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Key indicators

Single-crystal X-ray study
T = 180 K
 Mean σ (C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.084
 Data-to-parameter ratio = 19.4

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

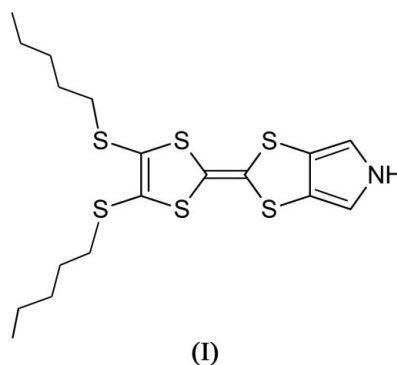
2-[4,5-Bis(*n*-pentylsulfanyl)-1,3-dithiol-2-ylidene]-1,3-dithiolo[4,5-*c*]pyrrole

The crystal structure of the title compound, C₁₈H₂₅NS₆, at 180 K contains molecules distorted significantly from planarity. The structure is non-centrosymmetric.

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Comment

In the crystal structure of the title compound, (I), at 180 K (Fig. 1), the molecules are distorted significantly from planarity.



The least-squares plane through atoms S1, S2, C1, C2, C3 and C4 forms a dihedral angle of 12.5 (1)° with the least-squares plane through atoms S1, S2, S3, S4, C5 and C6, which in turn forms a dihedral angle of 25.5 (1)° with the least-

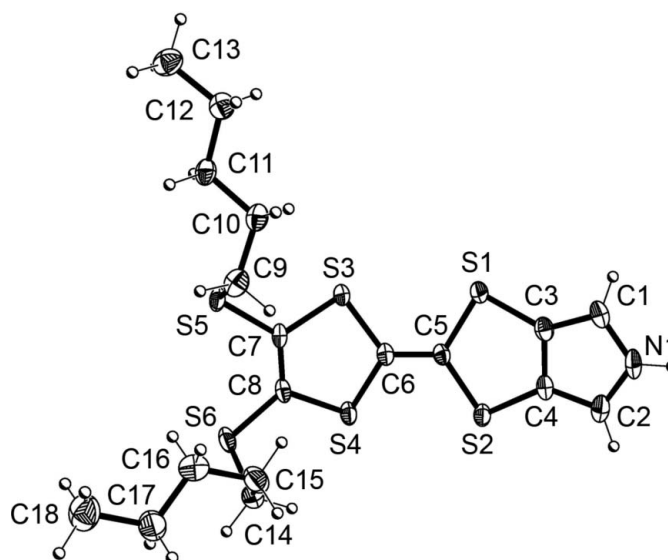


Figure 1
 The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radius.

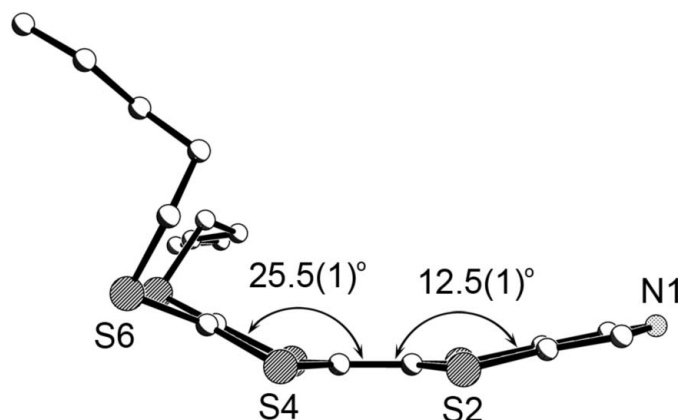


Figure 2
Side-on view of the molecule of (I), showing the non-planar conformation and the dihedral angles mentioned in the *Comment*.

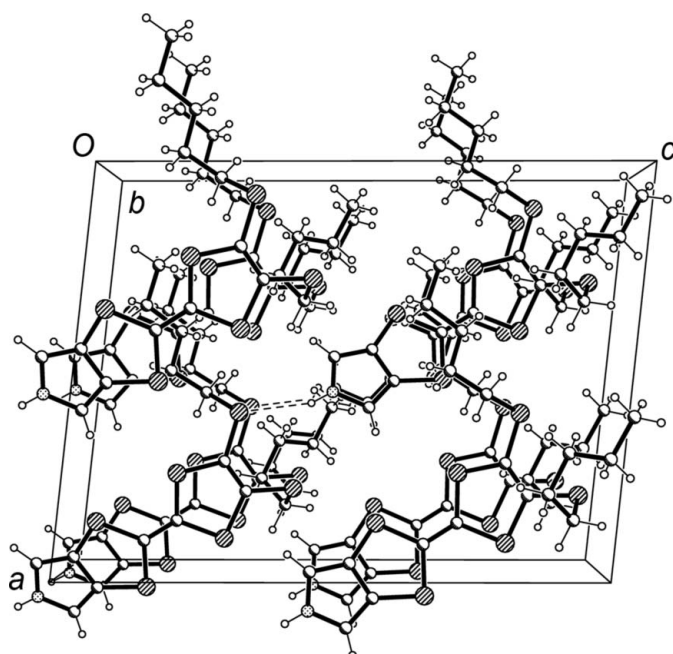


Figure 3
View of the structure along *b*, showing intermolecular N—H...S contacts as dashed lines.

squares plane through atoms S3, S4, S5, S6, C7 and C8 (Fig. 2). This distorted conformation contrasts with the essentially planar *N*-tosylated derivative reported previously (Jeppesen *et al.*, 2000). The N1—H vector points towards S5 in an adjacent molecule [$\text{H1B} \cdots \text{S5}^i = 2.67 \text{ \AA}$; symmetry code: $(i) \frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$], apparently directed towards a lone pair of electrons on sulfur (Fig. 3). These contacts can be envisaged to link the molecules into chains along $[\bar{1}01]$. The chains are aligned in a parallel manner and the structure is non-centrosymmetric.

Experimental

The title compound was prepared according to a literature procedure (Jeppesen *et al.*, 2000). Yellow needles of the title compound were obtained by slow diffusion of methanol into a solution of the title compound in dichloromethane.

Crystal data

$\text{C}_{18}\text{H}_{25}\text{NS}_6$
 $M_r = 447.75$
Monoclinic, *Cc*
 $a = 14.3456 (7) \text{ \AA}$
 $b = 7.8603 (4) \text{ \AA}$
 $c = 18.9621 (9) \text{ \AA}$
 $\beta = 96.254 (1)^\circ$
 $V = 2125.45 (18) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.399 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 0.65 \text{ mm}^{-1}$
 $T = 180 (2) \text{ K}$
Needle, yellow
 $0.35 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker SMART APEX CCD
diffractometer
Thin-slice ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2002)
 $T_{\min} = 0.756$, $T_{\max} = 0.938$

7249 measured reflections
4420 independent reflections
4209 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 28.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.084$
 $S = 1.08$
4420 reflections
228 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$
Absolute structure: Flack (1983),
1879 Friedel pairs
Flack parameter: 0.00 (7)

H atoms bound to C atoms were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the methylene groups, and C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl group. The methyl group was allowed to rotate around its local threefold axis. The H atom bound to N1 was visible in a difference Fourier map, but was subsequently placed in a calculated position with N—H = 0.88 Å and allowed to ride with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *S SAINT* (Bruker, 2001); data reduction: *S SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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