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N-Benzoyl-N'-phenylurea

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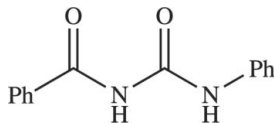
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.098; data-to-parameter ratio = 10.5.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$, the molecular conformation is determined by a strong intramolecular $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bond. In the crystal, pairs of molecules are connected by intermolecular $\text{N}-\text{H}\cdots\text{O}=\text{C}$ hydrogen bonds, forming centrosymmetric dimers. No specific interactions between dimers could be found.

Related literature

For related structures, see: Bart *et al.* (1989); Zhong *et al.* (1998); Moon *et al.* (2002), Yamin & Mardi (2003); Chen *et al.* (2004); Su (2005); Yan *et al.* (2007, 2008); Liu *et al.* (2008, 2008*a,b*). For graph-set notation, see: Etter (1990). The title compound was obtained as a byproduct during the synthesis of a copper(I) complex with *N*-benzoyl-*N'*-phenylthiourea prepared according to Frank & Smith (1948).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$
 $M_r = 240.26$
 Monoclinic, $P2_1/c$
 $a = 15.5641$ (8) Å
 $b = 4.6564$ (3) Å
 $c = 21.1029$ (15) Å
 $\beta = 128.716$ (4)°

$V = 1193.31$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 150$ K
 $0.54 \times 0.10 \times 0.09$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire2 (large Be window) detector
 Absorption correction: analytical [CrysAlis PRO (Oxford Diffraction, 2009)]; analytical numeric absorption correction using a multi-faceted crystal

model based on expressions derived by Clark & Reid (1995)]
 $T_{\min} = 0.971$, $T_{\max} = 0.993$
 4425 measured reflections
 2221 independent reflections
 1575 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.098$
 $S = 0.94$
 2221 reflections

211 parameters
 Only H-atom coordinates refined
 $\Delta\rho_{\max} = 0.22$ e Å⁻³
 $\Delta\rho_{\min} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}$	0.93 (2)	1.85 (2)	2.634 (2)	140 (1)
$\text{N2}-\text{H2}\cdots\text{O1}^i$	0.93 (2)	1.97 (2)	2.882 (1)	169 (1)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO* program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2175).

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supplementary materials

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N-Benzoyl-*N'*-phenylurea

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Comment

N-Benzoyl-*N'*-phenylurea halogeno derivatives are used as pesticides. For instance 1-(3,5-dichloro-2,4-difluorophenyl)-3-(2,6-difluorobenzoyl)urea was found to act as chitin synthesis inhibitor (Zhong *et al.*, 1998).

N-benzoyl-*N'*-phenylurea molecules adopt a conformation that allows formation of N—H···O=C intramolecular hydrogen bonds, denoted as **R**(6) in graph set notation (Etter, 1990). This conformation is commonly noted among all *N'*-mono-substituted or *N'*-unsubstituted *N*-benzoylureas and *N*-benzoylthioureas (see: related structures). Additional N—H···O=C intermolecular **R**²₂(8) hydrogen bonds bind two urea derivative molecules to form a centrosymmetric dimer (Fig. 1), which is also common. Only one known structure does not exhibit such a motif (Moon *et al.*, 2002).

No π - π stacking interactions can be found in this structure (closest ring centroids distance is about 5.60 Å with the dihedral angle between the rings α about 80°).

The dihedral angle N1—C1—N2—C2 describing the twist of two amide subunits of urea derivatives is equal to 3.0 (2)°. This value is in the range known from other studies: from 0.07° (Yan *et al.*, 2007) to 7.45° (Su, 2005).

Experimental

N-benzoyl-*N'*-phenylurea was obtained as a byproduct during the synthesis of a copper(I) complex with *N*-Benzoyl-*N'*-phenylthiourea prepared according to Frank & Smith (1948). Obviously the ligand underwent basic hydrolysis with CH₃ONa in acetone. Colourless single crystals suitable for X-ray diffraction analysis were isolated from the reaction mixture after it was kept at room temperature for a few days. From 5.12 g (0.02 mol) of thiourea derivative 0.87 g of *N*-benzoyl-*N'*-phenylurea was obtained. Yield: 18%.

Refinement

All hydrogen atoms were found from difference Fourier map and refined without constraints.

Figures

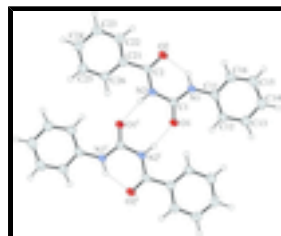


Fig. 1. Structure of centrosymmetric *N*-benzoyl-*N'*-phenylurea dimer. Ellipsoids are drawn at 50% probability level. Symmetry code: (i) $-x+1, -y+1, -z$.

