Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[4,5-Bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-yl]-1,3-thiazolidine-2-thione

Wei Yang,[a] Zhi-Gang Ren,[b]* Jie Dai[b] and Seik Weng Ng[c]

[a]Department of Basic Course, Suzhou Polytechnical Institute of Agriculture, Suzhou 215008, People's Republic of China, [b]School of Chemistry and Chemical Engineering, Suzhou University, Suzhou 215123, People's Republic of China, and [c]Department of Chemistry, University of Malaysia, Kuala Lumpur 50603, Malaysia
Correspondence e-mail: renzhigang@suda.edu.cn

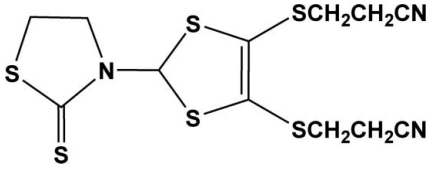
Received 4 April 2007; accepted 17 April 2007

Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.033; wR factor = 0.081; data-to-parameter ratio = 17.8.

The title compound, $C_{12}H_{13}N_3S_6$, belongs to a group of sulfur-rich compounds. In the crystal structure, the molecules form several short S···S intermolecular contacts [3.287 (1)–3.497 (1) Å], assembling them into a two-dimensional layer network. One $-CH_2-CH_2-$ group is disordered over two positions in an approximate 4:1 ratio.

Related literature

For related literature, see: Bryce (1991); Lorcy & Bellec (2004); Williams *et al.* (1985).



Experimental

Crystal data

$C_{12}H_{13}N_3S_6$
M_r = 391.61
Monoclinic, $C2/c$
a = 25.252 (5) Å
b = 8.2516 (17) Å
c = 16.017 (3) Å
β = 96.34 (3)°
V = 3317.0 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.82 mm^{-1}
T = 193 (2) K
0.65 × 0.60 × 0.20 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.618, T_{max} = 0.853
12131 measured reflections
3710 independent reflections
3591 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.081
S = 1.13
3710 reflections
209 parameters
38 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This work was supported by the NSF of the Education Committee of Jiangsu Province (Grant 06KJB150102) and the Research Fund for the Youth of Suzhou University (No. Q3109605).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2065).

References

Bryce, M. R. (1991). *Chem. Soc. Rev.* **20**, 355–390.
Jacobson, R. (1998). Private communication to the Rigaku Corporation, Tokyo, Japan.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lorcy, D. & Bellec, N. (2004). *Chem. Rev.* **104**, 5185–5202.
Rigaku (2001). *CrystalClear*. Version 1.3. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Williams, J. M., Beno, M. A., Wang, H. H., Leung, P. C. W., Emge, T. J., Geiser, U. & Carlson, K. D. (1985). *Acc. Chem. Res.* **18**, 261–267.

  doi:10.1107/S1600536807019034

supplementary materials

Acta Cryst. (2007). E**63**, o2598 [**doi:10.1107/S1600536807019034**]

3-[4,5-Bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-yl]-1,3-thiazolidine-2-thione

W. Yang, Z.-G. Ren, J. Dai and S. W. Ng

Comment

Sulfur-rich compounds, e.g. 4,5-bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-thione or 4,5-bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-one, are well known of being used as precursors for the synthesis of molecular materials of tetrathiafulvalene (TTF) (Bryce, 1991; Williams *et al.*, 1985). Recently TTF analogs with nitrogen containing heterocycles have been reported (Lorcy & Bellec, 2004). As short S···S contacts are efficient organizing forces in the self-assembly of new solid-state materials (Bryce, 1991; Williams *et al.*, 1985), the title compound (I) offers one example of this type of materials.

Compound (I) consists of a thiazolidine ring and a dithiole ring (Fig. 1). Both rings are not planar while the displaced atoms are maximum 0.22 (1) Å (C3) and 0.19 Å (C4) out of the least-squires planes, respectively. The least-squares planes are approximately vertical with a torsion angle of 85.68 (1)°. The C_2S_4 group C5—C6—S3—S4—S5—S6 is nearly planar while the C6 atom is 0.89 (1) Å out of the least-squires plane.

