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N-[2-(Benzylideneamino)phenyl]-*N*′-phenylurea

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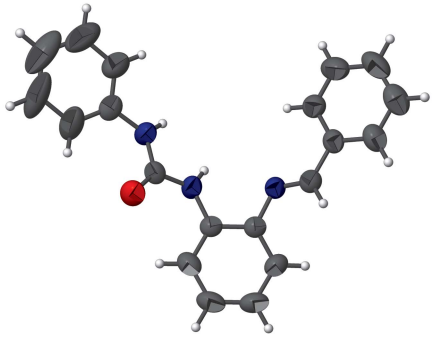
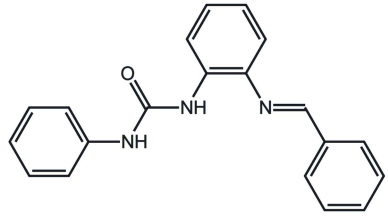
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{20}H_{17}N_3O$ [systematic name: 1-phenyl-3-{2-[(*E*)-(phenylmethylidene)amino]phenyl}urea], the middle benzene ring forms dihedral angles of 17.65 (17) and 29.48 (14)°, respectively, with the N- and C-bound phenyl rings, while the dihedral angle between the terminal rings is 46.53 (18)°. In the crystal, molecules are linked *via* N—H···O hydrogen bonds, forming helical supramolecular chains running parallel to the *c* axis *via* an $R_1^2(6)$ ring motif. The structure was refined as a two-component twin with a 0.966 (3):0.034 (3) domain ratio.

3D view



Chemical scheme



Structure description

Schiff bases, also known as imines or azomethines, are nitrogen analogues of aldehydes or ketones in which the carbonyl group has been replaced by an imine or azomethine group (Ghose, 1983). The classical synthesis reported by Schiff involves the condensation of a carbonyl compound with an amine. Various Schiff base-containing compounds are widely used owing to their antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral and antipyretic properties. In addition to the aforementioned activities, Schiff bases are used as pigments and dyes, catalysts, intermediates in organic synthesis and as polymer stabilizers (da Silva *et al.*, 2011). In this study, starting from *o*-phenylenediamine, we synthesized a new hybrid molecule containing imine and urea functionalities in the same molecule.

Referring to Fig. 1, the middle benzene ring (C8–C13) make dihedral angles of 17.65 (17) and 29.48 (14)°, respectively, with the terminal phenyl rings (C1–C6 and C15–C20). The dihedral angle between the latter is 46.53 (18)°. All bond lengths and angles

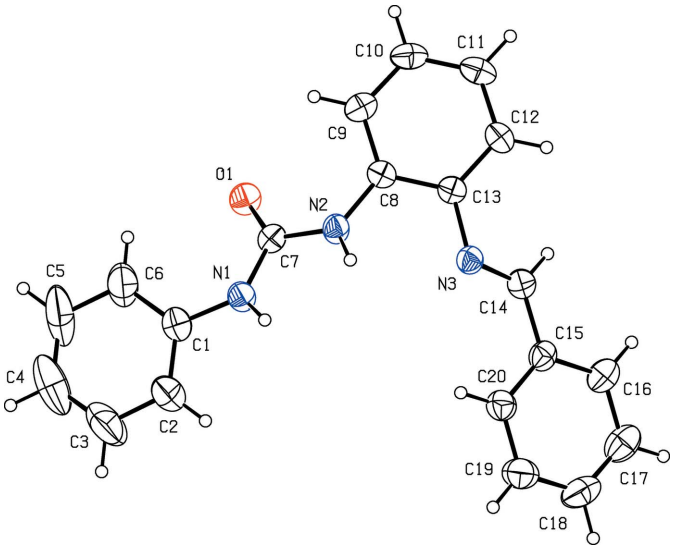


Figure 1
View of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

are within normal ranges and are in agreement with those reported for 1-(2-aminophenyl)-3- phenylurea (Mague *et al.*, 2015).

In the crystal, N—H···O hydrogen bonds link molecules into chains running parallel to the *c* axis with an $R_1^2(6)$ ring motif, Fig. 2 and Table 1.

