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4,4′-Bis[2,2-bis(4-methoxyphenyl)ethenyl]biphenyl

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 10.7

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

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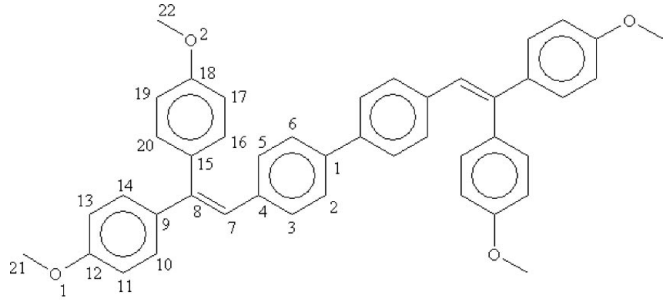
The molecule of 4,4′-bis[2,2-bis(4-methoxyphenyl)ethenyl]biphenyl, $C_{44}H_{38}O_4$, has a crystallographic center of symmetry at the midpoint of the biphenyl single bond, resulting in an asymmetric unit of one half-molecule. The geometry of the structure is as expected.

Comment

4,4′-Bis[2,2-bis(4-methoxyphenyl)ethenyl]biphenyl, (I), is a new product, prepared as part of our continuing research into materials for use as emitters in organic LEDs (Yang, Jin *et al.*, 2000; Yang, Heremans *et al.*, 2000). The structure contains a planar biphenyl moiety, which lies on a center of symmetry. This is a known artefact in structure determinations of biphenyl-containing materials, attributable to time-averaging of the librating rings (Lenstra *et al.*, 1994). All bond distances and angles are as expected. Relevant torsion angles are summarized in Table 1.



(I)

Experimental

4,4′-Bis[2,2-bis(4-methoxyphenyl)ethenyl]biphenyl, (I), was synthesized *via* a Horner reaction, starting from 4,4′-dimethoxybenzophenone and 4,4′-bis(diphenylphosphorylmethyl)biphenyl; the latter was prepared by a Michaelis–Arbuzov reaction starting from commercially available diphenylethoxyphosphine and 4,4′-bis(chloromethyl)biphenyl. 4,4′-Bis(diphenylphosphorylmethyl)biphenyl was synthesized by refluxing a solution of 5 g (0.02 mol) 4,4′-bis(chloromethyl)biphenyl and 10 g (0.04 mol) diphenylethoxyphosphine in 100 ml dimethylformamide (DMF) for 15 h. After the mixture cooled, the white precipitate was filtered off and washed with DMF and a small amount of diethyl ether. Purification was achieved by stirring the precipitate overnight in a hexane–acetone mixture. The yield was 8.3 g (71%) and m.p. > 623 K. MS (CI: NH_3): m/z = 583 (MH^+).

4,4′-Bis[2,2-bis(4-methoxyphenyl)ethenyl]biphenyl, (I), was synthesized by adding a solution of 3,4 g (0.014 mol) 4,4′-dimethoxybenzophenone in 170 ml of freshly dried benzene dropwise to a stirred slurry of 4 g (0.007 mol) of 4,4′-bis(diphenylphosphorylmethyl)biphenyl and 1.6 g (0.014 mol) of potassium *tert*-butoxide in

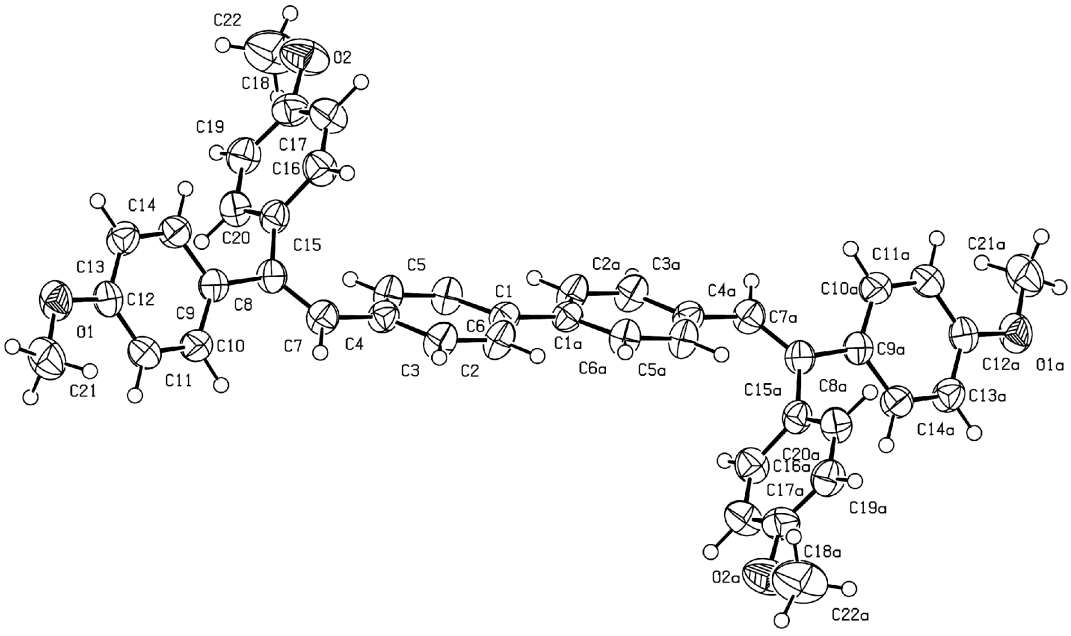


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

500 ml of benzene at reflux temperature and under a nitrogen atmosphere. After the addition, the mixture was kept under reflux and stirred for 20 h. After cooling to room temperature, any solid residue was filtered off and the clear filtrate was washed with 5% aqueous HCl and water. The solvent was removed under reduced pressure and the light green solid (dissolved in chloroform) was separated from impurities by prep. NP–HPLC (90/10 hexane/ethyl acetate). The yield was 0.65 g (14%) and m.p. = 483–485 K. MS (CI: NH_3): m/z = 631 (MH^+). Crystals suitable for X-ray diffraction were grown by evaporation of a saturated acetone solution.

Crystal data

$C_{44}H_{38}O_4$	$Z = 1$
$M_r = 630.74$	$D_x = 1.249$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.748$ (2) Å	Cell parameters from 25 reflections
$b = 8.792$ (3) Å	$\theta = 10$–$17°$
$c = 12.410$ (5) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 92.51$ (3)°	$T = 293$ (2) K
$\beta = 90.13$ (3)°	Prism, light yellow–green
$\gamma = 96.91$ (3)°	0.3 × 0.3 × 0.3 mm
$V = 838.4$ (5) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 25.0°$
$\omega/2\theta$ scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -10 \rightarrow 10$
5886 measured reflections	$l = -14 \rightarrow 14$
2943 independent reflections	3 standard reflections
2138 reflections with $I > 2\sigma(I)$	frequency: 7200 min
$R_{int} = 0.021$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.1052P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.102$	$\Delta\rho_{max} = 0.11$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
2943 reflections	
274 parameters	
All H-atom parameters refined	

Table 1
Selected geometric parameters (°).

C2—C1—C1^i—C2^i	180.00 (15)	C7—C8—C15—C16	−63.1 (2)
C3—C4—C7—C8	150.00 (17)	C7—C8—C9—C10	−24.7 (2)

Symmetry code: (i) $2-x, 2-y, -z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software* and *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2001); software used to prepare material for publication: *SHELXL*97.

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