Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,8-Bis(3-phenylquinoxalin-2-yl)-5λ^6-dibenzo[*b*,*d*]thiophene-5,5-dione

Tai-Hsiang Huang,[a] Wha-Tzong Whang,[a]* Yuh-Sheng Wen[b] and Jiann T. Lin[b]

[a]Department of Materials Science and Engineering, National Chiao Tung University, 1001 Ta Hsueh Rd, Hsin Chu, Taiwan, and [b]Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan

Correspondence e-mail: redman@chem.sinica.edu.tw

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.066
Data-to-parameter ratio = 12.0

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

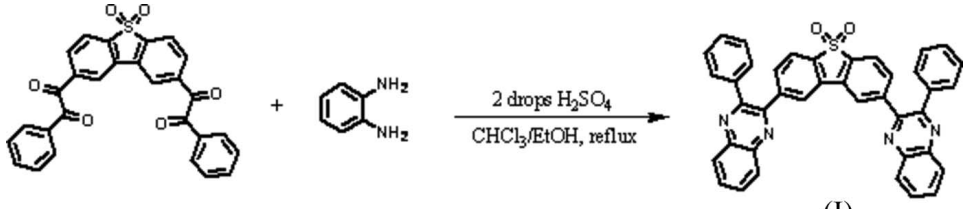
Received 2 November 2005
Accepted 10 November 2005
Online 16 November 2005

The bond lengths and angles in the title compound, $C_{40}H_{24}N_4O_2S$, are normal. The dihedral angles between the dibenzothiophene-*S*,*S*-dioxide and two quinoxaline groups are 34.88 (1) and 45.86 (1)°.

Comment

The application of organic electroluminescent (OEL) in flat-panel displays using small organic molecules or organic polymers has been intensively pursued after the reports of Kodak's team (Tang & Van Slyke>, 1987) and Cambridge's group (Burroughes *et al.*, 1990). Recently, the quinoxaline system has been introduced into small molecules (Thomas *et al.*, 2005) and successfully applied in organic light emitting devices (OLEDs) for electron-transport materials (Bettenhausen *et al.*, 1997).

In our search for new compounds that could be used as *n*-type OLEDs (Huang *et al.*, 2005), the title compound, (I) (Fig. 1), has been synthesized by the condensation of a bis-dione with a diamine (see scheme). All bond lengths and angles in (I) are normal (Table 1). The mean planes of the dibenzothiophene-*S*,*S*-dioxide (*P*1), two quinoxaline (*P*4 and *P*5) and two phenyl (*P*6 and *P*7) groups (see Fig. 1) make dihedral angles *P*1/*P*4, *P*1/*P*5, *P*1/*P*6, *P*1/*P*7, *P*4/*P*6 and *P*5/*P*7 of 34.88 (1), 45.86 (1), 52.50 (1), 56.68 (1), 42.50 (1) and 50.50 (1)°, respectively. The crystal packing is stabilized by van der Waals forces.



(I)

Experimental

A two-necked round-bottomed flask was charged with 1,2-phenylenediamine (216 mg, 2.2 mmol), 1-[5,5-dioxo-8-(2-oxo-2-phenylacetyl)-5H-5λ^6-dibenzothiophen-2-yl]-2-phenylethane-1,2-dione (528 mg, 1.1 mmol) and $CHCl_3$/ethanol (80 ml; ratio 1:2). Two drops of sulfuric acid were then added to initiate the reaction. The mixture was refluxed for 24 h. After cooling, the solvent was removed by Dean–Stark distillation. The resulting suspension was separated by filtration, washed with methanol and dried. The solid was sublimed to provided a powdery product. Crystals suitable for single-crystal X-ray diffraction were grown from a CH_2Cl_2 solution layered with *n*-hexane at room temperature. The compound was obtained as a colourless solid in 62% yield. FAB MS: *m*/*e* 624 $(M+H)^+$; ^{1}H NMR ($CDCl_3$): δ 7.39–7.42 (*m*, 6H, *ortho*-, *para*-C_6H_5), 7.50–7.54 (*m*, 6H, C_6H_3, *meta*-

C_6H_5), 7.69 (*d*, 2H, *J* = 8.0 Hz, C_6H_3), 7.83–7.86 (*m*, 4H, C_6H_4), 8.13 (*s*, 2H, C_6H_3), 8.19–8.25 (*m*, 4H, C_6H_4). Analysis calculated for $C_{40}H_{24}N_4O_2S$: C 76.90, H 3.87, N 8.97%; found: C 77.02, H 4.01, N 8.86%.

