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1-(4-Nitrobenzoyl)-3-phenylthiourea

Aamer Saeed[a]* and Michael Bolte[b]

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan, and [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: aamersaeed@yahoo.com

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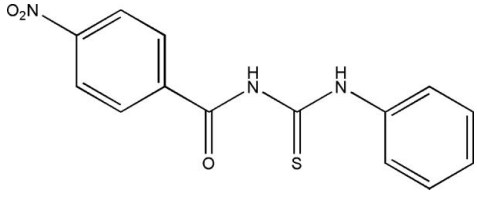
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.086; data-to-parameter ratio = 12.9.

Geometric parameters of the title compound, $C_{14}H_{11}N_3O_3S$, are in the usual ranges. The molecular conformation is stabilized by an N—H···O hydrogen bond. In the crystal structure, the molecules form centrosymmetric dimers connected by N—H···S hydrogen bonds. The molecule is almost planar (r.m.s. deviation for all atoms = 0.268 Å); only the torsion angles about the C—C bond between the carbonyl group and the nitrophenyl ring [O—C—C—C = −157.92 (16)°] and about the C—N bond between the phenyl ring and the amide group [C—N—C—C = 15.9 (3)°] differ significantly from 0 or 180°.

Related literature

For related literature, see: Koch (2001); Krishnamurthy *et al.* (1999); Saeed *et al.* (2007); Saeed & Pervez (2006); Sijia *et al.* (2003); Zeng *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{11}N_3O_3S$
M_r = 301.32
Monoclinic, $P2_1/c$
a = 8.2660 (10) Å
b = 12.1458 (9) Å
c = 13.6687 (16) Å
β = 92.651 (10)°
V = 1370.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 173 (2) K
0.36 × 0.35 × 0.33 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.915, T_{max} = 0.922
7976 measured reflections
2552 independent reflections
2189 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.086
S = 1.02
2552 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.85 (2)	2.68 (2)	3.4386 (14)	148.6 (17)
N2—H2···O1	0.86 (2)	1.93 (2)	2.6673 (16)	143 (2)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2340).

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supplementary materials

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1-(4-Nitrobenzoyl)-3-phenylthiourea

A. Saeed and M. Bolte

Comment

1-Aroyl-3-arylthioureas are extremely versatile building blocks for the synthesis of a diversity of heterocyclic compounds: imidazole-2-thiones (Zeng *et al.*, 2003) and 1,3-thiazolines (Saeed & Pervez, 2006) and 2-aroylimino-3-aryl-thiazolidin-4-ones (Saeed *et al.*, 2007). *N*,*N*-Dialkyl-*N*-aroylthioureas are efficient ligands for the separation of platinum group metals (Koch, 2001). 1,3-Dialkyl- or diarylthioureas have shown significant antifungal activity against plant pathogens Pyricularia oryzae and Drechslera oryzae (Krishnamurthy *et al.*, 1999) and 1-benzoyl-3-(4,6-disubstituted-pyrimidine-2-yl)thioureas have shown excellent herbicidal activity (Sijia *et al.*, 2003).

Geometric parameters of the title compound in Fig. 1 are in the usual ranges. The molecular conformation is stabilized by an N—H···O hydrogen bond. In the crystal of the title compound, the molecules form centrosymmetric dimers connected by N—H···S hydrogen bonds (Fig. 2).

Experimental

To a suspension of potassium thiocyanate (0.97 g, 10 mmol) in acetone (30 ml) a solution of 4-nitrobenzoyl chloride (1.85 g, 10 mmol) in acetone (40 ml) was added dropwise and the reaction mixture was refluxed for 45 min. After cooling to room temperature, a solution of aniline (0.93 g, 10 mmol) in acetone (10 ml) was added and the resulting was mixture refluxed for 2 h. The reaction mixture was poured into cold water when the thiourea was precipitated as a solid. Recrystallized from ethanol as colourless crystals (2.7 g, 9.0 mmol, 90%). m.p. 433 K. IR (KBr) cm^{-1}: 3351 (free NH), 3200 (assoc. NH), 1667 (CO), 1610 (arom.), 1529 (thioureido I) 1325 II, 1160 III, 744, 762; ^{1}H NMR ($CDCl_3$) δ: 7.31–7.75 (aromatic), 9.19 (1*H*, s, broad, NH); 12.76` (1*H*, s, broad, NH); ^{13}C NMR ($CDCl_3$) 126.2 (4 CH), 129.0 (2 CH), 129.20 (2CH), 130.7 (C), 132.1 (C),134.8 (C) 142.3 (C), 168.1 (C═O), 178.4 (C═S). EIMS m/e: 301, 168.9,126, 119, 91, 64.9. Analysis calculated for $C_{14}H_{11}N_3O_3S$: C 55.80, H 3.68, N 13.95, S 10.64%. Found: C 55.32, H 3.63, N 14.05, O 15.83, S 10.69%.

Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95Å and U_{iso}(H) = 1.2U_{eq}(C). The H atoms bonded to N were freely refined.

Figures

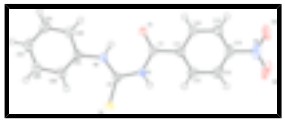

Fig. 1. Molecular structure of title compound.

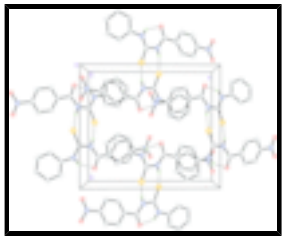

Fig. 2. Crystal packing, view onto the *bc* plane. H-atoms not involved in hydrogen bonds are omitted. Hydrogen bonds are shown as dashed lines.

1-(4-Nitrobenzoyl)-3-phenylthiourea

Crystal data

$C_{14}H_{11}N_3O_3S$	F_{000} = 624
M_r = 301.32	D_x = 1.460 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3023 reflections
a = 8.2660 (10) Å	θ = 3.5–25.7º
b = 12.1458 (9) Å	μ = 0.25 mm^{-1}
c = 13.6687 (16) Å	T = 173 (2) K
β = 92.651 (10)º	Plate, light brown
V = 1370.8 (3) Å^3	0.36 × 0.35 × 0.33 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	2552 independent reflections
Radiation source: fine-focus sealed tube	2189 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
T = 173(2) K	θ_{max} = 25.7º
ω scans	θ_{min} = 3.7º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −10→9
T_{min} = 0.915, T_{max} = 0.922	k = −14→14
7976 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.086	$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.4301P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
2552 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}

198 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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 $F^2 > 2\text{sigma}(F^2)$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.28377 (6)	0.03643 (3)	0.56649 (4)	0.04069 (15)
O1	0.43815 (14)	0.35452 (9)	0.41816 (8)	0.0331 (3)
N1	0.43245 (16)	0.16919 (11)	0.44820 (10)	0.0276 (3)
H1	0.468 (2)	0.1061 (18)	0.4329 (14)	0.041 (5)*
N2	0.28612 (16)	0.25938 (11)	0.56390 (10)	0.0272 (3)
H2	0.318 (3)	0.3148 (19)	0.5309 (15)	0.048 (6)*
N3	0.80042 (19)	0.19139 (16)	0.03823 (11)	0.0458 (4)
O3	0.8597 (2)	0.27153 (16)	0.00061 (11)	0.0732 (5)
O4	0.8076 (2)	0.09774 (16)	0.00561 (11)	0.0710 (5)
C1	0.47242 (18)	0.25966 (12)	0.39415 (11)	0.0249 (3)
C2	0.33267 (18)	0.16213 (12)	0.52899 (11)	0.0264 (3)
C11	0.55951 (18)	0.23703 (12)	0.30229 (11)	0.0246 (3)
C12	0.5533 (2)	0.13494 (13)	0.25513 (11)	0.0297 (3)
H12	0.4948	0.0760	0.2823	0.036*
C13	0.6324 (2)	0.11912 (15)	0.16862 (12)	0.0347 (4)
H13	0.6300	0.0497	0.1366	0.042*
C14	0.71447 (19)	0.20733 (15)	0.13050 (11)	0.0320 (4)
C15	0.7210 (2)	0.30996 (15)	0.17389 (12)	0.0336 (4)
H15	0.7772	0.3691	0.1451	0.040*
C16	0.64315 (19)	0.32438 (13)	0.26103 (11)	0.0293 (3)
H16	0.6468	0.3940	0.2927	0.035*
C21	0.19553 (18)	0.28671 (13)	0.64695 (11)	0.0256 (3)
C22	0.1432 (2)	0.39581 (14)	0.65039 (12)	0.0334 (4)
H22	0.1616	0.4443	0.5975	0.040*
C23	0.0637 (2)	0.43343 (15)	0.73191 (14)	0.0399 (4)
H23	0.0287	0.5078	0.7343	0.048*
C24	0.0352 (2)	0.36344 (16)	0.80929 (13)	0.0389 (4)
H24	−0.0188	0.3895	0.8646	0.047*
C25	0.0864 (2)	0.25506 (16)	0.80501 (12)	0.0360 (4)
H25	0.0666	0.2068	0.8578	0.043*