Synthesis and crystallization

To a solution of [*N*-(2-aminophenyl)-*N*′-phenylurea] (0.01 mol, 2.27 g) in absolute ethanol (50 mL) stirred at room temperature was added benzaldehyde (0.01 mol, 1.1 g). The mixture was stirred at reflux conditions, until complete, and then cooled to room temperature. The precipitate was filtered off and washed with cold ethanol. The resulting residue was purified by crystallization from EtOH to afford the title compound (2.1 g, 67% yield) as a light-orange solid (m.p. 473–475 K).

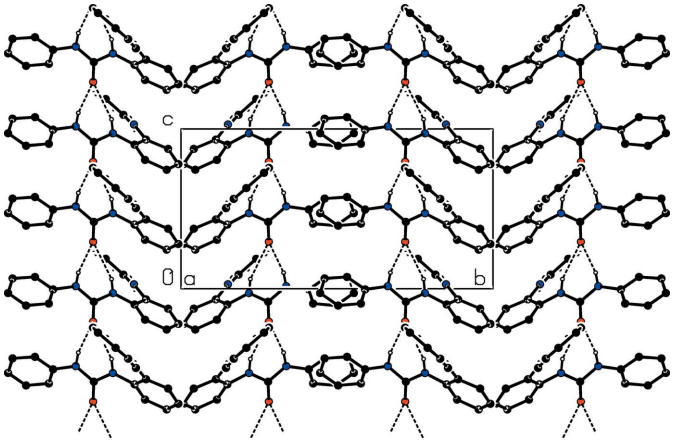


Figure 2
A view of the N—H···O hydrogen bonding (dashed lines) along the *a* axis in the crystal of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.08	2.921 (3)	166
N2—H2···O1^{i}	0.86	2.36	3.144 (3)	152

Symmetry code: (i) $-x+\frac{1}{2}, y, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{17}N_3O$
M_r	315.36
Crystal system, space group	Orthorhombic, $Pca2_1$
Temperature (K)	297
a, *b*, *c* (Å)	10.3381 (6), 17.6843 (13), 9.0562 (5)
V (Å^3)	1655.67 (18)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.08
Crystal size (mm)	0.42 × 0.23 × 0.20
Data collection	
Diffractometer	Rigaku Oxford Diffraction Xcalibur, Eos
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.980, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5335, 2499, 1574
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.066, 0.85
No. of reflections	2499
No. of parameters	217
No. of restraints	29
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.12, −0.12

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal investigated was refined as a two-component twin by non-merohedry. The twin ratio refined to a value of 0.966 (3):0.034 (3). The N-bound ring displayed high displacement parameters and was refined with several constraints.

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full crystallographic data

IUCrData (2017). **2**, x171519 [https://doi.org/10.1107/S2414314617015190X]

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N-[2-(Benzylideneamino)phenyl]-*N*′-phenylurea

Crystal data

$C_{20}H_{17}N_3O$
M_r = 315.36
Orthorhombic, $Pca2_1$
a = 10.3381 (6) Å
b = 17.6843 (13) Å
c = 9.0562 (5) Å
V = 1655.67 (18) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.265 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1544 reflections
θ = 4.5–25.2°
μ = 0.08 mm^{-1}
T = 297 K
Prism, light-orange
0.42 × 0.23 × 0.20 mm

Data collection

Rigaku Oxford Diffraction Xcalibur, Eos diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.0667 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.980, T_{max} = 0.988
5335 measured reflections
2499 independent reflections
1574 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 26.4°, θ_{min} = 3.0°
h = −12→12
k = −22→16
l = −8→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.066
S = 0.85
2499 reflections
217 parameters
29 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Special details

