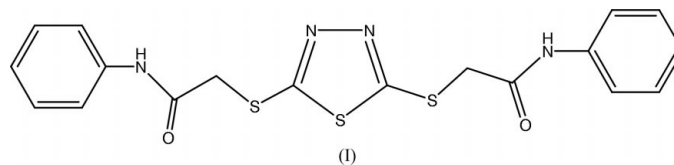


Yong-Hong Wen, Shu-Sheng
Zhang,* Bao-Hui Yu, Xue-Mei
Li and Qing LiuCollege of Chemistry and Molecular
Engineering, Qingdao University of Science
and Technology, 266042 Qingdao, Shandong,
People's Republic of ChinaCorrespondence e-mail:
zhangshush@public.qd.sd.cn

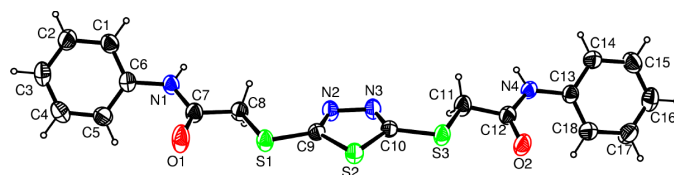
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.039
 wR factor = 0.102
Data-to-parameter ratio = 14.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N,N'*-Diphenyl-2,2'-(1,3,4-thiadiazolyl-2,5-dithio)-
diacetamideIn the title compound, $\text{C}_{18}\text{H}_{16}\text{N}_4\text{O}_2\text{S}_3$, molecules are linked
into ribbons along the b axis through intermolecular $\text{N}-\text{H}\cdots\text{O}$
interactions. The packing is further stabilized by $\text{C}-\text{H}\cdots\pi$
interactions.Received 4 January 2005
Accepted 14 January 2005
Online 22 January 2005

Comment

2,5-Dimercapto-1,3,4-thiadiazole (DMTD) is an effective
stabilizer for emulsions, and its derivatives can be absorbed by
plant cells, so they can be prepared as bactericides, herbicides
and insecticides, *etc* (Daniela *et al.*, 2003; Gurn, 2001). In this
paper, a new DMTD derivative of open-chain crown-ether type,
(I), was synthesized and an X-ray crystal structure
undertaken to elucidate its molecular conformation (Fig. 1).The bond lengths and angles in (I) are within normal ranges
(Allen *et al.*, 1987) and comparable with those in a related
compound (Zhang *et al.*, 2005). Owing to π -conjugation, the
bond lengths in the thiadiazole ring show intermediate char-
acter between single and double bonds. The molecule is non-
planar, with dihedral angles of $51.62(6)$ and $55.82(6)^\circ$
between the thiadiazole and the two phenyl rings; the two
phenyl rings make a dihedral angle of $5.94(14)^\circ$ with each
other.In the crystal structure, molecules of (I) are linked into
ribbons along the b axis by intermolecular $\text{N1}-\text{H1}\cdots\text{O1}^i$ and
 $\text{N4}-\text{H4}\cdots\text{O2}^i$ interactions (Fig. 2; symmetry code as in
Table 1). The packing is further stabilized by $\text{C}-\text{H}\cdots\pi$
interactions (Fig. 3 and Table 1).

Experimental

2-Chloro-*N*-phenylacetamide was prepared by the reaction of aniline
and chloroacetyl chloride in the presence of triethylamine according
to a literature method (Li, 2001). To a solution of 2,5-dimercapto-**Figure 1**
The structure of the compound (I), showing 50% probability displace-
ment ellipsoids and the atom-numbering scheme.

1,3,4-thiadiazole (1.50 g, 10 mmol) in acetone (40 ml) were added 2-chloro-*N*-phenylacetamide (3.36 g, 20 mmol), K_2CO_3 (3.04 g, 22 mmol) and NaI (0.5 g), successively, and the resulting mixture was stirred at 329 K for 2 h. After cooling to room temperature, the mixture was washed three times with water and then filtered. The filtered solid was washed three times with acetone. The title compound was obtained after drying of the white powder at room temperature for 48 h. Colorless single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethanol solution over a period of 10 d.