N-Benzoyl-*N'*-phenylurea

Crystal data

$C_{14}H_{12}N_2O_2$	$F(000) = 504$
$M_r = 240.26$	$D_x = 1.337 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Melting point: 482(2) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 15.5641 (8) \text{ \AA}$	Cell parameters from 2481 reflections
$b = 4.6564 (3) \text{ \AA}$	$\theta = 2.5\text{--}28.4^\circ$
$c = 21.1029 (15) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 128.716 (4)^\circ$	$T = 150 \text{ K}$
$V = 1193.31 (15) \text{ \AA}^3$	Needle, colourless
$Z = 4$	$0.54 \times 0.10 \times 0.09 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire2 (large Be window) detector	2221 independent reflections
Radiation source: Mo $K\alpha$ radiation	1575 reflections with $I > 2\sigma(I)$
graphite	$R_{\text{int}} = 0.022$
Detector resolution: $8.1883 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.5^\circ$
ω scans	$h = -18 \rightarrow 9$
Absorption correction: analytical [CrysAlis Pro (Oxford Diffraction, 2009); analytical numeric absorption correction using a multi-faceted crystal model based on expressions derived by Clark & Reid (1995)]	$k = -3 \rightarrow 5$
$T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.993$	$l = -21 \rightarrow 25$
4425 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.098$	Only H-atom coordinates refined
$S = 0.94$	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2]$
2221 reflections	where $P = (F_o^2 + 2F_c^2)/3$
211 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.58625 (7)	0.6582 (2)	-0.00745 (6)	0.0384 (3)
O2	0.82393 (7)	0.2951 (2)	0.21526 (6)	0.0396 (3)
N1	0.77093 (9)	0.6517 (3)	0.09887 (7)	0.0295 (3)
N2	0.64890 (9)	0.3566 (3)	0.09771 (7)	0.0284 (3)
C1	0.66640 (10)	0.5660 (3)	0.05886 (8)	0.0274 (3)
C2	0.72519 (10)	0.2381 (3)	0.17319 (8)	0.0282 (3)
C11	0.81196 (11)	0.8530 (3)	0.07373 (9)	0.0297 (4)
C12	0.74750 (12)	0.9966 (3)	-0.00011 (9)	0.0330 (4)
C13	0.79673 (13)	1.1897 (4)	-0.01876 (10)	0.0393 (4)
C14	0.90876 (14)	1.2390 (4)	0.03479 (11)	0.0469 (5)
C15	0.97228 (13)	1.0949 (4)	0.10784 (12)	0.0516 (5)
C16	0.92476 (12)	0.9038 (4)	0.12780 (11)	0.0411 (4)
C21	0.68355 (10)	0.0351 (3)	0.20329 (8)	0.0275 (3)
C22	0.75435 (12)	-0.0253 (3)	0.28626 (9)	0.0336 (4)
C23	0.72257 (13)	-0.2128 (4)	0.31894 (10)	0.0389 (4)
C24	0.61969 (13)	-0.3423 (4)	0.26921 (10)	0.0390 (4)
C25	0.54898 (12)	-0.2851 (3)	0.18661 (10)	0.0368 (4)
C26	0.58046 (11)	-0.0960 (3)	0.15362 (9)	0.0316 (4)
H1	0.8205 (12)	0.559 (3)	0.1480 (10)	0.039 (4)*
H13	0.7502 (12)	1.291 (4)	-0.0711 (10)	0.041 (4)*
H12	0.6663 (12)	0.961 (3)	-0.0395 (8)	0.032 (4)*
H2	0.5745 (13)	0.328 (3)	0.0717 (9)	0.041 (4)*
H23	0.7751 (12)	-0.257 (3)	0.3782 (10)	0.040 (4)*
H26	0.5289 (12)	-0.059 (3)	0.0951 (9)	0.032 (4)*
H22	0.8256 (13)	0.077 (4)	0.3210 (9)	0.044 (4)*
H25	0.4762 (13)	-0.375 (3)	0.1504 (9)	0.043 (4)*
H24	0.5963 (12)	-0.477 (4)	0.2916 (9)	0.043 (4)*
H14	0.9397 (13)	1.385 (4)	0.0187 (10)	0.056 (5)*
H15	1.0520 (15)	1.117 (4)	0.1465 (10)	0.060 (5)*
H16	0.9654 (14)	0.800 (4)	0.1778 (11)	0.055 (5)*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0223 (5)	0.0518 (7)	0.0288 (6)	-0.0014 (4)	0.0100 (5)	0.0106 (5)
O2	0.0231 (5)	0.0527 (7)	0.0304 (6)	-0.0018 (5)	0.0107 (5)	0.0068 (5)
N1	0.0205 (6)	0.0363 (7)	0.0235 (6)	-0.0025 (5)	0.0097 (5)	0.0026 (6)
N2	0.0199 (6)	0.0351 (7)	0.0239 (6)	-0.0017 (5)	0.0106 (5)	0.0007 (6)
C1	0.0234 (7)	0.0333 (8)	0.0226 (7)	-0.0020 (6)	0.0130 (6)	-0.0018 (7)
C2	0.0237 (7)	0.0320 (8)	0.0243 (7)	0.0018 (6)	0.0128 (6)	-0.0015 (7)
C11	0.0275 (7)	0.0316 (8)	0.0297 (8)	-0.0041 (6)	0.0178 (6)	-0.0048 (7)
C12	0.0308 (8)	0.0373 (9)	0.0289 (8)	-0.0040 (6)	0.0178 (7)	-0.0030 (7)
C13	0.0443 (9)	0.0417 (10)	0.0360 (9)	-0.0046 (7)	0.0270 (8)	-0.0006 (8)
C14	0.0473 (10)	0.0458 (11)	0.0562 (11)	-0.0118 (8)	0.0366 (9)	-0.0009 (9)
C15	0.0303 (9)	0.0560 (12)	0.0579 (12)	-0.0089 (8)	0.0223 (9)	0.0049 (10)
C16	0.0273 (8)	0.0438 (10)	0.0413 (10)	-0.0041 (7)	0.0162 (7)	0.0051 (9)
C21	0.0279 (7)	0.0267 (8)	0.0276 (7)	0.0058 (6)	0.0173 (6)	0.0011 (7)
C22	0.0333 (8)	0.0339 (9)	0.0286 (8)	0.0037 (6)	0.0169 (7)	0.0011 (7)
C23	0.0441 (9)	0.0422 (10)	0.0296 (8)	0.0067 (7)	0.0227 (7)	0.0047 (8)
C24	0.0471 (9)	0.0367 (9)	0.0444 (9)	0.0052 (7)	0.0340 (8)	0.0080 (8)
C25	0.0357 (8)	0.0356 (9)	0.0425 (9)	0.0002 (7)	0.0261 (8)	0.0015 (8)
C26	0.0282 (7)	0.0355 (9)	0.0286 (8)	0.0033 (6)	0.0165 (7)	0.0026 (7)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.2308 (16)	C14—H14	1.007 (19)
O2—C2	1.2304 (15)	C15—C16	1.381 (2)
N1—C1	1.3419 (16)	C15—H15	0.975 (18)
N1—C11	1.4114 (18)	C16—H16	0.956 (19)
N1—H1	0.926 (16)	C21—C26	1.394 (2)
N2—C2	1.3731 (18)	C21—C22	1.3950 (19)
N2—C1	1.4058 (18)	C22—C23	1.382 (2)
N2—H2	0.930 (15)	C22—H22	0.988 (16)
C2—C21	1.494 (2)	C23—C24	1.388 (2)
C11—C12	1.388 (2)	C23—H23	0.998 (16)
C11—C16	1.3907 (19)	C24—C25	1.386 (2)
C12—C13	1.388 (2)	C24—H24	0.983 (17)
C12—H12	1.001 (14)	C25—C26	1.388 (2)
C13—C14	1.380 (2)	C25—H25	0.980 (16)
C13—H13	0.984 (17)	C26—H26	0.979 (15)
C14—C15	1.378 (3)		
C1—N1—C11	128.07 (12)	C14—C15—C16	120.59 (15)
C1—N1—H1	113.5 (9)	C14—C15—H15	122.2 (10)
C11—N1—H1	118.4 (9)	C16—C15—H15	117.2 (11)
C2—N2—C1	127.79 (11)	C15—C16—C11	120.10 (16)
C2—N2—H2	118.7 (9)	C15—C16—H16	123.5 (10)
C1—N2—H2	112.3 (10)	C11—C16—H16	116.4 (10)
O1—C1—N1	125.41 (14)	C26—C21—C22	119.31 (14)

