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(*E*)-2-{[(4-Anilinophenyl)imino]methyl}-4-bromo-5-fluorophenol

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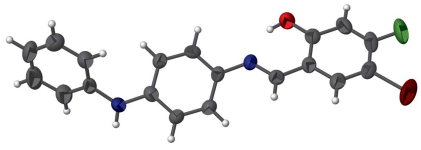
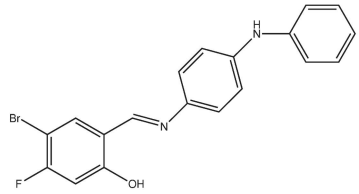
In the title compound, $C_{19}H_{14}BrFN_2O$, the dihedral angles between the central benzene ring and the pendant trisubstituted ring and phenyl group are 25.7 (2) and 51.7 (2)°, respectively. The molecular conformation is influenced by an intramolecular O—H···N hydrogen bond. In the crystal, N—H···O hydrogen bonds link molecules into *C*(11) chains propagating in [100] and weak aromatic π–π stacking is also observed [centroid–centroid separation = 3.682 (3) Å]

3D view



Chemical scheme



Structure description

Schiff bases (Schiff, 1864) contain the azomethine grouping (–*R*C═N–) and are prepared by condensation reactions between amines and active carbonyl compounds. As part of our studies in this area, we herein report the synthesis and structure of the title compound (Fig. 1).

The dihedral angles between the central benzene ring (C8–C13) and pendant trisubstituted ring (C1–C6) and phenyl ring (C14–C19) are 25.7 (2) and 51.7 (2)°, respectively; the dihedral angle between the outer rings is 75.9 (2)°. The molecular conformation is influenced by an intramolecular O—H···N hydrogen bond (Table 1, Fig. 1), which generates an *S*(6) ring. The bond lengths for imino group atoms [N2—C8 = 1.403 (5) and N2—C7 = 1.292 (5) Å] are consistent with those in related structures such as 2-amino-3-((*E*)-{[3-(trifluoromethyl)phenyl]imino}methyl)-4*H*-chromen-4-one (Atalay *et al.*, 2016) and (*Z*)-4-{[(*Z*)-(2-oxonaphthalen-1(2*H*)- ylidene)methyl]amino}-*N*-(thiazol-2(3*H*)-ylidene)benzenesulfonamide (Köysal *et al.*, 2015).

In the extended structure (Fig. 2), N—H···O hydrogen bonds link the molecules into *C*(11) chains propagating in [100]; thus O1 serves as an acceptor for both intra- and

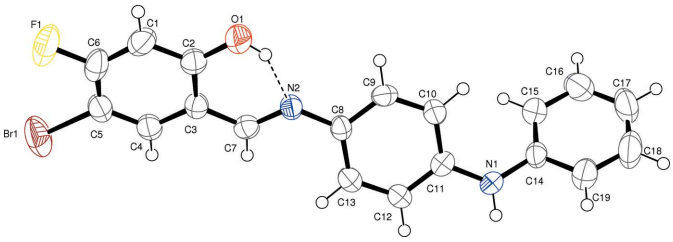


Figure 1
A view of the title compound with 50% probability displacement ellipsoids. The intramolecular O—H···N hydrogen bond is indicated by a dashed line.

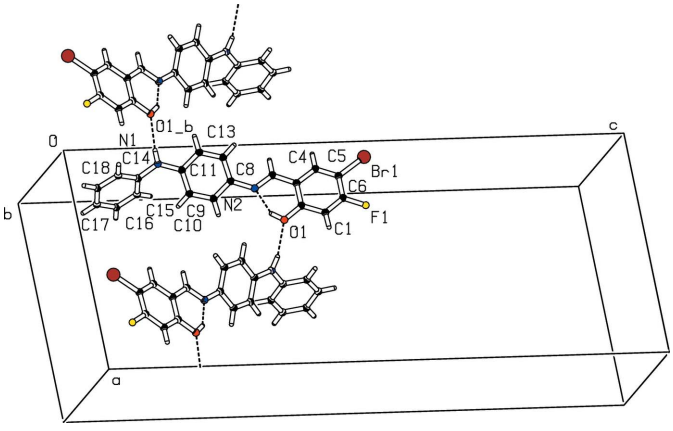


Figure 2
A partial packing view. Dashed lines indicate the hydrogen bonds.