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

Refinement. The H atoms attached to C and N atoms were placed in calculated positions (C—H = 0.93 Å and N—H = 0.86 Å) and treated as riding with U_{iso}(H) = 1.2 or 1.5U_{eq}(N,C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.17464 (16)	0.71846 (10)	0.29408 (19)	0.0609 (5)
N1	0.21897 (19)	0.66269 (12)	0.5158 (2)	0.0554 (6)
H1	0.2541	0.6711	0.6004	0.066*
N2	0.28671 (18)	0.78190 (12)	0.4728 (2)	0.0524 (6)
H2	0.3177	0.7775	0.5605	0.063*
N3	0.51212 (19)	0.84938 (10)	0.5281 (2)	0.0500 (5)
C1	0.1652 (3)	0.59015 (18)	0.4960 (3)	0.0578 (7)
C2	0.2172 (3)	0.5321 (2)	0.5740 (4)	0.0964 (12)
H2A	0.2877	0.5412	0.6352	0.116*
C3	0.1681 (4)	0.4604 (2)	0.5645 (6)	0.1319 (17)
H3	0.2041	0.4216	0.6204	0.158*
C4	0.0664 (5)	0.4460 (3)	0.4733 (5)	0.136 (2)
H4	0.0337	0.3973	0.4646	0.163*
C5	0.0134 (5)	0.5036 (3)	0.3950 (5)	0.151 (2)
H5	−0.0563	0.4943	0.3327	0.181*
C6	0.0625 (4)	0.5765 (2)	0.4074 (4)	0.1091 (13)
H6	0.0248	0.6159	0.3546	0.131*
C7	0.2223 (2)	0.72075 (16)	0.4189 (3)	0.0472 (6)
C8	0.3085 (2)	0.85135 (15)	0.4020 (3)	0.0473 (6)
C9	0.2214 (3)	0.88489 (17)	0.3065 (3)	0.0578 (7)
H9	0.1442	0.8605	0.2837	0.069*
C10	0.2486 (4)	0.95423 (17)	0.2450 (3)	0.0657 (8)
H10	0.1906	0.9759	0.1789	0.079*
C11	0.3603 (3)	0.99154 (17)	0.2803 (3)	0.0683 (8)
H11	0.3770	1.0389	0.2401	0.082*
C12	0.4482 (3)	0.95884 (16)	0.3756 (3)	0.0575 (7)
H12	0.5234	0.9847	0.4007	0.069*
C13	0.4250 (2)	0.88802 (15)	0.4337 (3)	0.0479 (7)
C14	0.6317 (3)	0.85566 (15)	0.5046 (3)	0.0545 (7)
H14	0.6585	0.8878	0.4293	0.065*
C15	0.7306 (3)	0.81545 (15)	0.5886 (3)	0.0539 (7)
C16	0.8592 (3)	0.83093 (18)	0.5624 (3)	0.0684 (8)
H16	0.8811	0.8696	0.4974	0.082*
C17	0.9554 (3)	0.7904 (2)	0.6301 (4)	0.0802 (10)
H17	1.0418	0.8013	0.6114	0.096*
C18	0.9223 (4)	0.7340 (2)	0.7255 (4)	0.0858 (11)
H18	0.9870	0.7056	0.7705	0.103*
C19	0.7937 (4)	0.71814 (17)	0.7563 (4)	0.0839 (10)
H19	0.7726	0.6802	0.8232	0.101*
C20	0.6977 (3)	0.75878 (17)	0.6877 (3)	0.0621 (8)
H20	0.6112	0.7484	0.7075	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0729 (12)	0.0698 (13)	0.0400 (10)	−0.0055 (10)	−0.0054 (9)	−0.0038 (10)
N1	0.0683 (15)	0.0557 (13)	0.0421 (11)	−0.0115 (13)	−0.0086 (11)	0.0021 (13)
N2	0.0584 (13)	0.0574 (14)	0.0413 (12)	−0.0126 (12)	−0.0069 (11)	0.0018 (12)
N3	0.0507 (12)	0.0504 (14)	0.0488 (12)	−0.0037 (12)	−0.0036 (11)	−0.0010 (13)
C1	0.0637 (17)	0.062 (2)	0.0476 (16)	−0.0081 (17)	0.0150 (16)	−0.0039 (17)
C2	0.093 (3)	0.064 (2)	0.132 (3)	−0.007 (2)	−0.003 (2)	0.013 (2)
C3	0.146 (4)	0.058 (3)	0.191 (5)	−0.008 (3)	0.027 (3)	0.012 (3)
C4	0.193 (5)	0.089 (3)	0.124 (4)	−0.064 (4)	0.073 (4)	−0.043 (3)
C5	0.187 (5)	0.163 (5)	0.103 (3)	−0.126 (5)	−0.007 (3)	−0.001 (4)
C6	0.119 (3)	0.118 (3)	0.090 (2)	−0.059 (3)	−0.026 (2)	0.024 (2)
C7	0.0437 (14)	0.0593 (19)	0.0386 (15)	0.0012 (14)	0.0055 (13)	−0.0026 (15)
C8	0.0549 (17)	0.0470 (16)	0.0401 (14)	0.0026 (14)	0.0039 (13)	−0.0019 (14)
C9	0.0563 (17)	0.0654 (19)	0.0516 (18)	0.0116 (16)	−0.0051 (14)	−0.0084 (16)
C10	0.081 (2)	0.064 (2)	0.0522 (18)	0.021 (2)	−0.0107 (15)	0.0018 (18)
C11	0.091 (2)	0.0533 (18)	0.0605 (17)	0.0100 (19)	0.001 (2)	0.0124 (17)
C12	0.0656 (19)	0.0483 (17)	0.0587 (18)	−0.0056 (15)	0.0060 (15)	0.0033 (16)
C13	0.0531 (15)	0.0501 (17)	0.0405 (15)	0.0014 (14)	0.0028 (13)	0.0000 (14)
C14	0.0575 (17)	0.0549 (17)	0.0512 (15)	−0.0035 (14)	0.0005 (15)	−0.0004 (15)
C15	0.0533 (17)	0.0558 (18)	0.0526 (16)	−0.0012 (15)	−0.0053 (15)	−0.0110 (15)
C16	0.0549 (17)	0.084 (2)	0.0657 (18)	0.0032 (18)	−0.0014 (16)	−0.0070 (18)
C17	0.061 (2)	0.104 (3)	0.075 (2)	0.012 (2)	−0.0023 (19)	−0.023 (2)
C18	0.079 (3)	0.094 (3)	0.085 (3)	0.026 (2)	−0.030 (2)	−0.017 (2)
C19	0.102 (3)	0.065 (2)	0.084 (2)	0.006 (2)	−0.025 (2)	0.004 (2)
C20	0.0677 (19)	0.0566 (19)	0.0622 (17)	−0.0030 (17)	−0.0063 (16)	−0.0011 (16)