C26	0.16682 (19)	0.21546 (14)	0.72428 (11)	0.0313 (4)
H26	0.2014	0.1409	0.7222	0.038*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0497 (3)	0.0230 (2)	0.0519 (3)	0.00346 (18)	0.0294 (2)	0.00788 (19)
O1	0.0479 (7)	0.0208 (6)	0.0321 (6)	−0.0008 (5)	0.0179 (5)	−0.0017 (5)
N1	0.0356 (7)	0.0207 (7)	0.0276 (7)	0.0039 (5)	0.0134 (5)	0.0024 (5)
N2	0.0341 (7)	0.0226 (7)	0.0261 (7)	−0.0001 (5)	0.0120 (5)	0.0010 (6)
N3	0.0408 (8)	0.0710 (12)	0.0259 (7)	0.0174 (8)	0.0050 (6)	−0.0059 (8)
O3	0.0937 (13)	0.0830 (12)	0.0467 (9)	0.0268 (10)	0.0450 (9)	0.0214 (9)
O4	0.0732 (11)	0.0910 (13)	0.0504 (9)	0.0029 (9)	0.0198 (8)	−0.0404 (9)
C1	0.0283 (8)	0.0229 (8)	0.0240 (7)	−0.0011 (6)	0.0054 (6)	0.0000 (6)
C2	0.0273 (7)	0.0254 (8)	0.0271 (7)	0.0016 (6)	0.0074 (6)	0.0026 (6)
C11	0.0273 (7)	0.0243 (8)	0.0224 (7)	0.0023 (6)	0.0038 (6)	0.0006 (6)
C12	0.0370 (8)	0.0261 (8)	0.0260 (7)	0.0001 (6)	0.0023 (6)	−0.0015 (6)
C13	0.0434 (9)	0.0336 (9)	0.0269 (8)	0.0076 (7)	−0.0003 (7)	−0.0088 (7)
C14	0.0324 (8)	0.0455 (10)	0.0184 (7)	0.0116 (7)	0.0045 (6)	−0.0012 (7)
C15	0.0352 (8)	0.0380 (9)	0.0284 (8)	0.0009 (7)	0.0104 (7)	0.0053 (7)
C16	0.0368 (8)	0.0249 (8)	0.0270 (8)	−0.0002 (6)	0.0092 (6)	−0.0012 (6)
C21	0.0248 (7)	0.0281 (8)	0.0244 (7)	−0.0015 (6)	0.0064 (6)	−0.0023 (6)
C22	0.0382 (9)	0.0284 (8)	0.0343 (8)	−0.0008 (7)	0.0093 (7)	−0.0017 (7)
C23	0.0400 (9)	0.0352 (9)	0.0456 (10)	0.0029 (7)	0.0125 (8)	−0.0114 (8)
C24	0.0334 (9)	0.0512 (11)	0.0331 (9)	−0.0017 (8)	0.0119 (7)	−0.0128 (8)
C25	0.0345 (9)	0.0482 (10)	0.0261 (8)	−0.0041 (7)	0.0097 (7)	0.0007 (7)
C26	0.0321 (8)	0.0344 (9)	0.0283 (8)	0.0002 (7)	0.0092 (6)	0.0019 (7)

Geometric parameters (Å, °)

S1—C2	1.6661 (15)	C13—H13	0.9500
O1—C1	1.2345 (18)	C14—C15	1.380 (3)
N1—C1	1.373 (2)	C15—C16	1.391 (2)
N1—C2	1.4111 (18)	C15—H15	0.9500
N1—H1	0.85 (2)	C16—H16	0.9500
N2—C2	1.3373 (19)	C21—C26	1.395 (2)
N2—C21	1.4276 (19)	C21—C22	1.395 (2)
N2—H2	0.86 (2)	C22—C23	1.397 (2)
N3—O3	1.215 (2)	C22—H22	0.9500
N3—O4	1.224 (2)	C23—C24	1.386 (3)
N3—C14	1.489 (2)	C23—H23	0.9500
C1—C11	1.501 (2)	C24—C25	1.385 (3)
C11—C12	1.397 (2)	C24—H24	0.9500
C11—C16	1.399 (2)	C25—C26	1.399 (2)
C12—C13	1.390 (2)	C25—H25	0.9500
C12—H12	0.9500	C26—H26	0.9500
C13—C14	1.383 (3)		
C1—N1—C2	129.32 (13)	C13—C14—N3	118.70 (16)