intermolecular hydrogen bonds. Weak aromatic π–π stacking between the C1–C6 rings is also observed [centroid–centroid separation = 3.682 (3) Å]

Synthesis and crystallization

The title compound was prepared by refluxing for 18 h a mixture of 5-bromo-3-fluoro-2-hydroxybenzaldehyde (0.01 g, 0.045 mmol) in 20 ml ethyl alcohol and *N*-phenylbenzene-1,4-diamine (0.08 g, 0.045 mmol) in 20 ml ethyl alcohol. Red prismatic crystals were obtained from the solution by slow evaporation (yield 73%; m.p. 168–170°C).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H01···N2	0.82	1.80	2.535 (4)	149
N1—H02···O1^{i}	0.84 (4)	2.11 (4)	2.927 (5)	166 (4)

Symmetry code: (i) $x-\frac{1}{2}, y, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{14}BrFN_2O$
M_r	385.23
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	293
a, b, c (Å)	13.4521 (15), 7.1810 (7), 34.204 (3)
V (Å^3)	3304.1 (6)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.51
Crystal size (mm)	0.40 × 0.25 × 0.07
Data collection	
Diffractometer	Agilent Xcalibur Eos
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku, 2015)
T_{min}, T_{max}	0.495, 0.896
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6640, 3349, 1552
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.110, 0.97
No. of reflections	3349
No. of parameters	221
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.51

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

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full crystallographic data

IUCrData (2017). **2**, x171708 [https://doi.org/10.1107/S2414314617017084]

(*E*)-2-{[(4-Anilinophenyl)imino]methyl}-4-bromo-5-fluorophenol

Sedanur Kalecik, Metin Yavuz, Fatih Kavraz, Hande Eserci and Erbil Ağar

(I)

Crystal data

$C_{19}H_{14}BrFN_2O$
M_r = 385.23
Orthorhombic, *Pbca*
a = 13.4521 (15) Å
b = 7.1810 (7) Å
c = 34.204 (3) Å
V = 3304.1 (6) Å^3
Z = 8
F(000) = 1552
D_x = 1.549 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 1184 reflections
θ = 3.8–20.9°
μ = 2.51 mm^{-1}
T = 293 K
Prism, red
0.40 × 0.25 × 0.07 mm

Data collection

Agilent Xcalibur Eos diffractometer
Radiation source: fine-focus sealed X-ray tube
w scans
Absorption correction: analytical (CrysAlis PRO; Rigaku, 2015)
T_{min} = 0.495, T_{max} = 0.896
6640 measured reflections
3349 independent reflections
1552 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 26.4°, θ_{min} = 3.0°
h = −16→9
k = −4→8
l = −36→42
6678 standard reflections every ··· reflections
intensity decay: ···

