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2,3-Bis(2-cyanoethylsulfanyl)-6,7-bis(methylsulfanyl)tetrathiafulvalene

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Key indicators

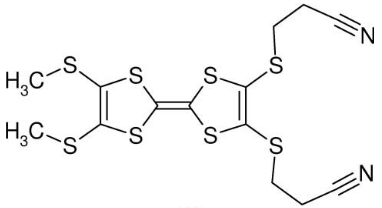
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.086
Data-to-parameter ratio = 27.7

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

The crystal structure of the title compound, $C_{14}H_{14}N_2S_8$, at 180 K contains pairs of molecules related by centres of inversion, arranged in a herringbone-type manner to form layers. $N\cdots\sigma^*(S-C)$ interactions exist between layers.

Comment

The crystal structure of the title molecule, (I) (Fig. 1), at 180 K contains pairs of molecules related by centres of inversion, with the planes through their tetrathiafulvalene (TTF) sections parallel and separated by *ca* 3.5 Å. These pairs are arranged in a herringbone-type manner, forming layers parallel to (100) (Fig. 2).



(I)

The 2-cyanoethylsulfanyl substituents lying at the surfaces of each layer are aligned so that each points approximately along [001] or [00$\bar{1}$]; the substituents point in opposite direc-

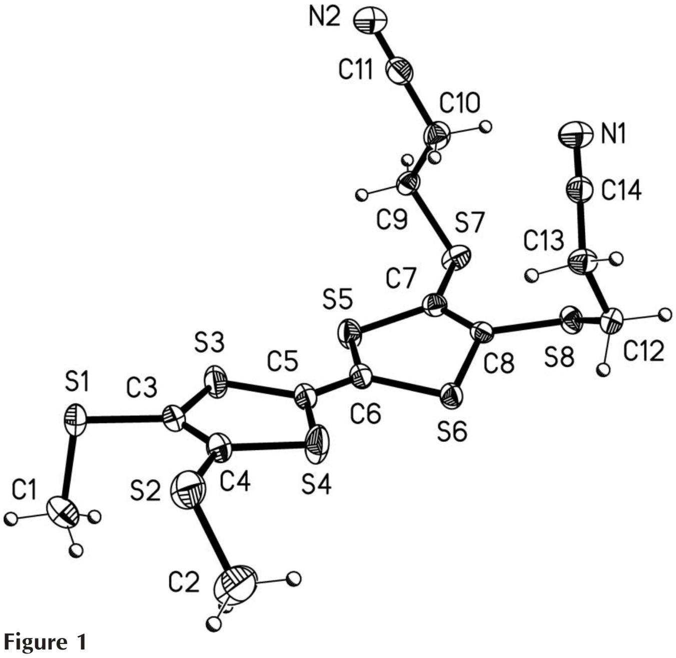


Figure 1
Molecular unit, showing displacement ellipsoids at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radii.

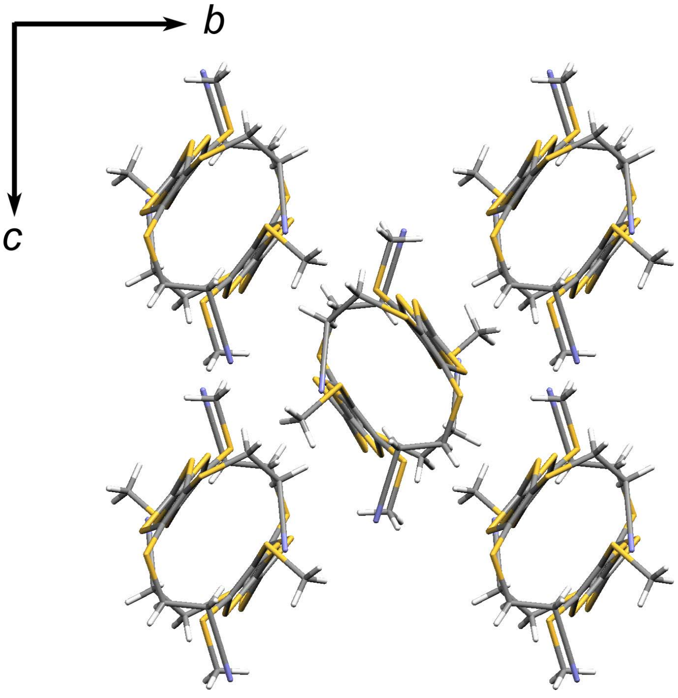


Figure 2
Projection of a single layer approximately on to (100), showing the herringbone-type arrangement of pairs of molecules.

