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Key indicators

Single-crystal X-ray study
$T = 100$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.106
Data-to-parameter ratio = 17.5

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

2,5-Bis(2,2-diphenylethenyl)thiophene

The ethene units of the title compound, $C_{32}H_{24}S$, a possible candidate for Organic Light-Emitting Devices, are tilted with respect to the thiophene unit by 6.2 (1) and 9.2 (1)°.

Comment

2,5-Bis(2,2-diphenylethenyl)thiophene, (I), is a new derivative of 1,4-bis(2,2-diphenylethenyl)benzene which has been successfully used as the optically active material in a single-layer white light (Yang, Jin *et al.*, 2000) and a single-layer blue light Organic Light-Emitting Device (OLED) (Yang, Heremans *et al.*, 2000). It was synthesized in a modified Horner–Wittig reaction from 2,5-bis(chloromethyl)thiophene (II) *via* 2,5-bis(diphenylphosphorylmethyl)thiophene (III).
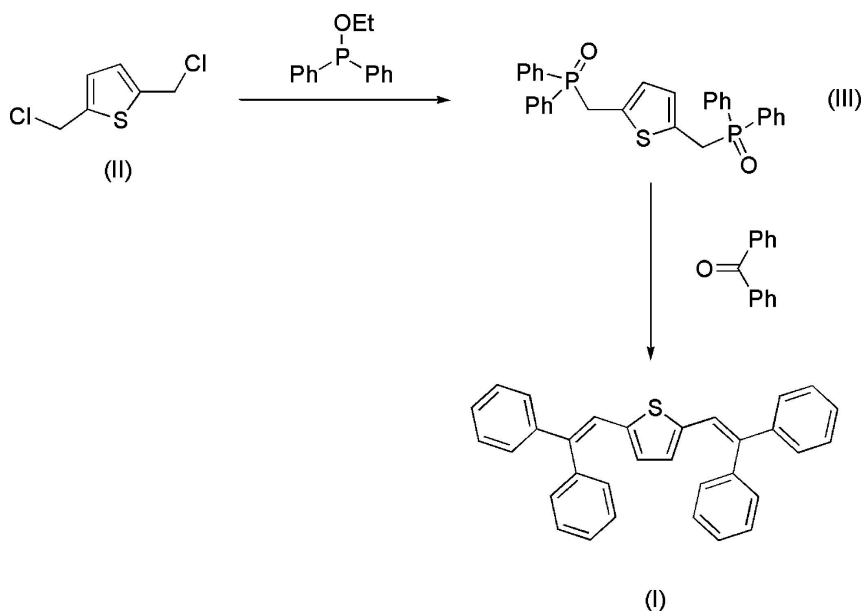
The thiophene (I) crystallizes in the monoclinic space group *C*2/*c* with one molecule in the asymmetric unit. Chemically equivalent atoms (indicated by *A* and *B* in the numbering scheme) are not related by crystallographic symmetry. Both the thiophene and the ethene units are each essentially planar, the largest deviation being 0.0364 (7) Å for C4*A* with respect to the thiophene unit defined by S1, C2*A*, C2*A*, C3*A*, C3*B*, C4*A* and C4*B*. The ethene units (C12*A*, C6*A*, C5*A*, C4*A*, and C12*B*, C6*B*, C5*B*, C4*B*) and the thiophene unit are slightly tilted with respect to each other, the respective dihedral angles being 6.2 (1)° and 9.2 (1)°. The dihedral angles formed by the ethene units with the phenyl rings are 72.39 (4) and 43.60 (7)° for the *A*-labeled unit and 68.39 (5) and 19.54 (9)° for the *B*-labeled unit.

The closest intermolecular contact present is between carbon atom C2A of the thiophene unit and hydrogen atom H10A^i of a neighboring benzene ring [C2A $\cdots$ H10A^i = 2.67 Å, C2A$\cdots$H10A^i—C10A^i = 165°, symmetry code (i): $\frac{1}{2}$ − x, $-\frac{1}{2}$ + y, $\frac{1}{2}$ − z]. No other significant intermolecular contacts are observed. The bond lengths and angles of (I) are unexceptional and within their expected ranges.