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.110
S = 0.97
3349 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0267P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 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 N—H and H atoms were located in a difference Fourier map. Their positional and isotropic thermal parameters were included in further stages of the refinement. All C-bound H atoms were positioned geometricaly and refined using a riding model with C—H = 0.93 - 0.97 Å and with U_{iso}(H) = 1.2 - 1.5 U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.65808 (5)	0.86784 (8)	0.50622 (2)	0.0847 (3)
F1	0.8795 (2)	0.8762 (4)	0.52160 (8)	0.0843 (9)
O1	0.88602 (19)	0.6698 (4)	0.65086 (9)	0.0586 (9)
H01	0.848013	0.636740	0.668277	0.088*
N2	0.7227 (2)	0.6000 (4)	0.68480 (10)	0.0403 (9)
N1	0.5591 (3)	0.4382 (6)	0.83117 (12)	0.0542 (12)
C8	0.6757 (3)	0.5548 (5)	0.72019 (12)	0.0366 (10)
C3	0.7295 (3)	0.7032 (6)	0.61908 (13)	0.0398 (11)
C7	0.6765 (3)	0.6471 (5)	0.65320 (13)	0.0414 (11)
H7	0.607388	0.644255	0.652690	0.050*
C11	0.5987 (3)	0.4717 (6)	0.79440 (13)	0.0411 (11)
C13	0.5808 (3)	0.6124 (6)	0.73096 (13)	0.0460 (12)
H13	0.541659	0.677957	0.713265	0.055*
C9	0.7308 (3)	0.4599 (5)	0.74768 (13)	0.0410 (11)
H9	0.795425	0.424950	0.741439	0.049*
C4	0.6786 (3)	0.7504 (6)	0.58495 (13)	0.0470 (12)
H4	0.609510	0.744233	0.584742	0.056*
C5	0.7267 (4)	0.8051 (6)	0.55214 (13)	0.0499 (12)
C10	0.6941 (3)	0.4148 (5)	0.78392 (13)	0.0429 (11)
H10	0.732748	0.346709	0.801366	0.052*
C12	0.5448 (3)	0.5731 (6)	0.76728 (13)	0.0472 (12)
H12	0.481906	0.615940	0.774085	0.057*
C2	0.8344 (3)	0.7128 (6)	0.61936 (13)	0.0432 (11)
C14	0.5920 (3)	0.3098 (7)	0.85908 (13)	0.0452 (12)
C6	0.8301 (5)	0.8171 (6)	0.55333 (15)	0.0577 (14)
C15	0.6228 (3)	0.1338 (7)	0.84917 (15)	0.0565 (13)
H15	0.625653	0.098961	0.823011	0.068*
C1	0.8829 (3)	0.7725 (6)	0.58571 (16)	0.0555 (14)
H1	0.951815	0.781849	0.585443	0.067*
C19	0.5873 (3)	0.3579 (8)	0.89792 (14)	0.0636 (14)
H19	0.566809	0.477105	0.904875	0.076*
C16	0.6498 (3)	0.0081 (8)	0.87793 (18)	0.0733 (16)
H16	0.671249	−0.110613	0.871086	0.088*
C17	0.6449 (4)	0.0586 (9)	0.91645 (18)	0.0784 (18)
H17	0.663745	−0.025643	0.935727	0.094*
C18	0.6125 (4)	0.2322 (10)	0.92675 (16)	0.0824 (18)
H18	0.607576	0.265250	0.952963	0.099*
H02	0.516 (3)	0.515 (5)	0.8390 (13)	0.049 (16)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1416 (6)	0.0693 (4)	0.0432 (3)	0.0091 (4)	−0.0233 (3)	0.0049 (3)
F1	0.127 (2)	0.078 (2)	0.0485 (19)	−0.0068 (18)	0.0331 (17)	0.0069 (18)
O1	0.0495 (19)	0.083 (2)	0.044 (2)	0.0085 (18)	−0.0003 (15)	0.007 (2)

