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5-[(*E*)-(2-Fluorobenzylidene)amino]-2-hydroxybenzoic acid

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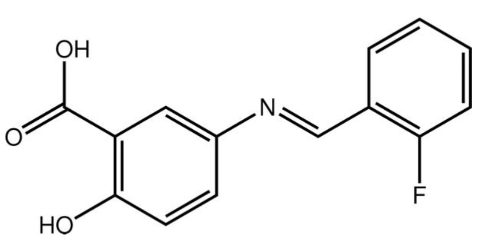
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.127; data-to-parameter ratio = 12.1.

In the title compound, $C_{14}H_{10}FNO_3$, the dihedral angle between the two benzene rings is 32.66 (14)°. An S(6) ring motif is formed due to an intramolecular O—H···O hydrogen bond between the hydroxy and carbonyl groups. In the crystal, molecules are consolidated into dimers with $R_2^2(8)$ ring motifs by pairs of O—H···O hydrogen bonds.

Related literature

For background and related crystal structures, see: Tahir & Shad (2010); Tahir *et al.* (2010*a*,*b*,*c*). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{10}FNO_3$
M_r = 259.23
Monoclinic, $P2_1/n$
a = 15.5688 (16) Å
b = 4.7139 (4) Å
c = 16.2248 (16) Å
β = 92.412 (4)°
V = 1189.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.30 × 0.22 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.972, T_{max} = 0.983
15880 measured reflections
2161 independent reflections
1156 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.127
S = 1.01
2161 reflections
179 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.88 (3)	1.77 (3)	2.642 (3)	176 (3)
O3—H3···O2	0.92 (3)	1.78 (3)	2.617 (3)	152 (3)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2774).

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supplementary materials

Acta Cryst. (2011). E**67**, o2372 [doi:10.1107/S160053681103162X]

5-[(*E*)-(2-Fluorobenzylidene)amino]-2-hydroxybenzoic acid

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Comment

Recently, we reported crystal structures containing the 5-amino-2-hydroxybenzoic acid moiety *i.e.* (II) 2-hydroxy-5-[(*E*)-(1*H*-indol-3-ylmethylidene)azaniumyl]benzoate (Tahir & Shad, 2010), (III) *i.e.* 2-{[(*E*)-1,3-benzodioxol-5-yl]methylideneamino}benzoic acid (Tahir *et al.*, 2010*b*), (IV) *i.e.* 5-[(*E*)-(2,6-dichlorobenzylidene)amino]-2-hydroxybenzoic acid (Tahir *et al.*, 2010*a*) and (V) *i.e.* 2-hydroxy-5-{[(*E*)-4-methoxybenzylidene]azaniumyl}benzoate (Tahir *et al.*, 2010*c*). The title compound (I), (Fig. 1) was prepared in continuation of the synthesis of various molecules having 5-amino-2-hydroxybenzoic acid.

In (I), group A (C1—C7/N1/O1—O3), derived from 5-amino-2-hydroxybenzoic acid, and group B (C8—C14/F1), derived from 2-fluorobenzaldehyde, are each planar with r.m.s. deviations of 0.0164 and 0.0182 Å, respectively. The A/B dihedral angle between is 32.78 (7)°. There exists an intramolecular O—H···O hydrogen bond which completes a S(6) ring motif (Table 1, Fig. 1). In the crystal packing the molecules are stabilized in the form of dimers due to intermolecular O—H···O hydrogen bonds (Table 1, Fig. 2) which form a $R_2^2(8)$ ring motif (Bernstein *et al.*, 1995).

Experimental

Equimolar quantities of 5-amino-2-hydroxybenzoic acid and 2-fluorobenzaldehyde were refluxed in methanol for 45 min resulting in yellow-brown solution. The solution was kept at room temperature which afforded violet prisms after 48 h.

Refinement

The coordinates of the hydroxyl-H atoms were refined freely, and with $U_{iso}(H) = 1.2U_{eq}(O)$. The C-bound H-atoms were positioned geometrically (C–H = 0.93 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

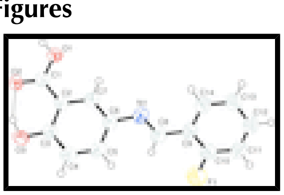

Fig. 1. View of the title compound with the atom numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii. The dotted line represents the intramolecular hydrogen bond.

