

Diacenaphtheno[7,8-*b*:7<sup>1</sup>,8<sup>1</sup>-*d*]thiopheneOliver C. Musgrave and  
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## Key indicators

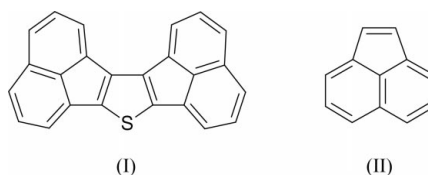
Single-crystal X-ray study  
 $T = 291\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.062  
 $wR$  factor = 0.145  
Data-to-parameter ratio = 19.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title compound,  $\text{C}_{24}\text{H}_{12}\text{S}$ , is essentially planar despite the presence of three fused five-membered rings; the maximum deviation from the plane of the molecule is  $0.035(2)\text{ \AA}$ . The alternation in the observed C—C bond lengths resembles that found in acenaphthylene and in thiophene. S··S interactions lead to the formation of a zigzag chain along *b*.

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## Comment

The title compound, (I), is readily prepared by the reaction of acenaphthylene, (II), with sulfur (Clapp, 1939), but remarkably little is known of its chemistry and its physical properties. The molecular structure of (I) is shown in Fig. 1. The molecule, despite containing three fused five-membered rings, is essentially planar; the maximum deviation from the plane is  $0.035(2)\text{ \AA}$  for atom C16. Considering the two acenaphthylene units separately, the maximum deviations from planarity are for C8 [ $0.013(2)\text{ \AA}$ ] in the group defined by atoms C1–C12, and for C16 [ $0.0220(19)\text{ \AA}$ ] in the group defined by atoms C13–C24.



In general, the bond angles and C—C bond lengths in the two acenaphthylene units resemble those found (by neutron diffraction) for acenaphthylene itself (Wood *et al.*, 1985). The alternation in the C—C bond lengths, which corresponds to that in the conjugated system shown in the canonical structure (I), resembles that observed in acenaphthylene, (II). The differences in the lengths of the corresponding C—C bonds range from  $0.004$  to  $0.028\text{ \AA}$ , while the changes in the bond angles are small, ranging from  $2.6$  to  $3.4^\circ$ . The heterocyclic ring

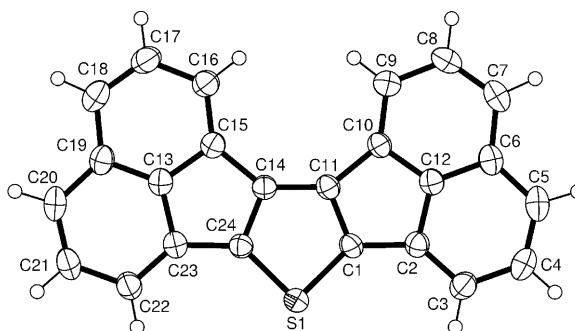
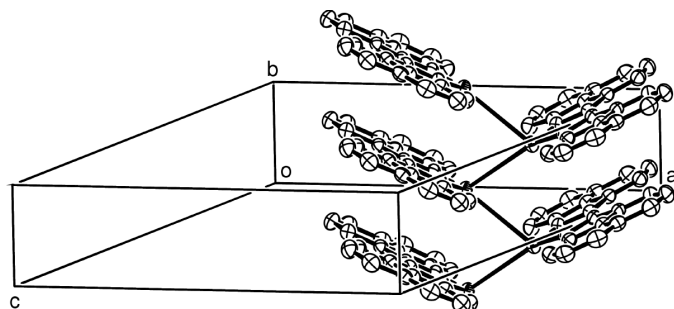


Figure 1

The structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level



**Figure 2**  
Crystal packing of (I), showing the molecular stacking and S...S interactions along *b*. Some molecules have been omitted for clarity.

of (I) shows a somewhat shorter C—C 'single' bond [1.407 (3) Å] than is present in thiophene itself (1.423 Å, measured by microwave spectroscopy; Bak *et al.*, 1961), but the C—S bond lengths are essentially unchanged.

In the crystal structure, the molecules are stacked along *b*, thus giving  $\pi$ – $\pi$  interactions along this direction; see Fig. 2. Despite their proximity [minimum centroid–centroid distance is 3.6440 (14) Å], no reaction occurred between the molecules when the crystalline solid was irradiated with UV light ( $\lambda = 254$  nm). The S...S distances of 3.5804 (9) Å are slightly less than the sum of the van der Waals radii and indicate interactions between the molecules resulting in an *n*-glide 'ladder' along *b* (Fig. 2). No hydrogen bonding was found by *PLATON* (Spek, 2003).

## Experimental

Diacenaphtheno[7,8-*b*:7<sup>1</sup>,8<sup>1</sup>-*d*]thiophene, (I), was prepared by heating acenaphthylene, (II), with sulfur at 498 K and purified as described previously (Clapp, 1939).

### Crystal data

$C_{24}H_{12}S$	$D_x = 1.459 \text{ Mg m}^{-3}$
$M_r = 332.40$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2369
$a = 15.0815$ (10) Å	reflections
$b = 3.8941$ (3) Å	$\theta = 2.6$ – $29.4^\circ$
$c = 26.0597$ (16) Å	$\mu = 0.22 \text{ mm}^{-1}$
$\beta = 98.498$ (2) $^\circ$	$T = 291$ (2) K
$V = 1513.65$ (18) Å <sup>3</sup>	Needle, red
$Z = 4$	$0.58 \times 0.08 \times 0.02 \text{ mm}$

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer	4413 independent reflections
$\varphi$ and $\omega$ scans	2683 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$R_{\text{int}} = 0.054$
$T_{\text{min}} = 0.782$ , $T_{\text{max}} = 0.996$	$\theta_{\text{max}} = 30.0^\circ$
12297 measured reflections	$h = -21 \rightarrow 17$
	$k = -5 \rightarrow 5$
	$l = -30 \rightarrow 36$

### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2]$
$wR(F^2) = 0.145$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} < 0.001$
4413 reflections	$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
226 parameters	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$

All H atoms were positioned geometrically and treated using a riding model; C—H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) and *OSCAIL* (McArdle, 1994, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

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