There are four kinds of intramolecular C—H···S and one kind of intermolecular C—H···N hydrogen bonds (Table 2). Besides these interactions, the neighboring molecules are connected by intermolecular S···S contacts (S3···S4^{i} 3.364 (1) Å; S2···S4^{ii} 3.497 (1) Å; S2···S2^{iii} 3.287 (1) Å [symmetry codes:(i)1/2-x,1/2+y,1/2-z; (ii)1/2-x,3/2-y,1-z; (iii)1/2-x,5/2-y,1-z)] to form a 2D layer network (Fig. 2).

Experimental

A solution of 1,3-thiazolidine-2-thione (0.12 g, 1 mmol) and 4,5-bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-one (0.27 g, 0.9 mmol) in 3 ml of $P(OEt)_3$ was refluxed for 1 h and cooled to room temperature. Addition of 6 ml of CH_3OH into the solution formed a colorless precipitate. Recrystallization from CH_2Cl_2 afforded colorless cryatals of (I) (yield 0.12 g, 35%). CH&N elemental analysis. Found: C, 36.60; H, 3.39; N, 10.47%. Calculated for $C_{12}H_{13}N_3S_6$: C, 36.83; H, 3.32; N, 10.74%.

Refinement

One C_2H_4 group is disordered over two sites with occupancy factors of 0.80 (2) and 0.20 (2) for C10/C10 A and C11/C11A. The H atoms are placed in geometrically idealized positions (C—H = 0.99 Å) with U_{iso}(H) = 1.2U_{eq}(C).

Figures

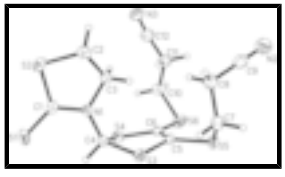


Fig. 1. The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii. The disordered atoms C10A and C11A have been omitted.

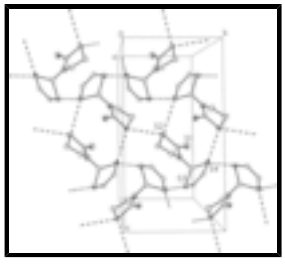


Fig. 2. The intermolecular S···S interactions (dashed lines) that link the molecules into a layer. The 2-cyanoethylthio groups and H atoms have been omitted.

3-[4,5-Bis(2-cyanoethylsulfanyl)-1,3-dithiol-2-yl]-1,3-thiazolidine-2-thione

Crystal data

$C_{12}H_{13}N_3S_6$	F_{000} = 1616
M_r = 391.61	D_x = 1.568 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 5529 reflections
a = 25.252 (5) Å	θ = 3.1–27.5º
b = 8.2516 (17) Å	μ = 0.82 mm^{-1}
c = 16.017 (3) Å	*T* = 193 (2) K
β = 96.34 (3)º	Platelet, colorless
V = 3317.0 (12) Å^3	0.65 × 0.60 × 0.20 mm
Z = 8	

Data collection

Rigaku Mercury diffractometer	3710 independent reflections
Radiation source: fine-focus sealed tube	3591 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 193(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (Jacobson, 1998)	*h* = −32→32
T_{min} = 0.618, T_{max} = 0.853	*k* = −10→9
12131 measured reflections	*l* = −17→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 3.7537P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.081	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.13	$\Delta\rho_{max}$ = 0.36 e Å^{-3}
3710 reflections	$\Delta\rho_{min}$ = −0.31 e Å^{-3}