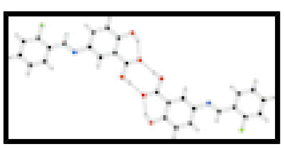

Fig. 2. The partial packing showing that molecules form dimeric aggregates via O—H···O hydrogen bonds (dotted lines).

5-[(*E*)-(2-Fluorobenzylidene)amino]-2-hydroxybenzoic acid

Crystal data

$C_{14}H_{10}FNO_3$	$F(000) = 536$
$M_r = 259.23$	$D_x = 1.447$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1156 reflections
$a = 15.5688$ (16) Å	$\theta = 2.5$–$25.3°$
$b = 4.7139$ (4) Å	$\mu = 0.11$ mm^{-1}
$c = 16.2248$ (16) Å	$T = 296$ K
$\beta = 92.412$ (4)°	Prism, violet
$V = 1189.7$ (2) Å^3	0.30 × 0.22 × 0.18 mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD diffractometer	2161 independent reflections
Radiation source: fine-focus sealed tube	1156 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.079$
Detector resolution: 8.10 pixels mm^{-1}	$\theta_{max} = 25.3°$, $\theta_{min} = 2.5°$
ω scans	$h = -18\rightarrow18$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$k = -5\rightarrow5$
$T_{min} = 0.972$, $T_{max} = 0.983$	$l = -19\rightarrow19$
15880 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.127$	$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.0687P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
2161 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
179 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.012 (2)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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}
F1	0.52929 (12)	−0.3124 (5)	0.56845 (13)	0.1012 (9)
O1	0.06399 (12)	0.7307 (4)	0.55258 (12)	0.0525 (8)
O2	0.05067 (11)	0.8698 (4)	0.42115 (11)	0.0495 (7)
O3	0.14217 (13)	0.6175 (4)	0.31108 (12)	0.0542 (8)
N1	0.28499 (15)	−0.0194 (4)	0.57191 (14)	0.0515 (9)
C1	0.08505 (17)	0.7167 (5)	0.47509 (18)	0.0417 (10)
C2	0.15088 (16)	0.5080 (5)	0.45759 (15)	0.0375 (9)
C3	0.17635 (17)	0.4699 (5)	0.37661 (17)	0.0434 (10)
C4	0.23883 (18)	0.2707 (6)	0.36048 (19)	0.0519 (11)
C5	0.27501 (17)	0.1124 (6)	0.42340 (19)	0.0514 (11)
C6	0.25157 (17)	0.1477 (5)	0.50467 (18)	0.0442 (10)
C7	0.18911 (17)	0.3435 (5)	0.52047 (17)	0.0432 (9)
C8	0.3618 (2)	−0.1094 (6)	0.57106 (19)	0.0549 (11)
C9	0.39800 (18)	−0.2991 (5)	0.63403 (18)	0.0487 (11)
C10	0.4808 (2)	−0.4013 (6)	0.6308 (2)	0.0584 (11)
C11	0.5166 (2)	−0.5910 (7)	0.6868 (2)	0.0656 (12)
C12	0.4681 (2)	−0.6790 (7)	0.7506 (2)	0.0651 (12)
C13	0.3861 (2)	−0.5771 (7)	0.7575 (2)	0.0671 (12)
C14	0.35127 (19)	−0.3924 (6)	0.70003 (19)	0.0607 (11)
H1	0.0276 (18)	0.869 (6)	0.5597 (16)	0.0630*
H3	0.1029 (18)	0.734 (6)	0.3343 (18)	0.0651*
H4	0.25617	0.24473	0.30685	0.0624*
H5	0.31634	−0.02248	0.41163	0.0618*
H7	0.17190	0.36688	0.57425	0.0518*
H8	0.39610	−0.05202	0.52858	0.0659*
H11	0.57227	−0.65780	0.68150	0.0788*
H12	0.49076	−0.80744	0.78922	0.0777*
H13	0.35381	−0.63410	0.80153	0.0803*
H14	0.29529	−0.32804	0.70528	0.0729*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

