Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-{[3′-Formyl-2,2′-bis(methoxymethoxy)biphenyl-3-yl]methyl}formamide

Yu-Xing Gao, Hua Fang, Qing-Le Zeng,* Wei-Zhu Chen and Yu-Fen Zhao

Department of Chemistry, The Key Laboratory for Chemical Biology of Fujian Province, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, People's Republic of China

Correspondence e-mail: qlzeng@xmu.edu.cn

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.066
wR factor = 0.207
Data-to-parameter ratio = 13.5

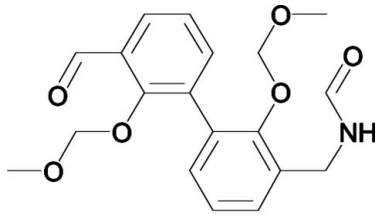
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 March 2006
Accepted 28 March 2006

The title compound, $C_{19}H_{21}NO_6$, was obtained by *ortho*-lithiation of 2,2′-bis(methoxymethoxy)biphenyl, followed by quenching with *N*,*N*-dimethylformaldehyde. The molecular packing is stabilized by van der Waals forces.

Comment

The title compound, (I), is the intermediate of a chiral salicylaldehyde, which can be converted into a chiral Schiff base used in enantioselective sulfoxidation (Zeng, Wang, Wang *et al.*, 2005; Zeng, Wang, Weng *et al.*, 2005). Bond lengths and angles in (I) are in agreement with values reported for a similar compound (Chen *et al.*, 2006). The dihedral angle between the planes of the benzene rings C6–C1 and C14–C19 is 62.8 (4)°.



(I)

Experimental

To a solution of 2,2′-bis(methoxymethoxy)biphenyl (4.12 g, 15 mmol) in dry tetrahydrofuran (35 ml) in an ice–salt bath, a 1.6 *M* LiBu solution (11 ml) was added. After 3 h, *N*,*N*-dimethylformaldehyde (5 ml) was added to the mixture. After another 3 h, the reaction was

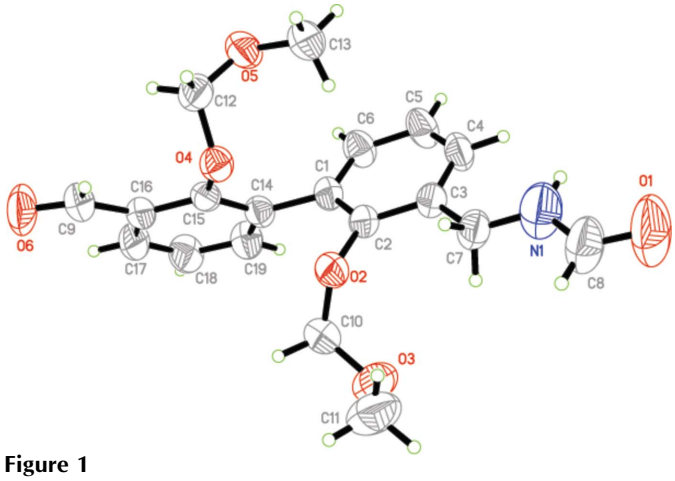


Figure 1
A plot of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

quenched by adding saturated NH_4Cl. 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 the title compound (62% yield). Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a petroleum ether–ethyl acetate solution (5:1 *v*/*v*).

Crystal data

$C_{19}H_{21}NO_6$	V = 917.3 (6) Å^3
M_r = 359.37	Z = 2
Triclinic, $P\overline{1}$	D_x = 1.301 Mg m^{-3}
a = 8.101 (3) Å	Mo $K\alpha$ radiation
b = 9.640 (3) Å	μ = 0.10 mm^{-1}
c = 12.419 (5) Å	T = 273 (2) K
α = 90.588 (7)°	Block, colourless
β = 101.626 (6)°	0.48 × 0.25 × 0.11 mm
γ = 104.636 (6)°	

Data collection

Bruker APEX area-detector diffractometer	4642 measured reflections
	3178 independent reflections
φ and ω scans	2485 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.017
	θ_{max} = 25.0°
T_{min} = 0.955, T_{max} = 0.989	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1084P)^2 + 0.3792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.066	
$wR(F^2)$ = 0.207	
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
3178 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
235 parameters	$\Delta\rho_{min}$ = −0.41 e Å^{-3}
H-atom parameters constrained	

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–0.98 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) for aromatic, methylene and imine H atoms, or 1.5U_{eq}(C) for methyl H atoms.

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: *ORTEP-3* (Farrugia, 1997) and *VIEWERPRO* (Accelrys, 2001); software used to prepare material for publication: *SHELXL97*.

This project was supported by the Key (Key Grant) Project of the Chinese Ministry of Education (grant No. 104201), the Natural Science Foundation of Fujian Province of China (grant No. C0310002) and the China Postdoctoral Science Foundation.

References

Accelrys (2001). *VIEWERPRO* Version 4.2. Accelrys Inc., Burlington, Massachusetts, USA.
Bruker (2001). *SAINT* (Version 6.22), *SMART* (Version 5.625) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, W.-Z., Zeng, Q.-L., Fang, H., Gao, Y.-X. & Zhao, Y.-F. (2006). *Acta Cryst.* E**62**, o869–o870.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zeng, Q.-L., Wang, H.-Q., Wang, T.-J., Cai, Y.-M., Weng, W. & Zhao, Y.-F. (2005). *Adv. Synth. Catal.* **347**, 1933–1936.
Zeng, Q.-L., Wang, H.-Q., Weng, W., Lin, W.-S., Gao, Y.-X., Huang, X.-T. & Zhao, Y.-F. (2005). *New J. Chem.* **29**, 1125–1127.