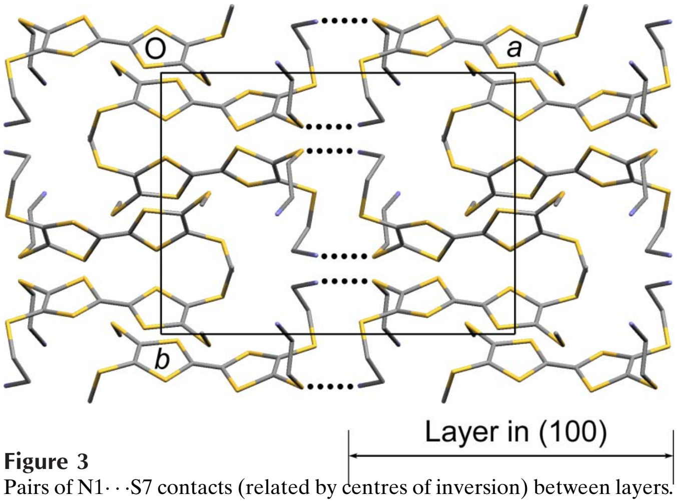


Figure 3
Pairs of N1$\cdots$S7 contacts (related by centres of inversion) between layers.

tions on the top and bottom surfaces of a given layer. Adjacent layers are arranged across centres of inversion so that the 2-cyanoethylsulfanyl substituents meeting at the layer interface point in opposite directions. Between layers, N$\cdots$S contacts of 3.3317 (17) Å exist [N1$\cdots$S7$^{\rm i}$; symmetry code: (i) $1 - x, 1 - y, -z$], in which the axis of the 2-cyanoethylsulfanyl group points towards the rear of the C7—S7 bond (Fig. 3). This arrangement is typical for a nucleophile approaching a C—S—C unit and has been interpreted as an interaction between a lone pair of electrons on N and the σ* orbital of the S—C bond (Rosenfield *et al.*, 1977). A similar approach within layers is made by N2 towards the backside of the C6—S6 bond [N2$\cdots$S6$^{\rm ii}$ = 3.7682 (18), symmetry code (ii): $x, y, -1 + z$].

Experimental

The compound was prepared according to the literature procedure (Simonsen *et al.*, 1996). Recrystallization from CH_2Cl_2/cyclohexane afforded orange needles.

Crystal data

$C_{14}H_{14}N_2S_8$
$M_r = 466.75$
Monoclinic, $P2_1/c$
$a = 16.2850$ (4) Å
$b = 11.6795$ (3) Å
$c = 10.7413$ (3) Å
$\beta = 103.635$ (1)°
$V = 1985.42$ (9) Å^3
$Z = 4$
$D_x = 1.562$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5703 reflections
$\theta = 2.2$–28.1°
$\mu = 0.90$ mm^{-1}
$T = 180$ (2) K
Block cut from needle, orange
0.25 × 0.15 × 0.15 mm

Data collection

Bruker–Nonius X8 APEX2 CCD diffractometer
Thin–slice ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\rm min} = 0.762$, $T_{\rm max} = 0.877$
28528 measured reflections
6059 independent reflections
4352 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\rm max} = 30.5°$
$h = -23 \rightarrow 23$
$k = -13 \rightarrow 16$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.14$
6059 reflections
219 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.63$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.61$ e Å^{-3}
Extinction correction: none

H atoms were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.99 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for methylene groups, and C—H = 0.98 Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl groups. The methyl groups were also allowed to rotate about their local threefold axes.

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*.

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