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2-{(*E*)-[(3-Chloro-4-methylphenyl)imino]methyl}-4-(trifluoromethoxy)phenol

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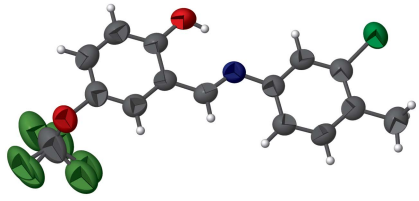
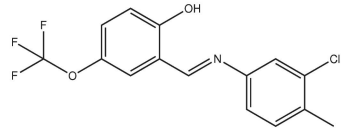
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{11}ClF_3NO_2$, the dihedral angle between the planes of the benzene rings is 8.3 (2)° and an intramolecular O—H···N hydrogen bond closes an *S*(6) ring. In the crystal, weak C—H···O links connect the molecules into [$\bar{1}$10] chains. The F atoms of the CF_3 group are disordered over two sets of sites with refined occupancies of 0.626 (11):0.374 (11).

3D view



Chemical scheme



Structure description

We herein report the synthesis and structure of the new Schiff base (Fig. 1). The bond distances of the imino group atoms [N1–C9 = 1.423 (5); N1—C8 = 1.275 (5) Å] are consistent with those in the related structures 2-[(2- bromophenyl)iminomethyl]-6-methylphenol (Karadağ *et al.*, 2010) and 2-amino-3-((*E*)-{[3-(trifluoromethyl)phenyl]-imino}-methyl)-4*H*-chromen-4-one (Atalay *et al.*, 2016). The dihedral angle between the benzene rings is 8.3 (2)° and an intramolecular O—H···N hydrogen bond (Table 1) closes an *S*(6) ring.

In the crystal (Fig. 2), weak C—H···O links connect the molecules into [$\bar{1}$10] chains.

Synthesis and crystallization

A mixture of 2-hydroxy-5-(trifluoromethoxy)benzaldehyde (0.19 g, 0.9 mol) in ethanol (20 ml) and 3-chloro-4-methyaniline (0.13 g, 0.9 mol) in ethanol (20 ml) were mixed and stirred for 5 h under reflux. Light-yellow plate-shaped crystals were obtained by slow evaporation of an ethanol solution (yield 67%; m.p. 361–363 K).

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1	0.82	1.88	2.609 (5)	148
C10—H10···O2^{i}	0.93	2.52	3.277 (5)	139

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

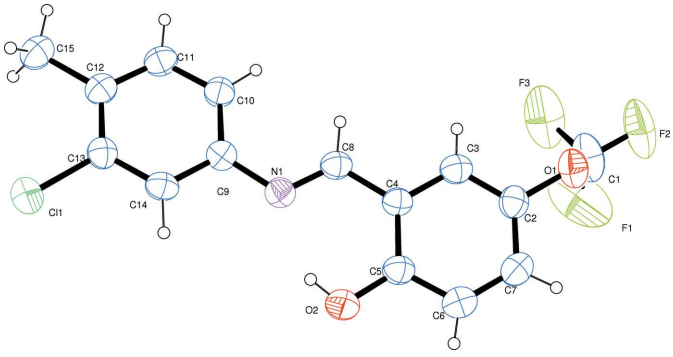


Figure 1
The molecular structure showing 30% probability displacement ellipsoids. Only the major-disorder component of the CF_3 group is shown.