Experimental

2,5-Bis(2,2-diphenylethenyl)thiophene (I) was prepared from 2,5-bis(chloromethyl)thiophene (II), which was synthesized according to the procedure of Takeshita & Tashiro (1991). 2,5-Bis(diphenylphosphorylmethyl)thiophene (III) was prepared by refluxing a solution of (II) (1.6 g, 0.0009 mol) and diphenylethoxyphosphine (5 g, 0.02 mol) in DMF (70 ml) for 15 h. After the red mixture had cooled to room temperature the solvent was evaporated under reduced pressure and the orange solid was dried. The yield was quantitative [m.p. 477–481 K (uncorrected)]. MS (CI, NH$_3$): m/z 513 (MH^+), 313 ($[M - P(O)Ph_2]^+$). 2,5-Bis(2,2-diphenylethenyl)thiophene (I) was prepared by dropwise addition of a solution of benzophenone (3.8 g, 0.021 mol) in freshly dried benzene (70 ml) to a stirred slurry of (III) (5.3 g, 0.010 mol) and potassium *tert*-butoxide (2.4 g, 0.021 mol) in benzene (300 ml) at reflux temperature and under a nitrogen atmosphere. After the addition the mixture was heated to reflux and stirred for 20 h. After the mixture had cooled it was filtered and the filtrate was washed with aqueous HCl (5%) and water. The solvent was then removed under reduced pressure and the dark red oil was crystallized from ethanol, resulting in a red solid which precipitated. The yield was extremely poor (under 1%) [m.p. 403 K (uncorrected)]. UV-vis (CH$_2$Cl$_2$): λ_{max} 394 nm. MS (CI, NH$_3$): m/z 441 (MH^+). ^{1}H NMR (CDCl$_3$, 400 MHz, TMS): δ 7.04 (s, 2H, ethenyl), 6.59 (s, 2H, thiophene). ^{13}C NMR (CDCl$_3$, 100 MHz, TMS): δ 142.13 (C12), 141.82 (C2), 140.02 (C6), 139.29 (C5), 130.10 (C7 and C11), 129.15 (C13 and C17), 128.56 (C3), 128.23 (C8 and C10), 128.13 (C9), 127.29 (C15), 126.82 (C14 and C16), 120.88 (C4). X-ray quality crystals were grown by layering a solution in dichloromethane with hexanes at 277 K for a few weeks.

Crystal data

C$_{32}$H$_{24}$S	D_x = 1.266 Mg m^{-3}
M_r = 440.57	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 9340
a = 26.7320 (14) Å	reflections
b = 9.7504 (5) Å	θ = 2.3–28.2°
c = 21.9268 (12) Å	μ = 0.16 mm^{-1}
β = 125.989 (1)°	T = 100 (2) K
V = 4624.3 (4) Å^3	Block, red
Z = 8	0.56 × 0.35 × 0.25 mm

Data collection

Bruker SMART APEX CCD diffractometer	5216 independent reflections
ω scans	4549 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan ($SADABS$ in $SAINT$-$Plus$; Bruker, 2003)	R_{int} = 0.015
	θ_{max} = 28.2°
	$h = -34 \rightarrow 35$
T_{min} = 0.819, T_{max} = 0.96	$k = -12 \rightarrow 11$
13241 measured reflections	$l = -28 \rightarrow 28$
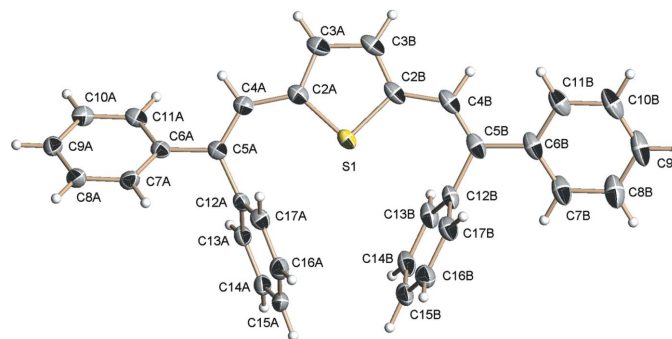
Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$+ 2.2188P]$
$wR(F^2)$ = 0.106	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
5216 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
298 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
H-atom parameters constrained	

Hydrogen atoms were placed in calculated positions with a C—H distance of 0.95 Å, and treated as riding, with U_{iso}(H) = 1.2U_{eq}(C).

The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: $SMART$ (Bruker, 2002); cell refinement: $SAINT$-$Plus$ (Bruker, 2003); data reduction: $SAINT$-$Plus$; program(s) used to solve structure: $SHELXTL$ (Bruker, 2000); program(s) used to refine structure: $SHELXTL$; molecular graphics: $SHELXTL$; software used to prepare material for publication: $SHELXTL$.

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