Crystal data

$C_{40}H_{24}N_4O_2S$	Z = 2
M_r = 624.69	D_x = 1.426 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 10.7261 (3) Å	Cell parameters from 2322 reflections
b = 12.0606 (4) Å	θ = 2.8–27.8°
c = 13.4213 (4) Å	μ = 0.16 mm^{-1}
α = 107.719 (2)°	T = 100.0 (1) K
β = 103.656 (2)°	Prism, colourless
γ = 108.751 (2)°	0.16 × 0.15 × 0.14 mm
V = 1454.81 (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	5107 independent reflections
φ and ω scans	3245 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	R_{int} = 0.042
T_{min} = 0.873, T_{max} = 0.980	θ_{max} = 25.0°
10660 measured reflections	$h = -12 \rightarrow 12$
	$k = -14 \rightarrow 13$
	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0197P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.066	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
S = 0.82	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
5107 reflections	Extinction correction: *SHELXL97*
425 parameters	Extinction coefficient: 0.0012 (4)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

S—O1	1.4353 (13)	N31—C30	1.325 (2)
S—O2	1.4407 (13)	N31—C37	1.365 (2)
S—C1	1.7669 (19)	N36—C29	1.320 (2)
S—C7	1.7747 (19)	N36—C38	1.370 (2)
N15—C14	1.322 (2)	C4—C13	1.489 (2)
N15—C21	1.366 (2)	C10—C29	1.489 (2)
N20—C13	1.315 (2)	C14—C23	1.483 (3)
N20—C22	1.362 (2)	C30—C39	1.484 (3)
O1—S—O2	117.01 (8)	C1—C6—C12	112.79 (17)
O1—S—C7	110.72 (8)	C12—C7—S	110.89 (14)
C6—C1—S	110.78 (14)	C7—C12—C6	112.89 (16)

H atoms were located geometrically and treated as riding, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

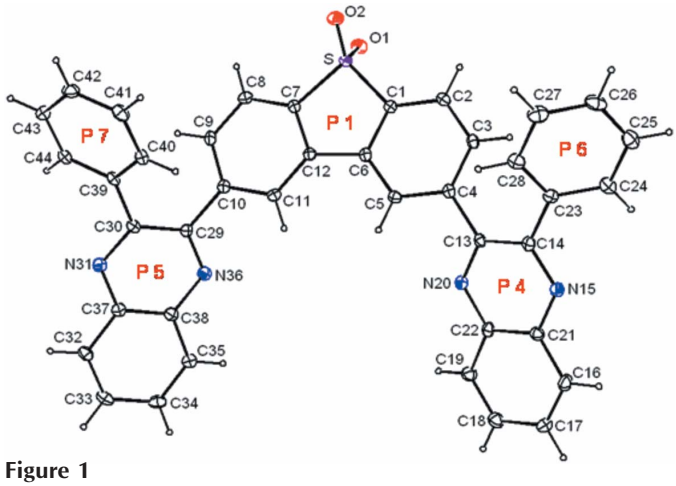


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

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 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work is supported by the Department of Materials Science and Engineering, National Chiao Tung University, and the Institute of Chemistry, Academia Sinica.

References

Bettenhausen, J., Greczmiel, M., Jandke, M. & Strohriegl, P. (1997). *Synth. Met.* **91**, 223–228.
Bruker (2001). *SMART*, *SAINT* (Apex2 Version 1.0-27) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burroughes, J. H., Bradley, D. D. C., Brown, A. R., Marks, R. N., Mackay, K., Friend, R. H., Burn, P. L. & Holmes, A. B. (1990). *Nature* (*London*), **347**, 539–540.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Huang, T.-H., Whang, W.-T., Shen, J. Y., Wen, Y.-S., Lin, J. T. & Wu, C.-C. (2005). *J. Mater. Chem.* Submitted.
Thomas, K. R. J., Velusamy, M., Lin, J. T., Tao, Y.-T. & Chuen, C.-H. (2005). *Chem. Mater.* **17**, 1860–1866.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tang, C. W. & Van Slyke, S. A. (1987). *Appl. Phys. Lett.* **51**, 913–915.