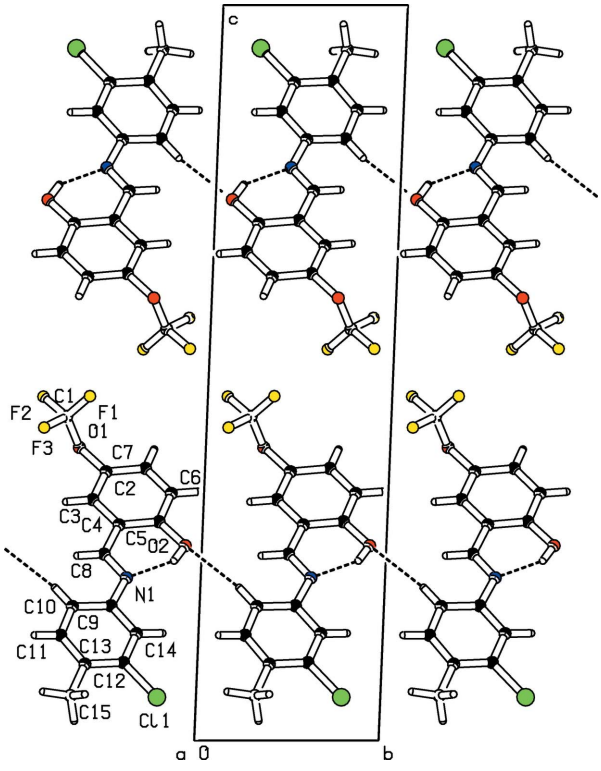


Figure 2
The packing, viewed along [100] with hydrogen bonds shown as dashed lines..

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{11}ClF_3NO_2$
M_r	329.70
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	4.5971 (4), 6.4372 (7), 25.237 (2)
α, β, γ (°)	86.984 (8), 86.173 (7), 78.096 (8)
V (Å^3)	728.57 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.30
Crystal size (mm)	0.42 × 0.24 × 0.05
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-AREA*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.907, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6174, 2581, 1173
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.061, 0.181, 0.89
No. of reflections	2581
No. of parameters	228
No. of restraints	96
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.29

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXT2016* (Sheldrick, 2015), *SHELXL2016* (Sheldrick, 2015) and *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The F atoms of the CF_3 group are disordered over two sets of sites with refined occupancies of 0.626 (11):0.374 (11).

Funding information

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References

Atalay, Ş., Gerçeker, S., Meral, S. & Alaman Ağar, A. (2016). *IUCrData*, **1**, x160797.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Karadağ, A. T,, Atalay, Ş. & Genç, H. (2010). *Acta Cryst.* E**66**, o2977.
Sheldrick, G. M. (2015*a*). *Acta Cryst*. A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst*. C**71**, 3–8.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

full crystallographic data

IUCrData (2017). **2**, x171725 [https://doi.org/10.1107/S2414314617017254]

2-{(*E*)-[(3-Chloro-4-methylphenyl)imino]methyl}-4-(trifluoromethoxy)phenol

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Crystal data

$C_{15}H_{11}ClF_3NO_2$
M_r = 329.70
Triclinic, $P\overline{1}$
a = 4.5971 (4) Å
b = 6.4372 (7) Å
c = 25.237 (2) Å
α = 86.984 (8)°
β = 86.173 (7)°
γ = 78.096 (8)°
V = 728.57 (12) Å^3
Z = 2
$F(000)$ = 336
D_x = 1.503 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4834 reflections
θ = 1.6–27.4°
μ = 0.30 mm^{-1}
T = 293 K
Plate, light yellow
0.42 × 0.24 × 0.05 mm

