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N-Ethyl-N-phenyl{[ethyl(phenyl)- carbamothioyl]disulfanyl}carbothio- amide

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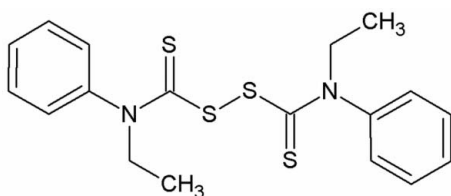
Received 13 June 2012; accepted 19 June 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.024; wR factor = 0.068; data-to-parameter ratio = 22.6.

The asymmetric unit of the title compound, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{S}_4$, contains one half-molecule, the complete molecule being generated by a twofold rotation axis. The plane through the NCS_2 group [maximum deviation = 0.01 (7) Å] is orthogonal to the phenyl ring, forming a dihedral angle of 89.4 (3)°. The crystal structure is stabilized by intermolecular $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For background to the chemistry of thiuram disulfides and their potential applications, see: Chieh (1977); McCleverty & Morrison (1976); Victoriano (2000). For related structures, see: Fun *et al.* (2001); Raya *et al.* (2005).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{20}\text{N}_2\text{S}_4$
 $M_r = 392.6$

Monoclinic, $C2/c$
 $a = 15.1923$ (2) Å

$b = 11.5954$ (2) Å
 $c = 12.3762$ (2) Å
 $\beta = 115.039$ (1)°
 $V = 1975.31$ (5) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.48$ mm⁻¹
 $T = 100$ K
 $0.40 \times 0.37 \times 0.15$ mm

Data collection

Bruker APEXII CCD
diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2008)
 $T_{\min} = 0.830$, $T_{\max} = 0.931$

19720 measured reflections
2481 independent reflections
2405 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.068$
 $S = 1.06$
2481 reflections

110 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.40$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

C_g is the centroid of the C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C8}-\text{H8B}\cdots\text{C}_g^i$	0.98	2.97	3.7972 (14)	143

Symmetry code: (i) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z$.

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT-Plus (Bruker, 2008); data reduction: SAINT-Plus and XPREP (Bruker, 2008); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

We thank the University of KwaZulu-Natal and the National Research Foundation (NRF) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RU2038).

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supporting information

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***N*-Ethyl-*N*-phenyl{[ethyl(phenyl)carbamothioyl]disulfanyl}carbothioamide**

Peter A. Ajibade, Benjamin C. Ejelonu and Bernard Omondi

S1. Comment

Thiuram disulfides are semi-esters of dialkyldithiocarbamic acids (Victoriano, 2000). They are unique among thiolato type ligands in that reductive scission of the S—S bond leads to chelating dithiocarbamtes anions which are particularly well suited to stabilize high oxidation state transition metals like dithiocarbamate ligands (Victoriano, 2000). Metal species with closed shell configuration typically react with thiuram disulfides to yield adducts. Some adducts of well defined thiuram complexes have been obtained by the reaction of group 12 halides and the ligands McCleverty & Morrison (1976)).

The structure of (I) Fig. 1, consists of two *N*ethyl-*N*-phenyldithiocarbamate units linked by an S—S bond. The plane of NCS₂ group is orthogonal to the plane of the phenyl ring forming a dihedral angle of 89.40 (3)° between them. The torsion angle between the thiocarbamate moieties (NCS₂) is 79.01 (8)°. The lattice is stabilized by C—H⋯π intermolecular interactions with a Cg⋯H distance of 3.7972 (14) Å.

The S—C, S=C and C—N bond distances are comparable to those of related structures (Fun *et al.* 2001; Raya *et al.* 2005).

S2. Experimental

A mixture of 6.44 ml of ethylaniline and 15.00 ml of concentrated aqueous ammonia in ice was added into 3.00 ml of ice-cold carbon disulfide and the resultant solution stirred for 6–7 h. The solid product obtained was filtered and rinsed three times with ice cold ethanol three times. The yellowish white was recrystallized in water/methanol mixture to yield crystal suitable for X-ray crystallographic analysis.