Geometric parameters (Å, º)

O1—C7	1.234 (3)	C9—C10	1.376 (3)
N1—C7	1.351 (3)	C9—H9	0.9300
N1—C1	1.409 (3)	C10—C11	1.368 (4)
N1—H1	0.8600	C10—H10	0.9300
N2—C7	1.361 (3)	C11—C12	1.381 (3)
N2—C8	1.404 (3)	C11—H11	0.9300
N2—H2	0.8600	C12—C13	1.380 (3)
N3—C14	1.259 (3)	C12—H12	0.9300
N3—C13	1.417 (3)	C14—C15	1.460 (4)
C1—C6	1.353 (4)	C14—H14	0.9300
C1—C2	1.357 (4)	C15—C16	1.377 (4)
C2—C3	1.368 (5)	C15—C20	1.388 (4)
C2—H2A	0.9300	C16—C17	1.371 (4)
C3—C4	1.361 (4)	C16—H16	0.9300
C3—H3	0.9300	C17—C18	1.364 (4)
C4—C5	1.356 (6)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.386 (4)
C5—C6	1.389 (5)	C18—H18	0.9300

C5—H5	0.9300	C19—C20	1.375 (4)
C6—H6	0.9300	C19—H19	0.9300
C8—C9	1.382 (3)	C20—H20	0.9300
C8—C13	1.398 (3)		

