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.62240 (16)	0.4941 (3)	0.19183 (19)	0.0339 (6)
C2	0.53158 (15)	0.4492 (2)	0.19493 (18)	0.0311 (6)
H2	0.5213	0.3974	0.1357	0.037*
C3	0.55159 (17)	0.2672 (2)	0.2854 (2)	0.0383 (6)
C4	0.55833 (16)	0.2144 (2)	0.3877 (2)	0.0374 (6)
C5	0.54128 (17)	0.2783 (3)	0.4762 (2)	0.0453 (7)
H5	0.5209	0.3565	0.4719	0.054*
C6	0.5544 (2)	0.2263 (3)	0.5710 (2)	0.0537 (8)
H6	0.5419	0.2699	0.6297	0.064*
C7	0.58575 (19)	0.1107 (3)	0.5796 (2)	0.0526 (8)
C8	0.6015 (2)	0.0476 (3)	0.4913 (3)	0.0578 (9)
H8	0.6215	−0.0308	0.4960	0.069*
C9	0.5886 (2)	0.0972 (3)	0.3963 (3)	0.0515 (8)
H9	0.6001	0.0525	0.3378	0.062*
C10	0.6043 (3)	0.0555 (4)	0.6826 (3)	0.0763 (12)
H10A	0.5796	−0.0231	0.6861	0.115*
H10B	0.5810	0.1053	0.7354	0.115*
H10C	0.6642	0.0493	0.6917	0.115*
C11	0.92455 (18)	−0.1348 (3)	0.1047 (2)	0.0389 (6)
C12	0.95257 (16)	−0.0066 (2)	0.12677 (18)	0.0343 (6)
H12	0.9296	0.0457	0.0734	0.041*
C13	0.84854 (16)	0.0968 (2)	0.2255 (2)	0.0343 (6)
C14	0.82866 (16)	0.1350 (2)	0.3310 (2)	0.0346 (6)
C15	0.78141 (18)	0.2390 (3)	0.3457 (2)	0.0431 (7)
H15	0.7594	0.2802	0.2899	0.052*
C16	0.76726 (18)	0.2810 (3)	0.4437 (2)	0.0480 (7)
H16	0.7368	0.3517	0.4528	0.058*
C17	0.79725 (17)	0.2206 (3)	0.5282 (2)	0.0455 (7)
C18	0.84065 (18)	0.1135 (3)	0.5126 (2)	0.0474 (7)
H18	0.8588	0.0692	0.5687	0.057*
C19	0.85718 (19)	0.0718 (3)	0.4153 (2)	0.0423 (7)
H19	0.8876	0.0011	0.4064	0.051*
C20	0.7859 (2)	0.2703 (4)	0.6343 (2)	0.0674 (10)
H20A	0.8353	0.3147	0.6536	0.101*
H20B	0.7774	0.2053	0.6814	0.101*
H20C	0.7377	0.3225	0.6356	0.101*
C21	0.8836 (4)	0.4288 (4)	0.8909 (3)	0.1003 (17)
H21A	0.8429	0.3832	0.8529	0.150*
H21B	0.9312	0.3788	0.9064	0.150*
H21C	0.9018	0.4964	0.8509	0.150*
C22	0.8190 (3)	0.3800 (3)	1.0582 (3)	0.0857 (13)
H22A	0.8084	0.4169	1.1232	0.129*
H22B	0.8626	0.3208	1.0653	0.129*
H22C	0.7684	0.3422	1.0340	0.129*
C23	0.83352 (18)	0.5851 (3)	1.0032 (2)	0.0455 (7)
H23	0.8535	0.6394	0.9549	0.055*
N1	0.84570 (16)	0.4713 (2)	0.98548 (19)	0.0502 (6)
O1	0.67437 (12)	0.46938 (19)	0.25434 (14)	0.0492 (5)
O2	0.63548 (12)	0.5615 (2)	0.10986 (16)	0.0503 (6)
H2A	0.6858	0.5778	0.1055	0.076*
O3	0.51997 (11)	0.38104 (15)	0.28637 (13)	0.0359 (4)
O4	0.57251 (17)	0.2197 (2)	0.20684 (15)	0.0638 (7)
O5	0.96276 (16)	−0.1973 (2)	0.0466 (2)	0.0787 (8)
O6	0.85445 (14)	−0.1647 (2)	0.14914 (19)	0.0592 (6)
H6A	0.8361	−0.2265	0.1233	0.089*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O7	0.92193 (11)	0.03483 (16)	0.22349 (13)	0.0362 (4)
O8	0.80891 (14)	0.1204 (2)	0.14990 (15)	0.0556 (6)
O9	0.79729 (13)	0.62689 (18)	1.07963 (16)	0.0478 (5)

Comment

Tartaric acid is one of the common chiral and small molecules, which is relatively cheap and easy to obtain. Employing this chiral organic molecule and related deprotonated anions as ligands, novel and chiral metal organic complexes can be constructed. Compared with tartaric acid, tartaric acid derivatives have more functional groups, which construct novel and multifunctional metal organic complexes [5, 6]. Here, the chiral tartaric acid derivative, named (2*S*, 3*S*)-2, 3-bis((4-methylbenzoyl)oxy) succinic acid, was chosen. It is a versatile ligand with various coordination modes, such as monodentate, chelated bidentate, bridge bidentate, etc. So the complexes constructed from this chiral ligand have diverse structures like chain, layer and network, and significant potential applications in optics, electricity, magnetism, etc. [6–8].

We anticipated to build La-organic frameworks using (2*S*, 3*S*)-2, 3-bis((4-methylbenzoyl)oxy) succinic acid, but we got the title compound unexpectedly. The asymmetric unit of the title complex contains one 2, 3-bis((4-methylbenzoyl)oxy) succinic acid molecule (distributed over two halves, located around twofold axis) and one *N,N*-dimethylformamide solvent. One C₂-symmetric title molecule is shown in the figure. Bond lengths and angles are all in the expected ranges [9, 10]. There are abundant weak interactions in the structure. Firstly, a variety of weak hydrogen bonds exist. The *N,N*-dimethylformamide and 2, 3-bis((4-methylbenzoyl)oxy) succinic acid were linked together through O2–H6A⋯O9ⁱ ((i) *x*, *y*−1, *z*−1) and O6–H2⋯O9ⁱⁱ ((ii) *x*, *y*, *z*−1) hydrogen bonds. Secondly, π⋯π interactions between aromatic rings are also contained in the structure. The geometry analysis shows a face-to-face alignment of the aromatic-aromatic rings. The distance between Cg1 ring (C4–C9) and Cg2 (C14–C19) ring is 3.8856(13) Å, and the dihedral angle between two rings in two planes is 3.4°.

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

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