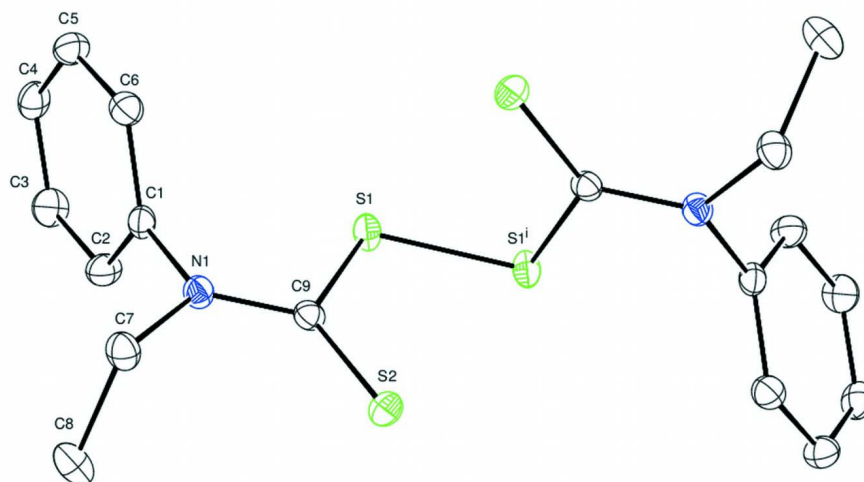


Figure 1

The asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level.

N-Ethyl-*N*- phenyl{[ethyl(phenyl)carbamothioyl]disulfanyl}carbothioamide

Crystal data

$C_{18}H_{20}N_2S_4$

$M_r = 392.6$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 15.1923 (2) \text{ \AA}$

$b = 11.5954 (2) \text{ \AA}$

$c = 12.3762 (2) \text{ \AA}$

$\beta = 115.039 (1)^\circ$

$V = 1975.31 (5) \text{ \AA}^3$

$Z = 4$

$F(000) = 824$

$D_x = 1.32 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 20252 reflections

$\theta = 2.3\text{--}28.5^\circ$

$\mu = 0.48 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, yellow

$0.4 \times 0.37 \times 0.15 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.830$, $T_{\max} = 0.931$

19720 measured reflections

2481 independent reflections

2405 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 28.4^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -20 \rightarrow 18$

$k = -15 \rightarrow 15$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.068$

$S = 1.06$

2481 reflections

110 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 1.5002P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.29 \text{ e \AA}^{-3}$

Special details

Experimental. Carbon-bound H-atoms were placed in calculated positions [C—H = 0.98 Å for Me H atoms, 0.99 Å for Methylene H atoms and 0.95 Å for aromatic H atoms; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and were included in the refining model approximation.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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. The following ALERTS were generated. Each ALERT has the format test-name_ALERT_alert-type_alert-level.

PLAT912_ALERT_4_C Missing # of FCF Reflections Above STh/L= 0.600 18 PLAT153_ALERT_1_G The su's on the Cell Axes are Equal ... 0.00020 Å ng. PLAT960_ALERT_3_G Number of Intensities with I . LT. - $2*\text{sig}(I)$... 1

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.20064 (7)	0.61036 (8)	0.15533 (8)	0.01472 (18)
C2	0.19847 (7)	0.51377 (9)	0.08775 (9)	0.01904 (19)
H2	0.143	0.4979	0.0157	0.023*
C3	0.27850 (8)	0.44056 (9)	0.12688 (10)	0.0220 (2)
H3	0.2781	0.3747	0.081	0.026*
C4	0.35898 (8)	0.46347 (9)	0.23282 (10)	0.0204 (2)
H4	0.4137	0.4136	0.259	0.025*
C5	0.35949 (8)	0.55942 (9)	0.30067 (9)	0.0201 (2)
H5	0.4141	0.5739	0.3739	0.024*
C6	0.28059 (7)	0.63409 (9)	0.26191 (9)	0.01764 (19)
H6	0.2812	0.7003	0.3075	0.021*
C7	0.12241 (7)	0.78155 (9)	0.03173 (9)	0.01789 (19)
H7A	0.1905	0.8075	0.0581	0.021*
H7B	0.0839	0.8481	0.0378	0.021*
C8	0.08306 (9)	0.74308 (10)	-0.09751 (10)	0.0250 (2)
H8A	0.1227	0.6795	-0.1047	0.038*
H8B	0.0853	0.8078	-0.1472	0.038*
H8C	0.0157	0.7171	-0.1241	0.038*
C9	0.04038 (7)	0.67095 (8)	0.13196 (8)	0.01398 (18)
N1	0.11909 (6)	0.68889 (7)	0.11179 (7)	0.01472 (16)
S1	0.060555 (17)	0.55219 (2)	0.23600 (2)	0.01695 (8)
S2	-0.060995 (17)	0.74667 (2)	0.07505 (2)	0.01764 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0137 (4)	0.0164 (4)	0.0163 (4)	0.0020 (3)	0.0085 (3)	0.0022 (3)
C2	0.0173 (4)	0.0203 (5)	0.0180 (4)	0.0012 (4)	0.0059 (4)	-0.0024 (4)
C3	0.0231 (5)	0.0197 (5)	0.0247 (5)	0.0045 (4)	0.0116 (4)	-0.0020 (4)
C4	0.0171 (4)	0.0211 (5)	0.0250 (5)	0.0055 (4)	0.0107 (4)	0.0056 (4)