Data collection

Stoe IPDS 2 diffractometer
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-AREA; Stoe & Cie, 2002)
T_{min} = 0.907, T_{max} = 0.986
6174 measured reflections
2581 independent reflections
1173 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −5→5
k = −7→7
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.181
S = 0.89
2581 reflections
228 parameters
96 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.098P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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 C10 and C16 were placed in calculated positions and refined using a riding model, with C–H distances of 0.93 Å and methyl C–H distances 0.96 Å. All other H atoms were freely refined.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	1.2010 (15)	0.7569 (14)	0.5585 (3)	0.1286 (17)	
C2	1.2114 (9)	0.5356 (8)	0.63470 (17)	0.0694 (12)	
C3	0.9931 (9)	0.6027 (7)	0.67272 (17)	0.0691 (11)	
H3	0.914764	0.747080	0.675766	0.083*	
C4	0.8865 (8)	0.4547 (6)	0.70721 (15)	0.0595 (10)	
C5	1.0128 (9)	0.2385 (7)	0.70263 (17)	0.0676 (11)	
C6	1.2400 (11)	0.1773 (8)	0.6644 (2)	0.0847 (13)	
H6	1.327412	0.034117	0.661899	0.102*	
C7	1.3387 (10)	0.3233 (8)	0.63015 (19)	0.0795 (13)	
H7	1.489359	0.279952	0.604209	0.095*	
C8	0.6507 (9)	0.5284 (7)	0.74676 (16)	0.0647 (11)	
H8	0.577359	0.673819	0.748866	0.078*	
C9	0.3055 (8)	0.4808 (7)	0.81702 (15)	0.0609 (10)	
C10	0.1480 (9)	0.6892 (7)	0.81789 (17)	0.0704 (12)	
H10	0.190670	0.788803	0.792070	0.084*	
C11	−0.0704 (9)	0.7473 (7)	0.85695 (18)	0.0736 (12)	
H11	−0.171975	0.888128	0.856866	0.088*	
C12	−0.1488 (9)	0.6093 (7)	0.89651 (16)	0.0658 (11)	
C13	0.0067 (9)	0.4005 (7)	0.89411 (16)	0.0660 (11)	
C14	0.2288 (9)	0.3368 (7)	0.85488 (17)	0.0663 (11)	
H14	0.326900	0.195268	0.854176	0.080*	
C15	−0.3891 (10)	0.6817 (8)	0.93910 (18)	0.0837 (13)	
H15A	−0.467896	0.830850	0.933691	0.125*	
H15B	−0.306594	0.655789	0.973358	0.125*	
H15C	−0.545647	0.604398	0.937355	0.125*	
Cl1	−0.0746 (3)	0.2116 (2)	0.94138 (5)	0.0940 (5)	
F1A	1.229 (3)	0.6214 (16)	0.5215 (3)	0.150 (3)	0.374 (11)
F2A	1.287 (4)	0.918 (3)	0.5377 (6)	0.141 (3)	0.374 (11)
F3A	0.8893 (14)	0.837 (3)	0.5742 (4)	0.145 (3)	0.374 (11)
F1B	1.036 (2)	0.6539 (13)	0.5373 (3)	0.139 (2)	0.626 (11)
F2B	1.335 (2)	0.8762 (19)	0.5297 (4)	0.151 (3)	0.626 (11)
F3B	0.9360 (13)	0.9123 (13)	0.5759 (2)	0.142 (2)	0.626 (11)
N1	0.5390 (7)	0.4024 (5)	0.77887 (13)	0.0641 (9)	
O1	1.3285 (6)	0.6855 (5)	0.60186 (13)	0.0859 (10)	
O2	0.9184 (7)	0.0908 (5)	0.73535 (13)	0.0896 (10)	
H2	0.783064	0.149011	0.755697	0.134*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.108 (3)	0.180 (4)	0.111 (4)	−0.070 (3)	−0.009 (3)	0.039 (3)
C2	0.059 (3)	0.083 (3)	0.069 (3)	−0.021 (2)	−0.005 (2)	−0.003 (2)
C3	0.062 (3)	0.069 (3)	0.076 (3)	−0.011 (2)	−0.008 (2)	−0.002 (2)
C4	0.058 (2)	0.055 (2)	0.064 (2)	−0.008 (2)	−0.009 (2)	−0.003 (2)
C5	0.068 (3)	0.057 (3)	0.075 (3)	−0.004 (2)	−0.007 (2)	−0.003 (2)

