organic papers

1,3,5-Tris(2-acetylthienyl)benzene

Chen-Hao Zhu,[a,b] **Ji-Qin Pu,**[a,b] **Jian-Yan Huang**[a] **and Jie-Ying Wu**[a,b]*

[a]Deparment of Chemistry, Anhui University, Hefei 230039, People's Republic of China, and [b]Key Laboratory of Opto-Electronic Information Acquisition and Manipulation (Anhui University), Ministry of Education, Hefei 230039, People's Republic of China

Correspondence e-mail: jywu1957@163.com

In the title molecule, $C_{24}H_{18}O_3S_3$, all bond lengths and angles are normal. The three thiophene rings make dihedral angles of 21.7 (3), 19.3 (7) and 4.8 (4)° with the central benzene ring. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds and π–π interactions.

Comment

Thiophene derivatives constitute a class of materials with various applications in conducting films, electrochromics and field-effect transistors (FETs) (Perepichka *et al.*, 2005). Their optical and electronic properties can be tuned by varying the functional substituents or introducing a microstructure (Xia *et al.*, 2004). We present here the crystal structure of the title compound, (I), which is a new derivative of thiophene.

(I)
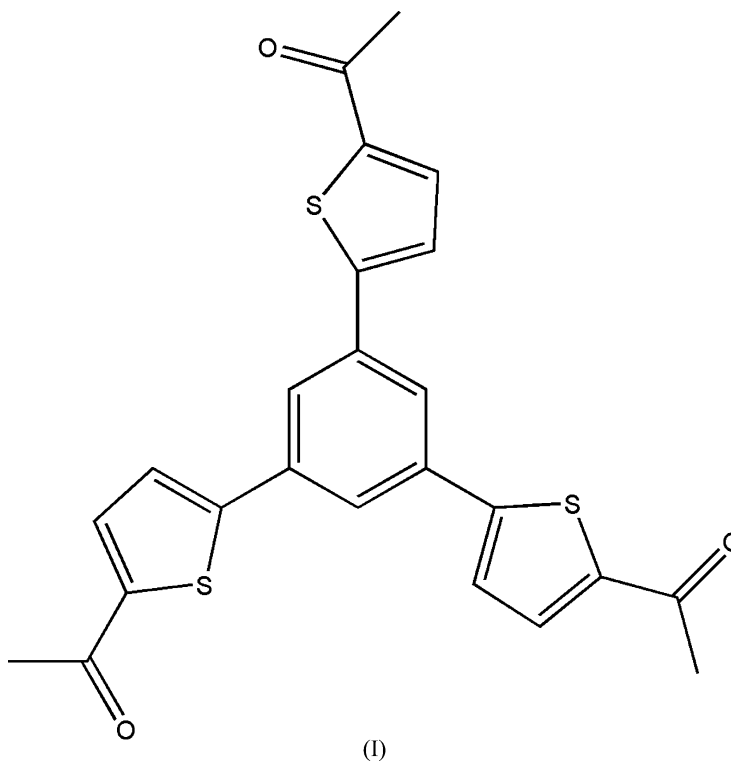
In (I) (Fig. 1), the bond lengths and angles show normal values (Allen *et al.*, 1987). The central benzene ring makes dihedral angles of 21.7 (3), 19.3 (7) and 4.8 (4)°, respectively, with the thiophene rings S1/C7–C10 (*A*), S2/C13–C16 (*B*) and S3/C19–C22 (*C*). The dihedral angles *A*/*B*, *A*/*C* and *B*/*C* are 30.6 (2), 20.5 (5) and 15.3 (1)°, respectively.