N2	0.052 (2)	0.036 (2)	0.033 (2)	0.0052 (19)	0.0005 (18)	0.0016 (19)
N1	0.058 (3)	0.068 (3)	0.037 (2)	0.024 (3)	0.011 (2)	0.004 (2)
C8	0.047 (3)	0.031 (2)	0.032 (3)	−0.001 (2)	−0.002 (2)	0.001 (2)
C3	0.048 (3)	0.035 (3)	0.037 (3)	−0.002 (2)	0.001 (2)	0.000 (2)
C7	0.042 (3)	0.038 (3)	0.044 (3)	0.001 (2)	0.002 (2)	−0.002 (2)
C11	0.043 (3)	0.043 (3)	0.038 (3)	−0.002 (2)	0.004 (2)	−0.003 (2)
C13	0.045 (3)	0.049 (3)	0.044 (3)	0.007 (3)	−0.003 (2)	0.007 (3)
C9	0.037 (3)	0.041 (3)	0.045 (3)	0.007 (2)	0.002 (2)	−0.006 (2)
C4	0.062 (3)	0.038 (3)	0.041 (3)	0.008 (3)	−0.001 (2)	−0.003 (2)
C5	0.076 (4)	0.038 (3)	0.036 (3)	0.011 (3)	−0.005 (3)	−0.003 (2)
C10	0.047 (3)	0.045 (3)	0.037 (3)	0.004 (2)	−0.003 (2)	0.003 (2)
C12	0.048 (3)	0.049 (3)	0.045 (3)	0.012 (2)	0.002 (2)	0.009 (3)
C2	0.051 (3)	0.037 (3)	0.042 (3)	0.003 (3)	0.002 (2)	−0.004 (2)
C14	0.036 (3)	0.067 (4)	0.032 (3)	0.000 (3)	−0.002 (2)	0.005 (3)
C6	0.103 (5)	0.032 (3)	0.038 (3)	0.005 (3)	0.021 (3)	−0.002 (2)
C15	0.057 (3)	0.067 (4)	0.045 (3)	0.009 (3)	0.005 (2)	0.007 (3)
C1	0.061 (3)	0.048 (3)	0.057 (4)	0.003 (3)	0.022 (3)	0.004 (3)
C19	0.069 (3)	0.079 (4)	0.043 (3)	0.004 (3)	0.000 (3)	−0.002 (3)
C16	0.066 (3)	0.081 (4)	0.073 (4)	0.018 (3)	0.005 (3)	0.020 (4)
C17	0.081 (4)	0.103 (5)	0.051 (4)	0.009 (4)	−0.004 (3)	0.031 (4)
C18	0.085 (4)	0.125 (6)	0.037 (4)	0.000 (4)	−0.006 (3)	0.004 (4)

Geometric parameters (Å, º)

Br1—C5	1.877 (5)	C9—H9	0.9300
F1—C6	1.341 (5)	C4—C5	1.354 (6)
O1—C2	1.318 (5)	C4—H4	0.9300
O1—H01	0.8200	C5—C6	1.394 (6)
N2—C7	1.292 (5)	C10—H10	0.9300
N2—C8	1.403 (5)	C12—H12	0.9300
N1—C11	1.387 (5)	C2—C1	1.390 (6)
N1—C14	1.399 (5)	C14—C15	1.373 (6)
N1—H02	0.84 (4)	C14—C19	1.374 (6)
C8—C9	1.378 (5)	C6—C1	1.354 (6)
C8—C13	1.392 (5)	C15—C16	1.384 (6)
C3—C4	1.396 (5)	C15—H15	0.9300
C3—C2	1.413 (5)	C1—H1	0.9300
C3—C7	1.426 (5)	C19—C18	1.380 (7)
C7—H7	0.9300	C19—H19	0.9300
C11—C12	1.385 (5)	C16—C17	1.368 (7)
C11—C10	1.394 (5)	C16—H16	0.9300
C13—C12	1.363 (5)	C17—C18	1.367 (7)
C13—H13	0.9300	C17—H17	0.9300
C9—C10	1.372 (5)	C18—H18	0.9300
C2—O1—H01	109.5	C11—C10—H10	120.2
C7—N2—C8	124.4 (4)	C13—C12—C11	122.2 (4)
C11—N1—C14	127.7 (4)	C13—C12—H12	118.9