C6	0.083 (3)	0.070 (3)	0.096 (3)	−0.003 (3)	0.001 (3)	−0.013 (3)
C7	0.068 (3)	0.083 (4)	0.084 (3)	−0.007 (3)	0.003 (2)	−0.014 (3)
C8	0.060 (2)	0.057 (3)	0.074 (3)	−0.004 (2)	−0.008 (2)	−0.006 (2)
C9	0.056 (2)	0.066 (3)	0.061 (2)	−0.012 (2)	−0.010 (2)	−0.001 (2)
C10	0.074 (3)	0.063 (3)	0.070 (3)	−0.006 (2)	−0.003 (2)	0.003 (2)
C11	0.066 (3)	0.065 (3)	0.084 (3)	0.002 (2)	−0.003 (2)	−0.006 (2)
C12	0.064 (3)	0.072 (3)	0.064 (3)	−0.014 (2)	−0.012 (2)	−0.011 (2)
C13	0.066 (3)	0.070 (3)	0.064 (3)	−0.019 (2)	−0.008 (2)	0.000 (2)
C14	0.062 (3)	0.058 (3)	0.077 (3)	−0.006 (2)	−0.010 (2)	0.000 (2)
C15	0.077 (3)	0.092 (3)	0.081 (3)	−0.013 (3)	0.002 (3)	−0.016 (3)
Cl1	0.1027 (9)	0.0869 (9)	0.0912 (9)	−0.0253 (7)	0.0124 (7)	0.0085 (7)
F1A	0.155 (6)	0.217 (5)	0.097 (5)	−0.082 (5)	−0.017 (4)	0.019 (4)
F2A	0.113 (5)	0.177 (5)	0.134 (5)	−0.053 (5)	−0.002 (5)	0.062 (4)
F3A	0.104 (4)	0.189 (7)	0.145 (5)	−0.054 (4)	−0.019 (4)	0.064 (5)
F1B	0.134 (4)	0.203 (5)	0.096 (4)	−0.075 (3)	−0.024 (3)	0.023 (3)
F2B	0.125 (4)	0.204 (5)	0.128 (4)	−0.065 (4)	0.003 (3)	0.072 (4)
F3B	0.116 (3)	0.171 (5)	0.144 (4)	−0.050 (3)	−0.017 (3)	0.050 (3)
N1	0.061 (2)	0.065 (2)	0.065 (2)	−0.0095 (17)	−0.0083 (17)	0.0029 (17)
O1	0.0700 (19)	0.104 (3)	0.084 (2)	−0.0227 (18)	−0.0023 (16)	0.0140 (19)
O2	0.100 (2)	0.0600 (19)	0.098 (2)	0.0005 (17)	0.0112 (19)	0.0085 (17)

Geometric parameters (Å, º)

C1—F2B	1.255 (7)	C8—N1	1.275 (5)
C1—F2A	1.263 (7)	C8—H8	0.9300
C1—F1B	1.267 (8)	C9—C14	1.376 (5)
C1—O1	1.293 (7)	C9—C10	1.387 (6)
C1—F1A	1.293 (8)	C9—N1	1.423 (5)
C1—F3A	1.455 (9)	C10—C11	1.368 (6)
C1—F3B	1.466 (8)	C10—H10	0.9300
C2—C3	1.360 (6)	C11—C12	1.381 (6)
C2—C7	1.379 (6)	C11—H11	0.9300
C2—O1	1.406 (5)	C12—C13	1.388 (6)
C3—C4	1.398 (5)	C12—C15	1.508 (6)
C3—H3	0.9300	C13—C14	1.388 (6)
C4—C5	1.401 (6)	C13—Cl1	1.737 (4)
C4—C8	1.446 (5)	C14—H14	0.9300
C5—O2	1.347 (5)	C15—H15A	0.9600
C5—C6	1.383 (6)	C15—H15B	0.9600
C6—C7	1.369 (6)	C15—H15C	0.9600
C6—H6	0.9300	O2—H2	0.8200
C7—H7	0.9300		
F2B—C1—F1B	119.1 (8)	N1—C8—C4	122.7 (4)
F2B—C1—O1	114.6 (6)	N1—C8—H8	118.6
F2A—C1—O1	113.5 (7)	C4—C8—H8	118.6
F1B—C1—O1	120.7 (7)	C14—C9—C10	118.4 (4)
F2A—C1—F1A	106.5 (12)	C14—C9—N1	116.9 (4)