209 parameters
38 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.325225 (16)	0.82215 (5)	0.42436 (3)	0.02940 (11)	
S2	0.247064 (18)	1.05371 (5)	0.48278 (2)	0.02610 (11)	
S3	0.201298 (15)	0.80445 (5)	0.18681 (2)	0.02303 (10)	
S4	0.194822 (17)	0.55846 (5)	0.31728 (3)	0.02608 (11)	
S5	0.092782 (16)	0.72706 (5)	0.09868 (3)	0.02730 (11)	
S6	0.08521 (2)	0.45029 (6)	0.25284 (3)	0.03507 (13)	
N1	0.22439 (5)	0.87341 (17)	0.35390 (8)	0.0224 (3)	
N2	−0.03936 (7)	1.0195 (2)	0.13715 (11)	0.0399 (4)	
N3	0.03213 (7)	0.6819 (2)	0.53333 (12)	0.0439 (4)	
C1	0.26529 (6)	0.90535 (18)	0.41376 (9)	0.0203 (3)	
C2	0.18570 (7)	1.0992 (2)	0.41697 (11)	0.0276 (3)	
H2A	0.1569	1.1284	0.4515	0.033*	
H2B	0.1909	1.1896	0.3781	0.033*	
C3	0.17259 (6)	0.9430 (2)	0.36893 (11)	0.0270 (3)	
H3A	0.1529	0.8677	0.4024	0.032*	
H3B	0.1506	0.9653	0.3151	0.032*	
C4	0.22825 (6)	0.7466 (2)	0.29289 (10)	0.0221 (3)	
H4	0.2669	0.7215	0.2917	0.027*	
C5	0.14035 (6)	0.69990 (18)	0.18623 (10)	0.0194 (3)	
C6	0.13725 (6)	0.58876 (19)	0.24673 (10)	0.0209 (3)	
C7	0.09026 (7)	0.9462 (2)	0.08893 (11)	0.0280 (3)	
H7A	0.1271	0.9875	0.0888	0.034*	
H7B	0.0704	0.9743	0.0341	0.034*	
C8	0.06425 (7)	1.0329 (2)	0.15818 (11)	0.0297 (4)	
H8A	0.0774	0.9842	0.2130	0.036*	
H8B	0.0752	1.1482	0.1596	0.036*	
C9	0.00602 (8)	1.0239 (2)	0.14670 (11)	0.0295 (4)	
C10	0.07459 (10)	0.4652 (3)	0.36427 (16)	0.0303 (6)	0.804 (6)

H10A	0.0514	0.3756	0.3795	0.036*	0.804 (6)
H10B	0.1091	0.4572	0.3999	0.036*	0.804 (6)
C10A	0.0490 (3)	0.5679 (11)	0.3294 (6)	0.025 (2)	0.196 (6)
H10C	0.0529	0.6857	0.3199	0.030*	0.196 (6)
H10D	0.0106	0.5409	0.3217	0.030*	0.196 (6)
C11	0.04842 (9)	0.6272 (3)	0.37826 (16)	0.0303 (6)	0.804 (6)
H11A	0.0708	0.7158	0.3595	0.036*	0.804 (6)
H11B	0.0133	0.6322	0.3443	0.036*	0.804 (6)
C11A	0.0726 (3)	0.5240 (12)	0.4167 (6)	0.028 (2)	0.196 (6)
H11C	0.1115	0.5420	0.4249	0.034*	0.196 (6)
H11D	0.0646	0.4104	0.4309	0.034*	0.196 (6)
C12	0.04136 (7)	0.6504 (3)	0.46880 (14)	0.0392 (5)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0214 (2)	0.0305 (2)	0.0344 (2)	0.00107 (15)	−0.00575 (16)	0.00277 (17)
S2	0.0355 (2)	0.0223 (2)	0.0197 (2)	−0.00333 (16)	−0.00102 (16)	−0.00293 (14)
S3	0.02128 (19)	0.0286 (2)	0.01934 (19)	−0.00545 (14)	0.00284 (14)	−0.00153 (14)
S4	0.0316 (2)	0.0218 (2)	0.0242 (2)	0.00501 (15)	−0.00018 (16)	0.00094 (14)
S5	0.0259 (2)	0.0270 (2)	0.0267 (2)	0.00202 (16)	−0.00711 (15)	−0.00758 (16)
S6	0.0436 (3)	0.0310 (2)	0.0334 (2)	−0.01937 (19)	0.0168 (2)	−0.01416 (18)
N1	0.0165 (6)	0.0277 (7)	0.0225 (7)	0.0013 (5)	0.0002 (5)	−0.0074 (5)
N2	0.0379 (9)	0.0416 (10)	0.0405 (10)	0.0022 (7)	0.0066 (7)	−0.0003 (7)
N3	0.0435 (10)	0.0484 (11)	0.0411 (10)	−0.0047 (8)	0.0109 (8)	−0.0173 (8)
C1	0.0232 (7)	0.0189 (7)	0.0183 (7)	−0.0039 (6)	0.0006 (5)	0.0029 (6)
C2	0.0294 (8)	0.0268 (8)	0.0267 (8)	0.0031 (7)	0.0037 (6)	−0.0052 (6)
C3	0.0188 (7)	0.0319 (9)	0.0300 (9)	0.0024 (6)	0.0018 (6)	−0.0098 (7)
C4	0.0169 (7)	0.0266 (8)	0.0225 (7)	0.0025 (6)	0.0007 (5)	−0.0055 (6)
C5	0.0175 (6)	0.0194 (7)	0.0213 (7)	0.0004 (5)	0.0014 (5)	−0.0054 (5)
C6	0.0215 (7)	0.0190 (7)	0.0226 (7)	0.0006 (6)	0.0045 (6)	−0.0063 (6)
C7	0.0305 (8)	0.0304 (9)	0.0224 (8)	0.0047 (7)	0.0001 (6)	0.0037 (6)
C8	0.0338 (9)	0.0264 (8)	0.0273 (9)	0.0043 (7)	−0.0036 (7)	−0.0028 (6)
C9	0.0406 (10)	0.0235 (8)	0.0242 (8)	0.0055 (7)	0.0028 (7)	−0.0002 (6)
C10	0.0401 (12)	0.0207 (10)	0.0329 (14)	−0.0023 (9)	0.0162 (9)	0.0002 (9)
C10A	0.018 (4)	0.027 (4)	0.030 (5)	0.001 (3)	0.002 (3)	−0.002 (4)
C11	0.0290 (11)	0.0281 (12)	0.0345 (15)	0.0031 (9)	0.0068 (9)	−0.0045 (10)
C11A	0.026 (4)	0.036 (5)	0.022 (5)	0.002 (4)	0.005 (3)	0.000 (4)
C12	0.0282 (9)	0.0433 (11)	0.0479 (12)	−0.0104 (8)	0.0119 (8)	−0.0189 (9)