C1—N1—H1	119.7 (13)	C14—C15—C16	118.10 (15)
C2—N1—H1	111.0 (13)	C14—C15—H15	120.9
C2—N2—C21	131.25 (14)	C16—C15—H15	120.9
C2—N2—H2	114.0 (14)	C15—C16—C11	120.43 (15)
C21—N2—H2	114.8 (14)	C15—C16—H16	119.8
O3—N3—O4	124.27 (16)	C11—C16—H16	119.8
O3—N3—C14	118.18 (17)	C26—C21—C22	119.99 (14)
O4—N3—C14	117.55 (18)	C26—C21—N2	124.90 (14)
O1—C1—N1	122.69 (13)	C22—C21—N2	114.98 (14)
O1—C1—C11	121.20 (13)	C21—C22—C23	119.72 (16)
N1—C1—C11	116.11 (13)	C21—C22—H22	120.1
N2—C2—N1	114.45 (13)	C23—C22—H22	120.1
N2—C2—S1	128.43 (11)	C24—C23—C22	120.73 (17)
N1—C2—S1	117.09 (11)	C24—C23—H23	119.6
C12—C11—C16	119.68 (14)	C22—C23—H23	119.6
C12—C11—C1	122.65 (13)	C25—C24—C23	119.20 (15)
C16—C11—C1	117.59 (13)	C25—C24—H24	120.4
C13—C12—C11	120.42 (15)	C23—C24—H24	120.4
C13—C12—H12	119.8	C24—C25—C26	121.15 (16)
C11—C12—H12	119.8	C24—C25—H25	119.4
C14—C13—C12	118.12 (15)	C26—C25—H25	119.4
C14—C13—H13	120.9	C21—C26—C25	119.20 (16)
C12—C13—H13	120.9	C21—C26—H26	120.4
C15—C14—C13	123.22 (14)	C25—C26—H26	120.4
C15—C14—N3	118.07 (16)		
C2—N1—C1—O1	9.0 (3)	O3—N3—C14—C13	173.47 (17)
C2—N1—C1—C11	−170.27 (15)	O4—N3—C14—C13	−7.1 (2)
C21—N2—C2—N1	−175.45 (15)	C13—C14—C15—C16	1.0 (3)
C21—N2—C2—S1	6.5 (3)	N3—C14—C15—C16	−178.89 (14)
C1—N1—C2—N2	−8.4 (2)	C14—C15—C16—C11	−0.6 (2)
C1—N1—C2—S1	169.96 (13)	C12—C11—C16—C15	−0.5 (2)
O1—C1—C11—C12	−157.92 (16)	C1—C11—C16—C15	−177.50 (15)
N1—C1—C11—C12	21.4 (2)	C2—N2—C21—C26	15.9 (3)
O1—C1—C11—C16	19.0 (2)	C2—N2—C21—C22	−168.24 (16)
N1—C1—C11—C16	−161.71 (14)	C26—C21—C22—C23	0.7 (3)
C16—C11—C12—C13	1.3 (2)	N2—C21—C22—C23	−175.42 (15)
C1—C11—C12—C13	178.13 (15)	C21—C22—C23—C24	−0.4 (3)
C11—C12—C13—C14	−0.9 (2)	C22—C23—C24—C25	−0.1 (3)
C12—C13—C14—C15	−0.3 (3)	C23—C24—C25—C26	0.3 (3)
C12—C13—C14—N3	179.65 (15)	C22—C21—C26—C25	−0.5 (2)
O3—N3—C14—C15	−6.6 (2)	N2—C21—C26—C25	175.20 (15)
O4—N3—C14—C15	172.86 (17)	C24—C25—C26—C21	0.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.85 (2)	2.68 (2)	3.4386 (14)	148.6 (17)
N2—H2···O1	0.86 (2)	1.93 (2)	2.6673 (16)	143 (2)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

Fig. 1

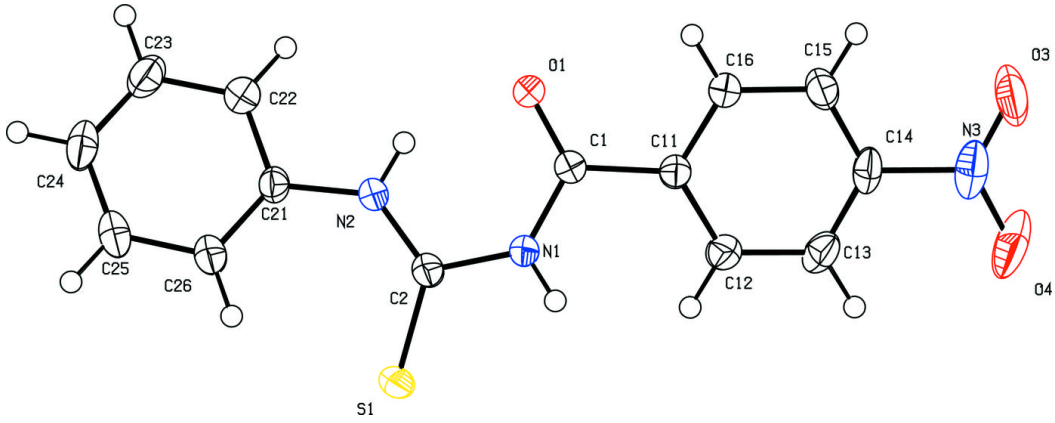
C23
C22
C24
C21
N2
O1
C1
C11
C16
C15
O3
N3
C14
C25
C26
C2
N1
C12
C13
O4
S1

Fig. 2