C5	0.0163 (5)	0.0234 (5)	0.0180 (4)	0.0005 (4)	0.0048 (4)	0.0032 (4)
C6	0.0186 (4)	0.0184 (4)	0.0164 (4)	-0.0001 (4)	0.0078 (4)	-0.0005 (3)
C7	0.0202 (5)	0.0167 (4)	0.0188 (4)	0.0004 (4)	0.0102 (4)	0.0032 (4)
C8	0.0332 (6)	0.0274 (5)	0.0183 (5)	0.0015 (4)	0.0146 (5)	0.0031 (4)
C9	0.0152 (4)	0.0147 (4)	0.0121 (4)	0.0013 (3)	0.0058 (3)	-0.0007 (3)
N1	0.0151 (4)	0.0154 (4)	0.0150 (4)	0.0025 (3)	0.0075 (3)	0.0020 (3)
S1	0.01842 (13)	0.01685 (13)	0.01994 (13)	0.00345 (8)	0.01235 (10)	0.00384 (8)
S2	0.01423 (13)	0.02036 (14)	0.01803 (13)	0.00421 (8)	0.00654 (10)	0.00127 (8)

Geometric parameters (Å, °)

C1—C2	1.3898 (13)	C7—N1	1.4768 (12)
C1—C6	1.3910 (13)	C7—C8	1.5183 (15)
C1—N1	1.4454 (12)	C7—H7A	0.99
C2—C3	1.3910 (14)	C7—H7B	0.99
C2—H2	0.95	C8—H8A	0.98
C3—C4	1.3884 (15)	C8—H8B	0.98
C3—H3	0.95	C8—H8C	0.98
C4—C5	1.3920 (15)	C9—N1	1.3372 (12)
C4—H4	0.95	C9—S2	1.6495 (9)
C5—C6	1.3892 (14)	C9—S1	1.8205 (9)
C5—H5	0.95	S1—S1 ⁱ	2.0112 (5)
C6—H6	0.95		
C2—C1—C6	121.25 (9)	N1—C7—H7A	109.1
C2—C1—N1	119.01 (8)	C8—C7—H7A	109.1
C6—C1—N1	119.71 (9)	N1—C7—H7B	109.1
C1—C2—C3	119.19 (9)	C8—C7—H7B	109.1
C1—C2—H2	120.4	H7A—C7—H7B	107.8
C3—C2—H2	120.4	C7—C8—H8A	109.5
C4—C3—C2	120.17 (10)	C7—C8—H8B	109.5
C4—C3—H3	119.9	H8A—C8—H8B	109.5
C2—C3—H3	119.9	C7—C8—H8C	109.5
C3—C4—C5	120.03 (9)	H8A—C8—H8C	109.5
C3—C4—H4	120	H8B—C8—H8C	109.5
C5—C4—H4	120	N1—C9—S2	125.78 (7)
C6—C5—C4	120.41 (9)	N1—C9—S1	110.70 (7)
C6—C5—H5	119.8	S2—C9—S1	123.49 (6)
C4—C5—H5	119.8	C9—N1—C1	121.70 (8)
C5—C6—C1	118.93 (9)	C9—N1—C7	121.70 (8)
C5—C6—H6	120.5	C1—N1—C7	116.18 (8)
C1—C6—H6	120.5	C9—S1—S1 ⁱ	103.26 (3)
N1—C7—C8	112.52 (8)		
C6—C1—C2—C3	-0.94 (15)	S2—C9—N1—C7	-0.83 (13)
N1—C1—C2—C3	177.36 (9)	S1—C9—N1—C7	-179.17 (7)
C1—C2—C3—C4	0.63 (16)	C2—C1—N1—C9	85.13 (12)
C2—C3—C4—C5	0.48 (16)	C6—C1—N1—C9	-96.55 (11)

C3—C4—C5—C6	-1.31 (16)	C2—C1—N1—C7	-87.62 (11)
C4—C5—C6—C1	1.01 (15)	C6—C1—N1—C7	90.70 (11)
C2—C1—C6—C5	0.12 (15)	C8—C7—N1—C9	-87.47 (11)
N1—C1—C6—C5	-178.16 (9)	C8—C7—N1—C1	85.28 (11)
S2—C9—N1—C1	-173.18 (7)	N1—C9—S1—S1 ⁱ	174.72 (6)
S1—C9—N1—C1	8.48 (11)	S2—C9—S1—S1 ⁱ	-3.66 (7)

Symmetry code: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

Cg is the centroid of the C1—C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8B \cdots Cg ⁱⁱ	0.98	2.97	3.7972 (14)	143

Symmetry code: (ii) $-x+1/2, -y+3/2, -z$.