C11—N1—H02	116 (3)	C11—C12—H12	118.9
C14—N1—H02	116 (3)	O1—C2—C1	120.1 (4)
C9—C8—C13	117.4 (4)	O1—C2—C3	121.3 (4)
C9—C8—N2	117.5 (4)	C1—C2—C3	118.5 (4)
C13—C8—N2	124.9 (4)	C15—C14—C19	118.9 (5)
C4—C3—C2	119.0 (4)	C15—C14—N1	122.3 (4)
C4—C3—C7	120.5 (4)	C19—C14—N1	118.7 (5)
C2—C3—C7	120.5 (4)	F1—C6—C1	118.5 (5)
N2—C7—C3	121.2 (4)	F1—C6—C5	119.4 (5)
N2—C7—H7	119.4	C1—C6—C5	122.2 (5)
C3—C7—H7	119.4	C14—C15—C16	120.3 (5)
C12—C11—N1	119.8 (4)	C14—C15—H15	119.8
C12—C11—C10	117.7 (4)	C16—C15—H15	119.8
N1—C11—C10	122.4 (4)	C6—C1—C2	120.3 (5)
C12—C13—C8	120.4 (4)	C6—C1—H1	119.9
C12—C13—H13	119.8	C2—C1—H1	119.9
C8—C13—H13	119.8	C14—C19—C18	121.0 (5)
C10—C9—C8	122.7 (4)	C14—C19—H19	119.5
C10—C9—H9	118.7	C18—C19—H19	119.5
C8—C9—H9	118.7	C17—C16—C15	119.9 (6)
C5—C4—C3	121.9 (4)	C17—C16—H16	120.0
C5—C4—H4	119.0	C15—C16—H16	120.0
C3—C4—H4	119.0	C18—C17—C16	120.4 (6)
C4—C5—C6	118.1 (5)	C18—C17—H17	119.8
C4—C5—Br1	121.9 (4)	C16—C17—H17	119.8
C6—C5—Br1	120.0 (4)	C17—C18—C19	119.4 (6)
C9—C10—C11	119.6 (4)	C17—C18—H18	120.3
C9—C10—H10	120.2	C19—C18—H18	120.3

C7—N2—C8—C9	162.7 (4)	C7—C3—C2—O1	0.3 (6)
C7—N2—C8—C13	−23.2 (7)	C4—C3—C2—C1	1.2 (6)
C8—N2—C7—C3	175.8 (4)	C7—C3—C2—C1	−178.5 (4)
C4—C3—C7—N2	178.9 (4)	C11—N1—C14—C15	−40.7 (7)
C2—C3—C7—N2	−1.5 (6)	C11—N1—C14—C19	143.4 (5)
C14—N1—C11—C12	165.8 (4)	C4—C5—C6—F1	−178.0 (4)
C14—N1—C11—C10	−17.5 (7)	Br1—C5—C6—F1	0.9 (6)
C9—C8—C13—C12	−0.7 (6)	C4—C5—C6—C1	1.4 (7)
N2—C8—C13—C12	−174.8 (4)	Br1—C5—C6—C1	−179.7 (4)
C13—C8—C9—C10	2.7 (6)	C19—C14—C15—C16	−0.7 (7)
N2—C8—C9—C10	177.3 (4)	N1—C14—C15—C16	−176.6 (4)
C2—C3—C4—C5	0.0 (7)	F1—C6—C1—C2	179.3 (4)
C7—C3—C4—C5	179.7 (4)	C5—C6—C1—C2	−0.2 (7)
C3—C4—C5—C6	−1.3 (7)	O1—C2—C1—C6	−179.9 (4)
C3—C4—C5—Br1	179.8 (3)	C3—C2—C1—C6	−1.1 (7)
C8—C9—C10—C11	−2.3 (6)	C15—C14—C19—C18	−0.4 (7)
C12—C11—C10—C9	−0.2 (6)	N1—C14—C19—C18	175.7 (4)
N1—C11—C10—C9	−176.9 (4)	C14—C15—C16—C17	0.6 (7)
C8—C13—C12—C11	−1.8 (7)	C15—C16—C17—C18	0.7 (8)

N1—C11—C12—C13	179.0 (4)	C16—C17—C18—C19	−1.7 (8)
C10—C11—C12—C13	2.2 (7)	C14—C19—C18—C17	1.6 (8)
C4—C3—C2—O1	179.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H01···N2	0.82	1.80	2.535 (4)	149
N1—H02···O1^{i}	0.84 (4)	2.11 (4)	2.927 (5)	166 (4)

Symmetry code: (i) $x-1/2, y, -z+3/2$.