Crystal data

$C_{18}H_{16}N_4O_2S_3$

$M_r = 416.53$

Monoclinic, $P2_1/c$

$a = 20.6438$ (19) Å

$b = 5.3153$ (5) Å

$c = 16.7247$ (15) Å

$\beta = 97.039$ (2)°

$V = 1821.3$ (3) Å³

$Z = 4$

$D_x = 1.519$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 3299

reflections

$\theta = 2.5$ – 26.0 °

$\mu = 0.43$ mm⁻¹

$T = 293$ (2) K

Plate, colorless

$0.34 \times 0.27 \times 0.04$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.853$, $T_{\max} = 0.977$

9757 measured reflections

3604 independent reflections

2949 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 26.1$ °

$h = -21 \rightarrow 25$

$k = -6 \rightarrow 6$

$l = -20 \rightarrow 18$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.102$

$S = 1.05$

3604 reflections

244 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.4102P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O1^i$	0.86	2.32	3.110 (2)	152
$N4-H4\cdots O2^i$	0.86	2.31	3.100 (2)	152
$C11-H11A\cdots Cg1^{ii}$	0.97	2.82	3.713 (2)	154

Symmetry codes: (i) $x, y - 1, z$; (ii) $1 - x, -y, 1 - z$. Cg1 denotes the centroid of the C1–C6 phenyl ring.

All H atoms were placed in calculated positions ($N-H = 0.86$, $C-H = 0.93$ – 0.97 Å) and constrained to ride on their parent atoms, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(N, C)$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: ORTEP-3 for Windows (Farrugia, 1997), PLATON (Spek, 2003) and CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON.

This project was supported by the National Natural Science Foundation of China (Nos. 20275020 and 20475030) and the Outstanding Adult–Young Scientific Research Encouraging Foundation of Shandong Province (No. 03BS081).

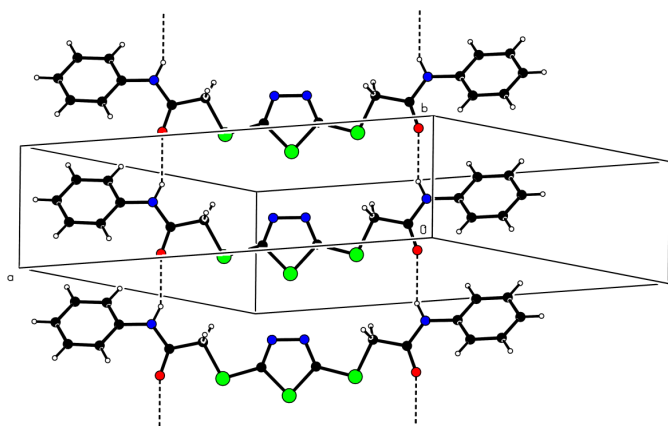


Figure 2

Packing diagram (PLATON; Spek, 2003) of the title compound, showing the formation of ribbons along the b axis. Dashed lines indicate hydrogen bonds.

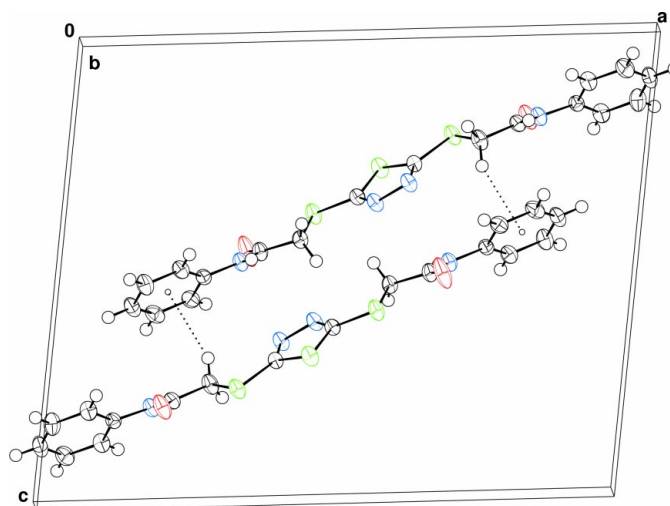


Figure 3

CAMERON (Watkin *et al.*, 1993) view of the dimer resulting from $C-H\cdots\pi$ interactions (dashed lines).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Daniela, V., Marco, F. & Enzo, G. (2003). *Bioorg. Med. Chem. Lett.* **13**, 1005–1009.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gurn, N. (2001). *J. Sci. Ind. Res.* **60**, 601–605.
- Li, Z. G. (2001). *Preparation of Organic Intermediates*, pp. 103–107. Beijing: Chemical Industry Press.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXTL. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Watkin, D. M., Pearce, L. & Prout, C. K. (1993). CAMERON. Chemical Crystallography Laboratory, University of Oxford, England.
- Zhang, S.-S., Wen, Y.-H., Yu, B.-H. & Li, X.-M. (2005). *J. Heterocycl. Chem.* Submitted.