

5-(2-Cyanoethylsulfanyl)-4-methylsulfanyl-  
1,3-dithiole-2-thioneAnders Madsen, Kent A. Nielsen,  
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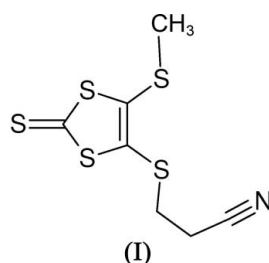
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The crystal structure of the title compound,  $C_7H_7NS_5$ , at 180 K, reveals intermolecular  $N \cdots \sigma^*(S-C)$  and  $S \cdots \sigma^*(S-C)$  interactions.Received 27 March 2006  
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## Comment

The crystal structure of the title compound, (I) (Fig. 1), was solved using data recorded at 180 K.

## Key indicators

Single-crystal X-ray study  
 $T = 180$  K  
Mean  $\sigma(C-C) = 0.003$  Å  
 $R$  factor = 0.026  
 $wR$  factor = 0.061  
Data-to-parameter ratio = 17.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The C atom of the methyl group is coplanar with the 1,3-dithiole-2-thione plane [torsion angle  $C4-S4-C2-C3 = -179.89$  ( $16^\circ$ )]. However, in the analogous bis(methylsulfanyl) compound (Simonsen *et al.*, 1990), both methyl groups lie out of the molecular plane [ $C-S-C$  torsion angles are  $-121.6$  (3) and  $156.2$  ( $2^\circ$ )]. The  $C6-C7 \equiv N1$  group of the 2-cyanoethylsulfanyl substituent lies above the 1,3-dithiole-2-thione plane, pointing approximately parallel to the  $S3-C3$  bond (Fig. 1). A similar conformation is observed in one molecule of the asymmetric unit of the related bis(2-cyanoethylsulfanyl) compound (Yu *et al.*, 2003). The  $C6-C7 \equiv N1$  group points towards  $S4$  of an adjacent molecule, forming an  $N1 \cdots S4^i$  contact of  $3.326$  ( $2$ ) Å [symmetry code: (i)  $-x, 1 - y,$

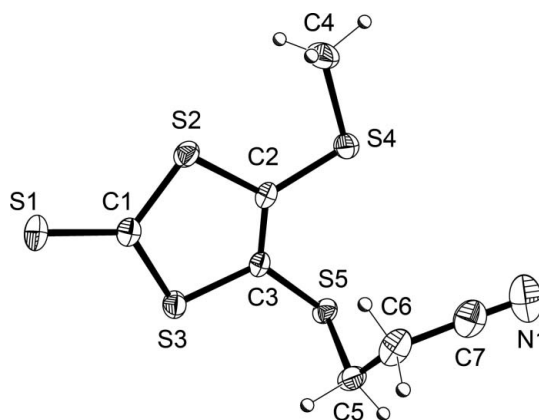


Figure 1

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.

1 - z]. This arrangement is typical of a nucleophile approaching a C—S—C unit and has been interpreted as an interaction between a electron lone pair on N and the  $\sigma^*$  orbital of the S—C bond (Rosenfield *et al.*, 1977). Similar interactions involve  $S1 \cdots S2^{ii} = 3.5354(7) \text{ \AA}$  and  $S1 \cdots S5^{iii} = 3.5816(7) \text{ \AA}$  [symmetry codes: (ii)  $2 - x, 1 - y, 2 - z$ ; (iii)  $1 + x, \frac{1}{2} - y, \frac{1}{2} + z$ ], with geometry indicative of interactions from lone pairs in  $sp^2$ -hybridized atomic orbitals on S1 (Fig. 2).

## Experimental

The compound was prepared according to the literature (Simonsen *et al.*, 1996). Recrystallization from methanol afforded yellow needle-shaped crystals.

### Crystal data

$C_7H_7NS_5$   
 $M_r = 265.44$   
 Monoclinic,  $P2_1/c$   
 $a = 5.0793(3) \text{ \AA}$   
 $b = 15.7214(12) \text{ \AA}$   
 $c = 13.9016(11) \text{ \AA}$   
 $\beta = 94.186(3)^\circ$   
 $V = 1107.13(14) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.592 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 Cell parameters from 3035 reflections  
 $\theta = 2.9\text{--}25.5^\circ$   
 $\mu = 1.00 \text{ mm}^{-1}$   
 $T = 180(2) \text{ K}$   
 Needle, yellow  
 $0.30 \times 0.10 \times 0.10 \text{ mm}$

### Data collection

Bruker–Nonius X8 APEX-II CCD diffractometer  
 thin-slice  $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.803, T_{\max} = 0.907$   
 14011 measured reflections

2108 independent reflections  
 1645 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$   
 $\theta_{\max} = 25.9^\circ$   
 $h = -4 \rightarrow 6$   
 $k = -19 \rightarrow 19$   
 $l = -16 \rightarrow 16$

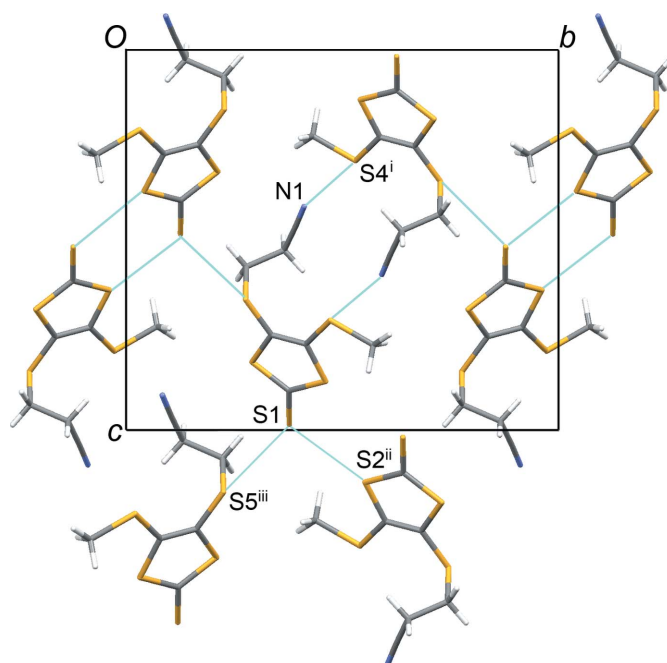
### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.061$   
 $S = 1.08$   
 2108 reflections  
 119 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0261P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.26 \text{ e \AA}^{-3}$

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.

Data collection: APEX2 (Bruker–Nonius, 2004); cell refinement: SAINT (Bruker, 2003); data reduction: 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.



**Figure 2**  
 Projection of (I) along  $a$ , showing intermolecular  $N \cdots \sigma^*(S-C)$  and  $S \cdots \sigma^*(S-C)$  interactions as pale green lines.

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