O1—C1—N2	118.47 (11)	C26—C21—C2	123.99 (13)
N1—C1—N2	116.12 (12)	C22—C21—C2	116.69 (13)
O2—C2—N2	122.27 (13)	C23—C22—C21	120.37 (14)
O2—C2—C21	120.53 (12)	C23—C22—H22	121.3 (9)
N2—C2—C21	117.20 (12)	C21—C22—H22	118.3 (9)
C12—C11—C16	119.63 (14)	C22—C23—C24	120.06 (15)
C12—C11—N1	124.25 (12)	C22—C23—H23	119.3 (9)
C16—C11—N1	116.12 (13)	C24—C23—H23	120.7 (9)
C11—C12—C13	119.38 (14)	C25—C24—C23	120.03 (15)
C11—C12—H12	120.2 (8)	C25—C24—H24	119.1 (9)
C13—C12—H12	120.4 (8)	C23—C24—H24	120.9 (9)
C14—C13—C12	120.96 (16)	C24—C25—C26	120.07 (15)
C14—C13—H13	120.3 (9)	C24—C25—H25	121.3 (9)
C12—C13—H13	118.7 (9)	C26—C25—H25	118.6 (9)
C15—C14—C13	119.33 (16)	C25—C26—C21	120.16 (14)
C15—C14—H14	123.1 (10)	C25—C26—H26	118.5 (9)
C13—C14—H14	117.5 (10)	C21—C26—H26	121.4 (9)
N1—C1—N2—C2	3.0 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O2	0.93 (2)	1.85 (2)	2.634 (2)	140 (1)
N2—H2 \cdots O1 ⁱ	0.93 (2)	1.97 (2)	2.882 (1)	169 (1)

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

Fig. 1