F1	0.0551 (12)	0.1469 (19)	0.1031 (17)	0.0089 (12)	0.0211 (12)	0.0358 (14)
O1	0.0594 (14)	0.0547 (12)	0.0440 (13)	0.0135 (10)	0.0101 (10)	−0.0004 (10)
O2	0.0534 (13)	0.0504 (12)	0.0450 (12)	0.0078 (10)	0.0043 (10)	0.0038 (10)
O3	0.0612 (14)	0.0565 (13)	0.0456 (13)	0.0044 (10)	0.0096 (10)	0.0015 (10)
N1	0.0474 (16)	0.0455 (14)	0.0613 (17)	0.0035 (12)	−0.0011 (13)	0.0003 (12)
C1	0.0439 (18)	0.0368 (15)	0.0443 (18)	−0.0081 (14)	0.0023 (15)	−0.0009 (14)
C2	0.0398 (16)	0.0336 (14)	0.0391 (17)	−0.0055 (12)	0.0034 (13)	−0.0014 (13)
C3	0.0431 (17)	0.0398 (15)	0.0476 (18)	−0.0057 (14)	0.0050 (14)	0.0029 (14)
C4	0.053 (2)	0.0519 (18)	0.0517 (19)	−0.0005 (16)	0.0132 (16)	−0.0083 (16)
C5	0.0437 (18)	0.0448 (17)	0.066 (2)	0.0021 (14)	0.0058 (16)	−0.0085 (16)
C6	0.0419 (17)	0.0376 (15)	0.0529 (19)	−0.0046 (14)	0.0009 (14)	−0.0005 (14)
C7	0.0454 (17)	0.0377 (15)	0.0469 (17)	−0.0032 (13)	0.0075 (14)	−0.0038 (13)
C8	0.049 (2)	0.0498 (17)	0.066 (2)	−0.0056 (15)	0.0025 (16)	0.0062 (16)
C9	0.0419 (18)	0.0461 (17)	0.058 (2)	0.0001 (14)	0.0006 (15)	−0.0005 (15)
C10	0.0444 (19)	0.068 (2)	0.063 (2)	−0.0011 (17)	0.0061 (17)	0.0017 (18)
C11	0.049 (2)	0.078 (2)	0.069 (2)	0.0194 (18)	−0.0075 (18)	−0.0099 (19)
C12	0.065 (2)	0.068 (2)	0.061 (2)	0.0096 (18)	−0.0120 (19)	0.0010 (17)
C13	0.056 (2)	0.085 (2)	0.060 (2)	0.0020 (19)	−0.0019 (17)	0.0117 (19)
C14	0.0460 (19)	0.069 (2)	0.067 (2)	0.0058 (17)	0.0003 (17)	0.0043 (18)

Geometric parameters (Å, °)

F1—C10	1.354 (4)	C8—C9	1.454 (4)
O1—C1	1.314 (3)	C9—C14	1.391 (4)
O2—C1	1.239 (3)	C9—C10	1.379 (4)
O3—C3	1.360 (3)	C10—C11	1.376 (4)
O1—H1	0.88 (3)	C11—C12	1.371 (5)
O3—H3	0.92 (3)	C12—C13	1.373 (4)
N1—C6	1.426 (3)	C13—C14	1.371 (4)
N1—C8	1.270 (4)	C4—H4	0.9300
C1—C2	1.457 (4)	C5—H5	0.9300
C2—C7	1.395 (4)	C7—H7	0.9300
C2—C3	1.400 (4)	C8—H8	0.9300
C3—C4	1.385 (4)	C11—H11	0.9300
C4—C5	1.367 (4)	C12—H12	0.9300
C5—C6	1.393 (4)	C13—H13	0.9300
C6—C7	1.373 (4)	C14—H14	0.9300
C1—O1—H1	110.7 (17)	F1—C10—C9	118.1 (3)
C3—O3—H3	103.4 (18)	F1—C10—C11	118.1 (3)
C6—N1—C8	119.3 (2)	C10—C11—C12	118.3 (3)
O1—C1—O2	121.9 (2)	C11—C12—C13	120.0 (3)
O1—C1—C2	115.2 (2)	C12—C13—C14	120.6 (3)
O2—C1—C2	122.8 (3)	C9—C14—C13	121.3 (3)
C1—C2—C3	119.9 (2)	C3—C4—H4	120.00
C3—C2—C7	119.1 (2)	C5—C4—H4	120.00
C1—C2—C7	121.0 (2)	C4—C5—H5	119.00
O3—C3—C4	117.0 (2)	C6—C5—H5	119.00
O3—C3—C2	123.4 (2)	C2—C7—H7	119.00
C2—C3—C4	119.6 (2)	C6—C7—H7	119.00