Geometric parameters (Å, °)

S1—C1	1.6535 (16)	C3—H3A	0.9900
S2—C1	1.7450 (17)	C3—H3B	0.9900
S2—C2	1.8146 (19)	C4—H4	1.0000
C2—C3	1.519 (2)	C7—C8	1.528 (2)
N1—C3	1.472 (2)	C7—H7A	0.9900
N1—C1	1.355 (2)	C7—H7B	0.9900
N1—C4	1.442 (2)	C8—C9	1.463 (3)

S3—C4	1.8227 (17)	C8—H8A	0.9900
S3—C5	1.7635 (15)	C8—H8B	0.9900
C5—C6	1.343 (2)	C10—C11	1.519 (3)
S4—C6	1.7580 (17)	C10—H10A	0.9900
S4—C4	1.8301 (17)	C10—H10B	0.9900
S5—C5	1.7571 (17)	C10A—C11A	1.502 (13)
S6—C6	1.7521 (16)	C10A—H10C	0.9900
S5—C7	1.8155 (18)	C10A—H10D	0.9900
S6—C10	1.838 (2)	C11—C12	1.493 (3)
S6—C10A	1.878 (10)	C11—H11A	0.9900
N2—C9	1.140 (3)	C11—H11B	0.9900
N3—C12	1.115 (3)	C11A—C12	1.598 (10)
C2—H2A	0.9900	C11A—H11C	0.9900
C2—H2B	0.9900	C11A—H11D	0.9900
C1—S2—C2	92.60 (8)	S5—C7—H7A	108.6
C5—S3—C4	96.48 (8)	C8—C7—H7B	108.6
C6—S4—C4	96.34 (7)	S5—C7—H7B	108.6
C6—S6—C10A	97.0 (3)	H7A—C7—H7B	107.6
C1—N1—C4	121.14 (13)	C9—C8—C7	113.04 (15)
C1—N1—C3	115.46 (13)	C9—C8—H8A	109.0
C4—N1—C3	121.42 (13)	C7—C8—H8A	109.0
N1—C1—S1	127.54 (12)	C9—C8—H8B	109.0
N1—C1—S2	110.54 (11)	C7—C8—H8B	109.0
S1—C1—S2	121.92 (9)	H8A—C8—H8B	107.8
C3—C2—S2	104.05 (12)	N2—C9—C8	178.8 (2)
C3—C2—H2A	110.9	C11—C10—S6	108.34 (16)
S2—C2—H2A	110.9	C11—C10—H10A	110.0
C3—C2—H2B	110.9	S6—C10—H10A	110.0
S2—C2—H2B	110.9	C11—C10—H10B	110.0
H2A—C2—H2B	109.0	S6—C10—H10B	110.0
N1—C3—C2	105.42 (13)	H10A—C10—H10B	108.4
N1—C3—H3A	110.7	C11A—C10A—S6	108.1 (6)
C2—C3—H3A	110.7	C11A—C10A—H10C	110.1
N1—C3—H3B	110.7	S6—C10A—H10C	110.1
C2—C3—H3B	110.7	C11A—C10A—H10D	110.1
H3A—C3—H3B	108.8	S6—C10A—H10D	110.1
N1—C4—S3	113.31 (11)	H10C—C10A—H10D	108.4
N1—C4—S4	113.80 (11)	C12—C11—C10	110.9 (2)
S3—C4—S4	106.70 (8)	C12—C11—H11A	109.5
N1—C4—H4	107.6	C10—C11—H11A	109.5
S3—C4—H4	107.6	C12—C11—H11B	109.5
S4—C4—H4	107.6	C10—C11—H11B	109.5
C6—C5—S5	125.02 (12)	H11A—C11—H11B	108.0
C6—C5—S3	116.79 (12)	C10A—C11A—C12	99.3 (6)
S5—C5—S3	117.46 (9)	C10A—C11A—H11C	111.9
C5—C6—S6	126.12 (13)	C12—C11A—H11C	111.9
C5—C6—S4	116.81 (12)	C10A—C11A—H11D	111.9
S6—C6—S4	116.33 (9)	C12—C11A—H11D	111.9
C5—S5—C7	102.13 (8)	H11C—C11A—H11D	109.6

