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N-(*N*-Phenylbenzimidoyl)thiobenzanilide

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.153
Data-to-parameter ratio = 17.1

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

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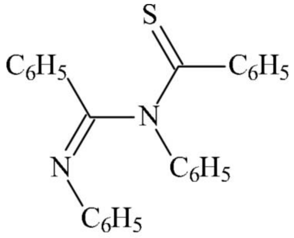
The oxidation of thiobenzanilide by lead(IV) tetraacetate yields bis(*N*-phenylbenzimidoyl) disulfide and *N*-(*N*-phenylbenzimidoyl)thiobenzanilide, $C_{26}H_{20}N_2S$. The latter molecule consists of a PhN═C(Ph)— fragment that is linked to a PhC(S)—N(Ph)— fragment by a C—N single bond [1.410 (3) Å]. The two fragements are aligned at approximately 90° to one another.

Comment

Symmetrical bis(*N*-arylimidoyl) disulfides are generally prepared by the oxidation of *N*-arylthioamides with iodine (Schaeffer *et al.*, 1967); for bis(*N*-phenylbenzimidoyl) disulfide, lead(IV) tetraacetate is a better reagent (Hahn *et al.*, 1955). In our hands, the reagent along with calcium carbonate led to an improved yield; however, in addition to bis(*N*-phenylbenzimidoyl) sulfide, a small quantity of an imine compound (as identified by its IR spectrum) was isolated; its structure is reported here.



(I)

The molecular structure of the title compound, (I), is illustrated in Fig. 1, and selected bond distances and angles are given in Table 1. The molecule consists of a PhN═C(Ph)— fragment linked to a PhC(S)—N(Ph)— fragment by a C—N single bond [1.410 (3) Å]. The two fragements are aligned at approximately 90° to one another, the torsion angle N2—C26—N1—C25 being −115.3 (3)°. A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) did not find any examples of a compound having the XN═C(X)—N(X)—C(S)X molecular fragment.

Experimental

To a stirred solution of thiobenzanilide (1 g, 5 mmol) and lead(IV) tetraacetate (2.43 g, 5.5 mmol) in anhydrous diethyl ether (200 ml) was added calcium carbonate (0.8 g, 8 mmol). The reaction was monitored by thin-layer chromatography. After the thiobenzanilide had reacted, the excess oxidant was removed with ethylene glycol, and the residue neutralized with aqueous sodium bicarbonate. The ether layer was dried over sodium sulfate and evaporated to yield a dark-yellow residue. The residue was dissolved in benzene and chromatographed on a silica-gel column by gradual elution with diethyl ether (0 to 10%) in benzene. The CHN elemental percentages corresponded to bis(*N*-phenylbenzimidoyl) disulfide ($C_{26}H_{20}N_2S_2$; m.p. 375–377 K), which did not have an imine stretch band in the IR spectrum. Elution of the residue with a diethyl ether–benzene (5:95 *v*/*v*) mixture afforded the title compound, (I), which was recrystallized from an acetone–methanol mixture.

Crystal data

$C_{26}H_{20}N_2S$
$M_r = 392.50$
Monoclinic, $P2_1/c$
$a = 11.0162$ (12) Å
$b = 19.980$ (2) Å
$c = 9.5802$ (10) Å
$\beta = 99.092$ (3)°
$V = 2082.1$ (4) Å^3
$Z = 4$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 750 reflections
$\theta = 2.8$–19.2°
$\mu = 0.17$ mm^{-1}
$T = 295$ (2) K
Block, yellow
0.2 × 0.1 × 0.1 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
12173 measured reflections
3659 independent reflections
2236 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\rm max} = 25.0°$
$h = -9 \rightarrow 11$
$k = -21 \rightarrow 23$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.153$
$S = 1.01$
3659 reflections
214 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0747P)^2 + 0.3122P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C25	1.656 (3)	N2—C26	1.271 (3)
N1—C25	1.361 (3)	N2—C13	1.410 (3)
N1—C7	1.431 (2)	C1—C25	1.483 (3)
N1—C26	1.458 (3)	C19—C26	1.483 (3)
C25—N1—C7	125.7 (2)	C18—C13—N2	121.0 (2)
C25—N1—C26	119.3 (2)	C20—C19—C26	118.2 (2)
C7—N1—C26	114.6 (2)	C24—C19—C26	121.8 (2)
C26—N2—C13	121.4 (2)	N1—C25—C1	118.9 (2)
C2—C1—C25	118.1 (2)	N1—C25—S1	120.2 (2)
C6—C1—C25	121.9 (2)	C1—C25—S1	120.9 (2)
C8—C7—N1	118.2 (2)	N2—C26—N1	122.3 (3)
C12—C7—N1	121.7 (2)	N2—C26—C19	120.1 (2)
C14—C13—N2	118.9 (2)	N1—C26—C19	117.2 (2)

The phenyl rings were refined as rigid hexagons (aromatic C—C bond lengths = 1.39 Å) in order to increase the reflection-to-variable ratio above 10. The H atoms were positioned geometrically [C—H = 0.93 Å and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$] and were included in the refinement in the riding-model approximation.

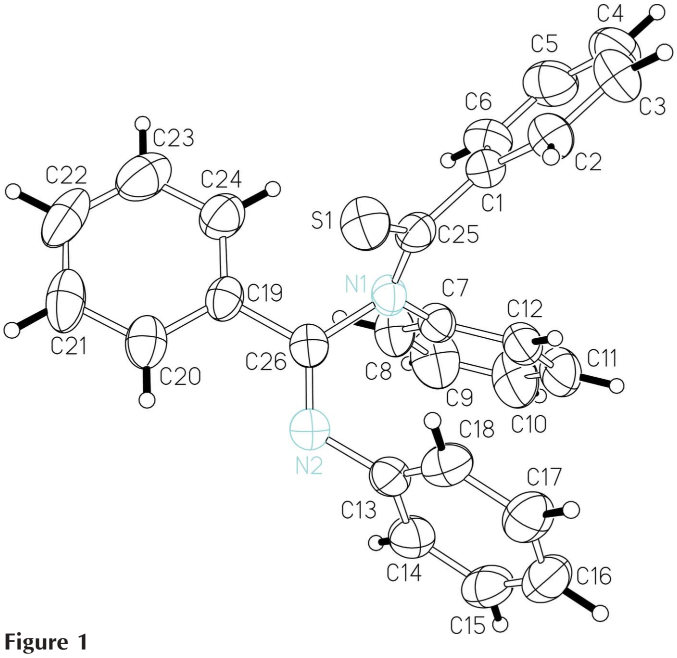


Figure 1
ORTEPII (Johnson, 1976) plot of compound (I), with displacement ellipsoids at the 50% probability level.

Data collection: *SMART* (Bruker, 1995); cell refinement: *SAINT* (Bruker, 1995); data reduction: *SAINT*; 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*.

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