C3—C4—C5	120.0 (3)	N1—C8—H8	119.00
C4—C5—C6	121.7 (3)	C9—C8—H8	119.00
C5—C6—C7	118.2 (3)	C10—C11—H11	121.00
N1—C6—C7	117.8 (2)	C12—C11—H11	121.00
N1—C6—C5	123.8 (2)	C11—C12—H12	120.00
C2—C7—C6	121.4 (3)	C13—C12—H12	120.00
N1—C8—C9	122.4 (3)	C12—C13—H13	120.00
C10—C9—C14	116.0 (3)	C14—C13—H13	120.00
C8—C9—C10	121.6 (3)	C9—C14—H14	119.00
C8—C9—C14	122.4 (3)	C13—C14—H14	119.00
C9—C10—C11	123.7 (3)		

C8—N1—C6—C5	33.0 (4)	C4—C5—C6—C7	1.4 (4)
C8—N1—C6—C7	−150.8 (3)	N1—C6—C7—C2	−177.6 (2)
C6—N1—C8—C9	−175.0 (2)	C5—C6—C7—C2	−1.3 (4)
O1—C1—C2—C3	178.2 (2)	N1—C8—C9—C10	178.1 (3)
O1—C1—C2—C7	−1.6 (3)	N1—C8—C9—C14	−0.8 (4)
O2—C1—C2—C3	−1.4 (4)	C8—C9—C10—F1	2.2 (4)
O2—C1—C2—C7	178.8 (2)	C8—C9—C10—C11	−177.0 (3)
C1—C2—C3—O3	−0.6 (4)	C14—C9—C10—F1	−178.9 (3)
C1—C2—C3—C4	−179.9 (2)	C14—C9—C10—C11	1.9 (4)
C7—C2—C3—O3	179.2 (2)	C8—C9—C14—C13	178.3 (3)
C7—C2—C3—C4	−0.1 (4)	C10—C9—C14—C13	−0.6 (4)
C1—C2—C7—C6	−179.6 (2)	F1—C10—C11—C12	179.2 (3)
C3—C2—C7—C6	0.6 (4)	C9—C10—C11—C12	−1.6 (5)
O3—C3—C4—C5	−179.1 (2)	C10—C11—C12—C13	−0.2 (5)
C2—C3—C4—C5	0.2 (4)	C11—C12—C13—C14	1.5 (5)
C3—C4—C5—C6	−0.8 (4)	C12—C13—C14—C9	−1.1 (5)
C4—C5—C6—N1	177.5 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.88 (3)	1.77 (3)	2.642 (3)	176 (3)
O3—H3···O2	0.92 (3)	1.78 (3)	2.617 (3)	152 (3)

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

Fig. 1

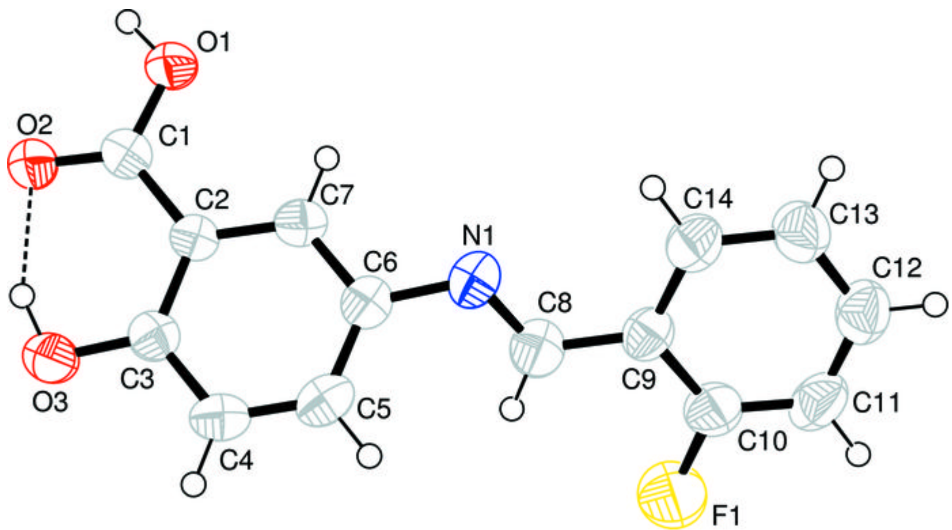

Fig. 2