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(*R*)-2,2′-Bis(methoxymethoxy)-1,1′-binaphthalenyl-3-carbaldehyde

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The title compound, $C_{25}H_{22}O_5$, was obtained from (*R*)-2,2′-bis(methoxymethoxy)-1,1′-binaphthalenyl by *ortho*-lithiation and quenching with *N*,*N*-dimethylformaldehyde. The molecular packing in the crystal structure is stabilized by weak C—H···O hydrogen-bonding interactions.

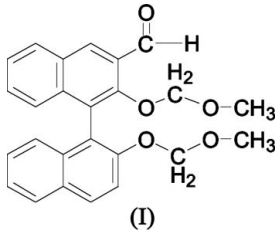
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.120
Data-to-parameter ratio = 7.7

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

Comment

The title compound, (I), is an intermediate of chiral salicylaldehyde derivatives, for subsequent conversion to chiral Schiff base used in enantioselective sulfoxidation (Zeng, Wang, Wang *et al.*, 2005; Zeng, Wang, Weng *et al.*, 2005). Bond lengths and angles are in agreement with values reported for a similar compound (Tachi *et al.*, 1999). The dihedral angle between the planes of the naphthalene ring systems is 74.92 (6) Å.



(I)

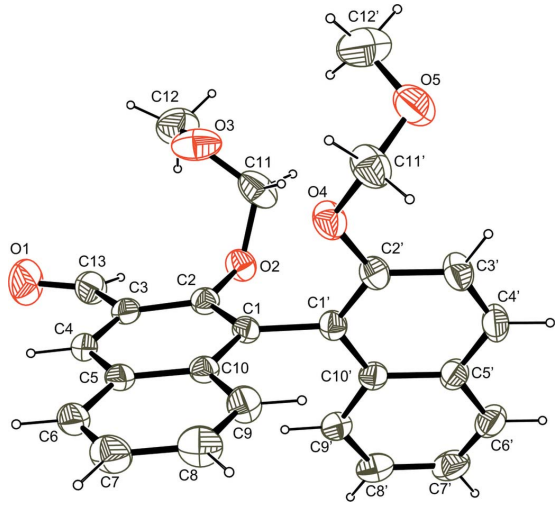


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

The molecular packing in the crystal structure is stabilized by weak C—H···O hydrogen-bond interactions (Table 1 and Fig.1).

Experimental

To a solution of (*R*)-2,2′-bis(methoxymethoxy)-1,1′-binaphthalenyl (4.30 g, 11 mmol) in dry tetrahydrofuran (40 ml) in an ice–salt bath, an LiBu (7 ml, 1.6 *M*) solution in hexane was added. To the resulting slurry in the ice–salt bath, *N*,*N*-dimethylformaldehyde (0.9 ml, 12 mmol) was added after 3 h. After a further 3 h, a saturated aqueous NH_4Cl solution was added to quench the reaction. The organic layer was extracted with ethyl acetate, dried over anhydrous $MgSO_4$ and concentrated under vacuum. The residual liquid was purified by column chromatography to give 1.74 g of the title compound in 39% yield. Single crystals were obtained from a petroleum ether and ethyl acetate (10:1) solution by slow evaporation.

Crystal data

$C_{25}H_{22}O_5$
M_r = 402.43
Orthorhombic, $P2_12_12_1$
a = 9.102 (2) Å
b = 12.322 (3) Å
c = 18.542 (4) Å
V = 2079.6 (8) Å^3
Z = 4
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5357 reflections
θ = 2.2–27.3°
μ = 0.09 mm^{-1}
T = 273 (2) K
Block, colorless
0.45 × 0.45 × 0.30 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.959, T_{max} = 0.978
10490 measured reflections
2103 independent reflections
1991 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°
$h = -9 \rightarrow 10$
$k = -14 \rightarrow 13$
$l = -22 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.120
S = 1.15
2103 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.3987P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2^{i}	0.93	2.58	3.283 (4)	133
C12—H12C···O1^{ii}	0.96	2.55	3.354 (6)	142

Symmetry codes: (i) $x-1, y, z$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

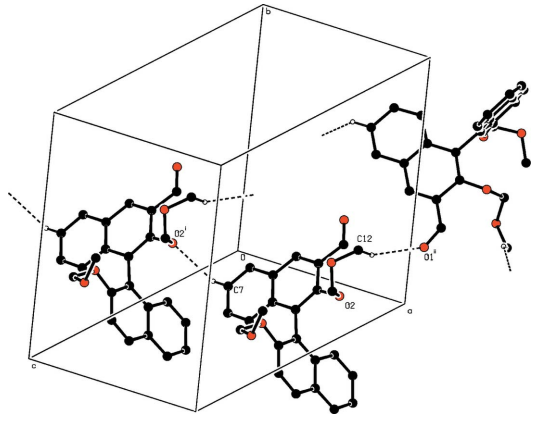


Figure 2
Packing view, showing the weak C—H···O hydrogen-bond interactions (dashed lines) linking the molecules. [Symmetry codes: (i) $x-1, y, z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.]

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent C atoms, with C—H distances of 0.95 (aromatic), 0.97 (CH_2) and 0.98 Å (CH_3), with U_{iso}(H) = 1.2$_{eq}$(aromatic and CH_2) or 1.5U_{eq}(CH_3). In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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