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N^S,N^O-Diphenyl(thiooxamide)

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The molecule of the title compound, PhNHC(=S)-C(=O)NHPh or $C_{14}H_{12}N_2OS$, consists of two planar fragments, one of which includes the central NHC(=S)-C(=O)NH chain together with the Ph substituent on the C=O side; the second Ph ring, the one on the C=S side, all by itself, makes up the second planar fragment. Its plane is twisted about the N—C bond by 52.87 (9)° with respect to the plane of the first fragment. The C=S and C=O double bonds adopt the *transoid* conformation with the torsion angle S=C—C=O equal to −179.6 (2)°. The molecules in the crystal are linked into centrosymmetric dimers due to the N—H···O hydrogen bond involving the thioamide NH group.

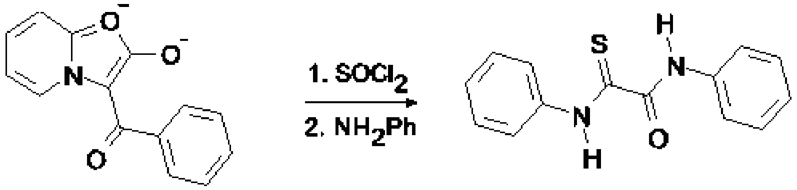
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.117
Data-to-parameter ratio = 9.7

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

Comment

The molecular structure of the title compound is shown in Fig. 1. The molecule consists of two planar fragments: the C9–C14 phenyl ring makes up one of them and the C3–C8 phenyl ring together with the N1—C1(O1)—C2(S1)—N2 chain atoms attached to C3 forms the other one. The intermolecular hydrogen bond [N2···O1^i 2.42 (3) Å, N2···O1^i 3.136 (4) Å and N2—H2···O1^i 148 (3)°; symmetry code: (i) $1-x$, $1-y$, $-z$] links the molecules in the crystal into centrosymmetric dimers. In the thiooxamide part of the molecule, the S and O atoms are *trans* with respect to each other; the torsion angle S1—C2—C1—O1 is −179.6 (2)°. The conjugation between the thioamide and amide moieties of the molecule is weak, as the C1—C2 bond [1.532 (4) Å] is significantly longer than the standard Csp^2—Csp^2 bond length in conjugated systems (1.46–1.48 Å; Allen *et al.*, 1987). The bond distances C1=O1 [1.231 (3) Å] and N1—C3 [1.413 (4) Å] are longer than the standard bond lengths for C=O (1.22 Å) and C_{Ar}—Nsp^2 (1.36 Å) (Allen *et al.*, 1987). This effect may be explained by a weak delocalization of electron density in the amide group. The bond angle C1—N1—C3 has an abnormal value of 131.5 (3)°. This large bond angle may be explained by steric strain in the planar fragment O1—C1—N1—C3—C4—H4. The second phenyl ring (C9—C14) is twisted about the N2—C9 bond with respect to the planar moiety N2—C2(S1)—C1(O1)—N1–(C3—C8) by 52.87 (9)°. Due to this twisting, the C2—N2—C9 bond angle is not distorted and has a generally accepted value of 126.1 (3)°. Only one related structure



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(Krayushkin *et al.*, 1996) (with *n*-butyl instead of phenyl as in our case) was found in the Cambridge Structural Database (Allen & Kennard, 1993). The main structural features of this molecule are essentially identical with those of the title compound.

Experimental

A mixture of 0.5 g (2 mmol) 3-benzoxazolo[3,2-*a*]pyridinium-2-olate and 10 ml thionyl chloride ($SOCl_2$) was kept under reflux for 1 h. Thionyl chloride was evaporated and the precipitate obtained was washed with benzene (3 × 10 ml) and dissolved in dichloromethane CH_2Cl_2 (20 ml). 0.4 g (4.3 mmol) aniline (C_6H_7N) was added to this solution. After heating at 313 K for 1 h and leaving to stand overnight at room temperature, the dichloromethane was evaporated, and the residue washed with water (3 × 20 ml) and recrystallized from a mixture of chloroform and diethyl ether (4:1). The yield was 0.2 g (37%).

Crystal data

$C_{14}H_{12}N_2OS$
$M_r = 256.32$
Monoclinic, $P2_1/c$
$a = 4.026\ (5)$ Å
$b = 14.682\ (9)$ Å
$c = 20.728\ (12)$ Å
$\beta = 90.27\ (2)^\circ$
$V = 1225.1\ (17)$ Å^3
$Z = 4$

$D_x = 1.390$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 13.0$–15.0°
$\mu = 0.25$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.38 × 0.12 × 0.06 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
2089 measured reflections
2051 independent reflections
1287 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.070$
$\theta_{\rm max} = 25.0^\circ$

$h = -4 \rightarrow 4$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 24$
2 standard reflections every 200 reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.117$
$S = 0.95$
2051 reflections
212 parameters

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.048$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.660 (3)	N1—H1	0.85 (3)
O1—C1	1.231 (3)	C2—N2	1.310 (4)
C1—N1	1.316 (4)	N2—C9	1.428 (4)
C1—C2	1.532 (4)	N2—H2	0.81 (3)
N1—C3	1.413 (4)		
O1—C1—N1	126.9 (3)	C1—C2—S1	121.6 (2)
O1—C1—C2	119.3 (3)	C2—N2—C9	126.1 (3)
N1—C1—C2	113.7 (2)	C4—C3—N1	123.0 (3)
C1—N1—C3	131.5 (3)	C8—C3—N1	117.0 (3)
N2—C2—C1	112.1 (2)	C14—C9—N2	120.2 (3)
N2—C2—S1	126.3 (2)	C10—C9—N2	119.2 (3)

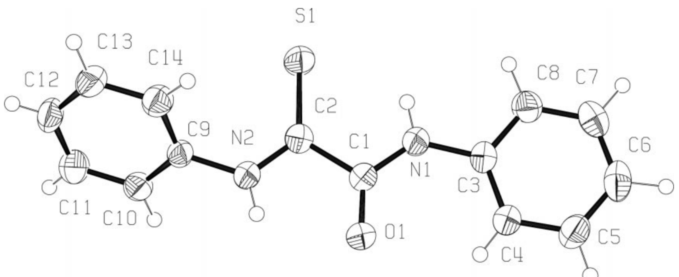

Figure 1
ORTEP-3 (Farrugia, 1998) view of the molecule of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1	0.85 (3)	2.38 (3)	2.938 (3)	124 (3)
N2—H2···O1	0.81 (3)	2.17 (4)	2.632 (4)	116 (3)
C4—H4···O1	0.88 (3)	2.45 (3)	2.977 (4)	119 (3)
N2—H2···O1$^{\rm i}$	0.81 (3)	2.42 (3)	3.136 (4)	148 (3)
C14—H14···S1	0.93 (3)	2.97 (3)	3.250 (4)	99 (2)

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

All H atoms were refined isotropically; the C—H bonds are in the range 0.82–1.00 Å.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *WinGX*98 (Farrugia, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1998).

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