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(3,5-Bis(phenylamino)-1,2-dithiolan-4-yl)-(2,5-dichlorophenyl)methanone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.034
wR factor = 0.091
Data-to-parameter ratio = 13.7

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

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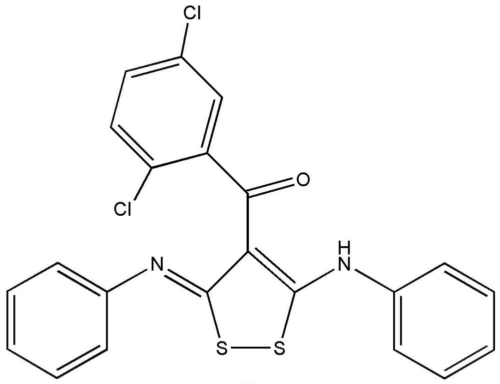
In the title molecule, $C_{22}H_{14}Cl_2N_2OS_2$, all bond lengths and angles are within normal ranges. Intermolecular N—H⋯O hydrogen bonds link the molecules into centrosymmetric dimers. The crystal packing is stabilized by van der Waals forces.

Comment

Many heterocyclic compounds are known as potentially important pharmaceuticals in view of their bioactivities (Shi *et al.*, 1995; Xu *et al.*, 2002). In our search for new biologically active heterocyclic compounds, the title compound, (I), was synthesized. Here we report its crystal structure.



(I)

In (I), the bond lengths and angles in the 1,2-dithiole ring (Table 1) are in good agreement with earlier reported data (By *et al.*, 1992). The 1,2-dithiole ring is essentially planar, with a maximum deviation from the mean plane of 0.024 (3) Å for C1. The mean planes of N1/C11–C16, N2/C17–C22 and Cl1/Cl2/C5–C10 make dihedral angles of 74.4 (2), 84.1 (3) and 76.7 (3)°, respectively, with the 1,2-dithiole ring. The molecular conformation is determined by the intramolecular N—H⋯O hydrogen bond (Table 2). Intermolecular N—H⋯O hydrogen bonds (Table 2) link the molecules into centrosymmetric dimers. The crystal packing (Fig. 2) is stabilized by van der Waals forces.

Experimental

A mixture of phenyl isothiocyanate (2.7 g, 0.02 mol), 2-bromo-1-(2,5-dichlorophenyl)ethanone (1.95 g, 0.01 mol), powdered potassium hydroxide (0.05 mol) and acetone (50 ml) was stirred for 1 h at room temperature. The solution was then filtered, concentrated and purified by recrystallization to afford the title compound (yield 3.98 g, 87%). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethyl acetate at room temperature.

Crystal data

$C_{22}H_{14}Cl_2N_2OS_2$
$M_r = 457.37$
Triclinic, $P\overline{1}$
$a = 10.0686\ (18)$ Å
$b = 10.3118\ (19)$ Å
$c = 10.3920\ (19)$ Å
$\alpha = 87.470\ (3)°$
$\beta = 85.492\ (2)°$
$\gamma = 78.284\ (2)°$
$V = 1052.8\ (3)$ Å^3
$Z = 2$
$D_x = 1.443$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1634 reflections
$\theta = 2.8$–$25.6°$
$\mu = 0.52$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
$0.20 \times 0.18 \times 0.14$ mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.887$, $T_{max} = 0.929$
5699 measured reflections
3656 independent reflections
2602 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -7 \rightarrow 12$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.092$
$S = 1.08$
3656 reflections
266 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.0711P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.7981 (19)	N2—C3	1.328 (2)
S1—S2	2.0627 (9)	N2—C17	1.432 (3)
S2—C3	1.7404 (19)	C1—C2	1.454 (3)
N1—C1	1.260 (2)	C2—C3	1.392 (3)
N1—C11	1.414 (2)	C2—C4	1.430 (3)
C1—S1—S2	96.48 (7)	C3—N2—C17	123.63 (18)
C3—S2—S1	95.17 (7)	C3—C2—C1	116.78 (17)
C1—N1—C11	119.59 (17)		
C1—S1—S2—C3	2.10 (10)	C17—N2—C3—C2	174.1 (2)
C11—N1—C1—C2	−178.05 (18)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.89	1.95	2.619 (3)	130
N2—H2···O1^{i}	0.89	2.17	2.898 (3)	138

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

All H atoms were placed in calculated positions, with N—H = 0.89 Å and C—H = 0.93 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

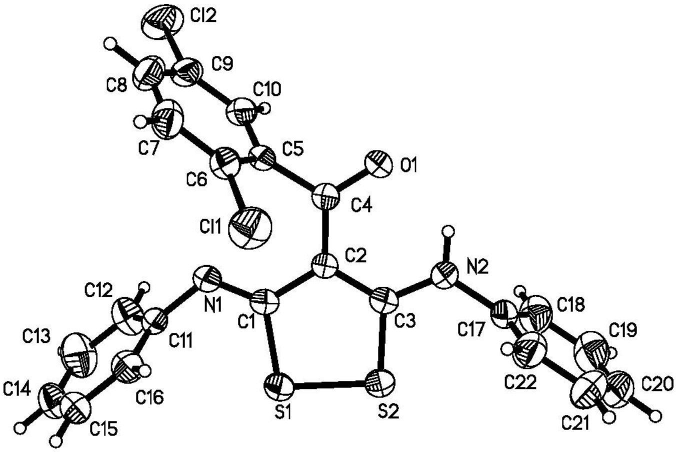


Figure 1
View of (I), with displacement ellipsoids drawn at the 40% probability level.

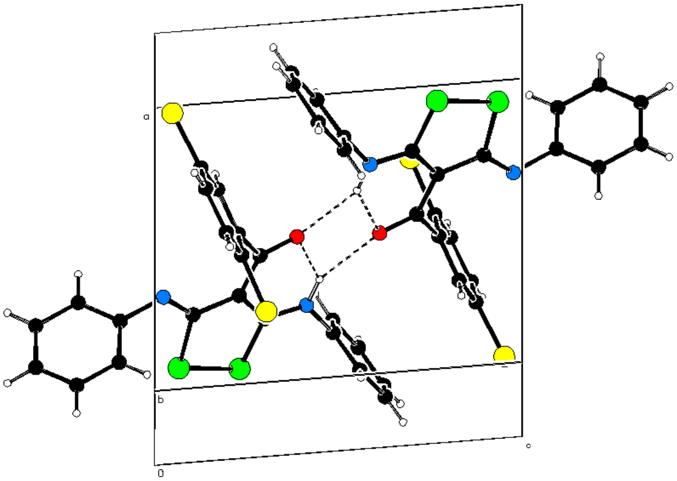


Figure 2
The crystal packing, showing the hydrogen-bonded (dashed lines) centrosymmetric dimer.

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