C7—N1—C1	128.2 (2)	C11—C10—C9	120.5 (3)
C7—N1—H1	115.9	C11—C10—H10	119.7
C1—N1—H1	115.9	C9—C10—H10	119.7
C7—N2—C8	127.6 (2)	C10—C11—C12	120.0 (3)
C7—N2—H2	116.2	C10—C11—H11	120.0
C8—N2—H2	116.2	C12—C11—H11	120.0
C14—N3—C13	118.6 (2)	C13—C12—C11	120.3 (3)
C6—C1—C2	119.0 (3)	C13—C12—H12	119.9
C6—C1—N1	123.2 (3)	C11—C12—H12	119.9
C2—C1—N1	117.8 (3)	C12—C13—C8	119.5 (2)
C1—C2—C3	121.4 (4)	C12—C13—N3	123.9 (2)
C1—C2—H2A	119.3	C8—C13—N3	116.6 (2)
C3—C2—H2A	119.3	N3—C14—C15	123.8 (3)
C4—C3—C2	119.9 (4)	N3—C14—H14	118.1
C4—C3—H3	120.1	C15—C14—H14	118.1
C2—C3—H3	120.1	C16—C15—C20	119.5 (3)
C5—C4—C3	119.3 (5)	C16—C15—C14	119.3 (3)
C5—C4—H4	120.4	C20—C15—C14	121.1 (2)
C3—C4—H4	120.4	C17—C16—C15	121.3 (3)
C4—C5—C6	120.4 (5)	C17—C16—H16	119.4
C4—C5—H5	119.8	C15—C16—H16	119.4
C6—C5—H5	119.8	C18—C17—C16	118.9 (3)
C1—C6—C5	120.0 (4)	C18—C17—H17	120.5
C1—C6—H6	120.0	C16—C17—H17	120.5
C5—C6—H6	120.0	C17—C18—C19	121.0 (3)
O1—C7—N1	124.1 (3)	C17—C18—H18	119.5
O1—C7—N2	123.4 (3)	C19—C18—H18	119.5
N1—C7—N2	112.5 (2)	C20—C19—C18	119.7 (3)
C9—C8—C13	119.4 (2)	C20—C19—H19	120.1
C9—C8—N2	123.8 (2)	C18—C19—H19	120.1
C13—C8—N2	116.7 (2)	C19—C20—C15	119.5 (3)
C10—C9—C8	120.2 (3)	C19—C20—H20	120.2
C10—C9—H9	119.9	C15—C20—H20	120.2
C8—C9—H9	119.9		

C7—N1—C1—C6	−30.1 (5)	C10—C11—C12—C13	1.0 (4)
C7—N1—C1—C2	152.2 (3)	C11—C12—C13—C8	−3.5 (3)
C6—C1—C2—C3	0.1 (5)	C11—C12—C13—N3	177.8 (2)
N1—C1—C2—C3	177.9 (3)	C9—C8—C13—C12	3.5 (3)
C1—C2—C3—C4	1.2 (7)	N2—C8—C13—C12	−175.6 (2)
C2—C3—C4—C5	−1.4 (7)	C9—C8—C13—N3	−177.7 (2)
C3—C4—C5—C6	0.3 (8)	N2—C8—C13—N3	3.2 (3)
C2—C1—C6—C5	−1.2 (6)	C14—N3—C13—C12	−37.0 (3)

N1—C1—C6—C5	−178.9 (3)	C14—N3—C13—C8	144.3 (2)
C4—C5—C6—C1	1.0 (7)	C13—N3—C14—C15	−176.5 (2)
C1—N1—C7—O1	1.1 (4)	N3—C14—C15—C16	−174.6 (3)
C1—N1—C7—N2	−177.5 (2)	N3—C14—C15—C20	9.3 (4)
C8—N2—C7—O1	1.3 (4)	C20—C15—C16—C17	1.3 (4)
C8—N2—C7—N1	179.9 (2)	C14—C15—C16—C17	−174.8 (3)
C7—N2—C8—C9	34.6 (3)	C15—C16—C17—C18	−0.2 (4)
C7—N2—C8—C13	−146.4 (2)	C16—C17—C18—C19	−1.2 (5)
C13—C8—C9—C10	−0.9 (3)	C17—C18—C19—C20	1.4 (5)
N2—C8—C9—C10	178.1 (2)	C18—C19—C20—C15	−0.3 (4)
C8—C9—C10—C11	−1.6 (4)	C16—C15—C20—C19	−1.1 (4)
C9—C10—C11—C12	1.6 (4)	C14—C15—C20—C19	175.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.08	2.921 (3)	166
N2—H2···O1^{i}	0.86	2.36	3.144 (3)	152

Symmetry code: (i) −*x*+1/2, *y*, *z*+1/2.