The crystal packing (Fig. 2) demonstrates weak intermolecular C—H···O hydrogen bonds (Table 1) and π–π
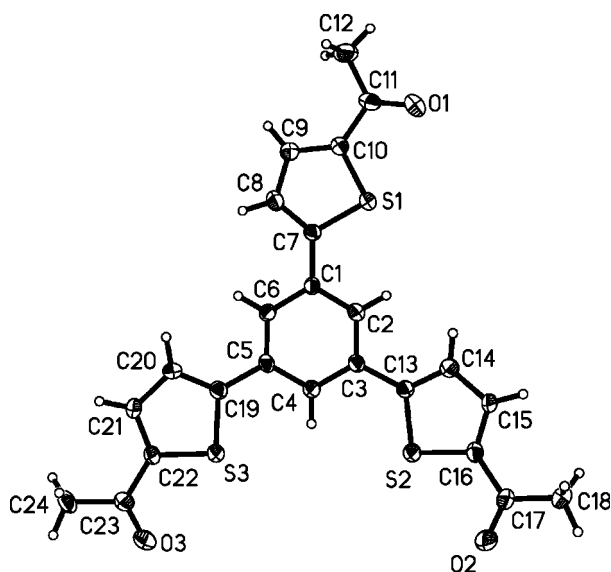
Figure 1
The molecular structure of (I), showing the atomic numbering and 30% probability displacement ellipsoids.
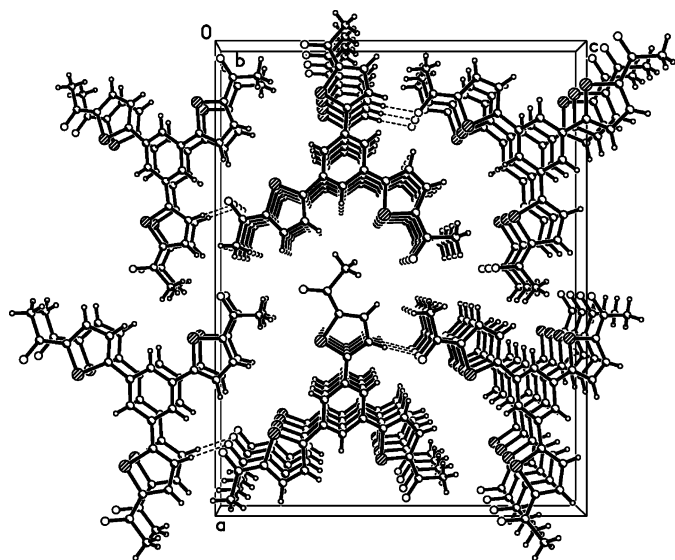

Figure 2
The packing of (I), viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

interactions in the stacks of molecules related by a translation along the *b* axis, with the shortest distance C2···C5^{ii} of 3.448 (9) Å [symmetry code: (ii) *x*, 1 + *y*, *z*].

Experimental

For the preparation of 1,3,5-tris(2-thienyl)benzene, a three-necked flask was charged with a mixture of 2-acetylthiophene (1 ml, 9.3 mmol) and dry ethanol (50 ml) in ice–water, stirred vigorously. Tetrachlorosilane (10 ml, 88 mmol) was added to the solution. Stirring continued under nitrogen for 18 h. This mixture was poured into water saturated with ammonium chloride (100 ml), stirred vigorously and extracted with dichloromethane (4 × 100 ml). The organic layer was dried over anhydrous magnesium sulfate and the solvent was removed under reduced pressure. The residue was purified by column

chromatography with light petroleum ether as the eluent to give a white solid product (0.62 g, yield 61.73%).

For the preparation of 1,3,5-tris(2-acetylthienyl)benzene, a flask was charged with a mixture of dichloromethane (12 ml) and powdered aluminium trichloride (3.37 g, 25 mmol) in ice–water, stirred vigorously for 10 min. Acetyl chloride (3.35 ml, 50 mmol) was added to the solution, then 1,3,5-tris(2-thienyl)benzene (1.72 g, 5 mmol) was dissolved in dichloromethane (5 ml) and added to the solution dropwise. The reaction mixture was stirred at room temperature for 2 h and then poured into ice–water. The resulting white solid was filtered and air-dried, then washed with dry methanol three times to give 2.05 g of the white product (yield 91%). Single crystals of (I) were obtained by slow evaporation of a dichloromethane solution at room temperature.

Crystal data

$C_{24}H_{18}O_3S_3$	$Z = 4$
$M_r = 450.56$	$D_x = 1.395$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation
$a = 25.620$ (16) Å	$\mu = 0.37$ mm^{-1}
$b = 4.179$ (3) Å	$T = 298$ (2) K
$c = 20.043$ (13) Å	Block, white
$V = 2146$ (2) Å^3	$0.68 \times 0.39 \times 0.08$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	10099 measured reflections
φ and ω scans	3183 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2210 reflections with $I > 2\sigma(I)$
$T_{min} = 0.787$, $T_{max} = 0.971$	$R_{int} = 0.080$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0036P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.171$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.97$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
3183 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
271 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	1928 Friedel pairs
	Flack parameter: 0.09 (14)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C14—H14$\cdots$O1^i	0.93	2.52	3.399 (10)	159

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

organic papers

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Perepichka, I. F., Perepichka, D. F., Hong, M. & Wudl, F. (2005). *Adv. Mater.* **17**, 2281–2305.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Xia, C.-J., Fan, X.-W., Locklin, J. C., Advincula, R., Gies, A. & Nonidez, W. (2004). *J. Am. Chem. Soc.* **126**, 8735–8743.