C6—S6—C10	101.62 (9)	N3—C12—C11	171.9 (3)
C8—C7—S5	114.69 (13)	N3—C12—C11A	143.3 (4)
C8—C7—H7A	108.6		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3B···S3	0.99	2.86	3.2875 (19)	107
C4—H4···S1	1.00	2.58	3.1113 (18)	113
C7—H7A···S3	0.99	2.76	3.274 (2)	113
C8—H8B···N3^{i}	0.99	2.60	3.136 (3)	114
C10—H10B···S4	0.99	2.79	3.299 (2)	113

Symmetry codes: (i) *x*, −*y*+2, *z*−1/2.

Fig. 1

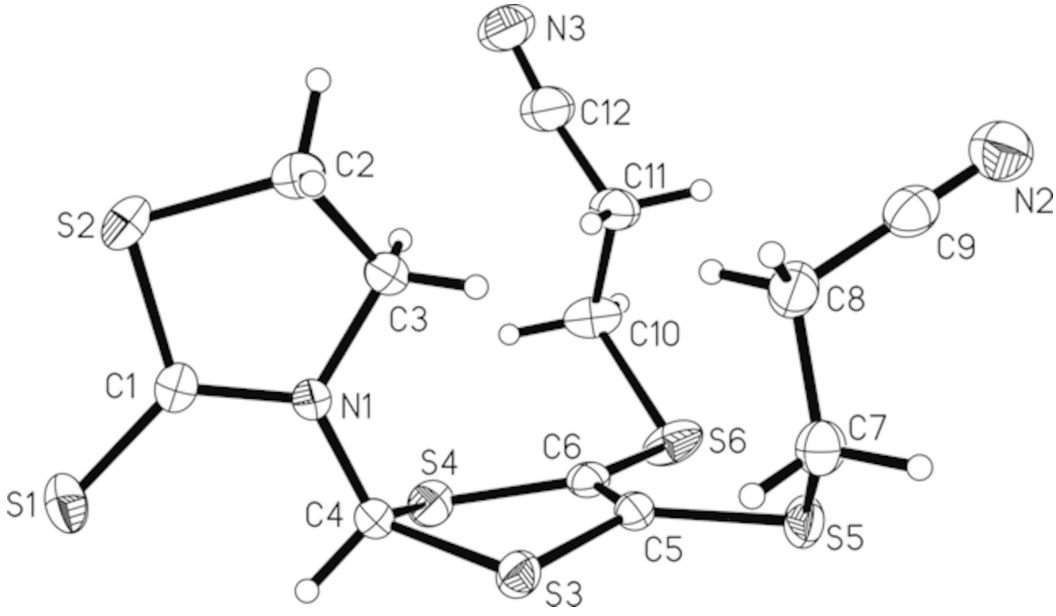


Fig. 2