O1—C1—F1A	115.4 (7)	C10—C9—N1	124.7 (4)
F2A—C1—F3A	103.6 (12)	C11—C10—C9	119.6 (4)
O1—C1—F3A	105.9 (6)	C11—C10—H10	120.2
F1A—C1—F3A	111.4 (10)	C9—C10—H10	120.2
F2B—C1—F3B	99.7 (9)	C10—C11—C12	123.9 (4)
F1B—C1—F3B	89.5 (6)	C10—C11—H11	118.1
O1—C1—F3B	104.8 (6)	C12—C11—H11	118.1
C3—C2—C7	121.5 (4)	C11—C12—C13	115.5 (4)
C3—C2—O1	119.8 (4)	C11—C12—C15	121.9 (4)
C7—C2—O1	118.5 (4)	C13—C12—C15	122.6 (4)
C2—C3—C4	120.0 (4)	C14—C13—C12	121.9 (4)
C2—C3—H3	120.0	C14—C13—Cl1	118.4 (3)
C4—C3—H3	120.0	C12—C13—Cl1	119.8 (3)
C3—C4—C5	118.9 (4)	C9—C14—C13	120.7 (4)
C3—C4—C8	119.3 (4)	C9—C14—H14	119.6
C5—C4—C8	121.8 (4)	C13—C14—H14	119.6
O2—C5—C6	119.9 (4)	C12—C15—H15A	109.5
O2—C5—C4	120.8 (4)	C12—C15—H15B	109.5
C6—C5—C4	119.3 (4)	H15A—C15—H15B	109.5
C7—C6—C5	121.3 (5)	C12—C15—H15C	109.5
C7—C6—H6	119.3	H15A—C15—H15C	109.5
C5—C6—H6	119.3	H15B—C15—H15C	109.5
C6—C7—C2	119.0 (5)	C8—N1—C9	121.1 (4)
C6—C7—H7	120.5	C1—O1—C2	119.2 (4)
C2—C7—H7	120.5	C5—O2—H2	109.5

C7—C2—C3—C4	−2.0 (6)	C11—C12—C13—C14	0.9 (6)
O1—C2—C3—C4	−176.8 (4)	C15—C12—C13—C14	−179.5 (4)
C2—C3—C4—C5	1.5 (6)	C11—C12—C13—Cl1	−179.4 (3)
C2—C3—C4—C8	−178.9 (4)	C15—C12—C13—Cl1	0.2 (6)
C3—C4—C5—O2	179.4 (4)	C10—C9—C14—C13	−2.3 (6)
C8—C4—C5—O2	−0.2 (6)	N1—C9—C14—C13	179.0 (3)
C3—C4—C5—C6	0.3 (6)	C12—C13—C14—C9	0.8 (6)
C8—C4—C5—C6	−179.3 (4)	Cl1—C13—C14—C9	−178.9 (3)
O2—C5—C6—C7	179.3 (4)	C4—C8—N1—C9	−179.5 (3)
C4—C5—C6—C7	−1.6 (7)	C14—C9—N1—C8	−170.8 (4)
C5—C6—C7—C2	1.1 (7)	C10—C9—N1—C8	10.6 (6)
C3—C2—C7—C6	0.7 (7)	F2B—C1—O1—C2	−174.4 (10)
O1—C2—C7—C6	175.6 (4)	F2A—C1—O1—C2	167.0 (13)
C3—C4—C8—N1	178.7 (4)	F1B—C1—O1—C2	−21.0 (11)
C5—C4—C8—N1	−1.6 (6)	F1A—C1—O1—C2	−69.7 (10)
C14—C9—C10—C11	2.1 (6)	F3A—C1—O1—C2	54.0 (11)
N1—C9—C10—C11	−179.2 (4)	F3B—C1—O1—C2	77.4 (7)
C9—C10—C11—C12	−0.4 (7)	C3—C2—O1—C1	−87.8 (6)
C10—C11—C12—C13	−1.1 (6)	C7—C2—O1—C1	97.2 (6)
C10—C11—C12—C15	179.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.88	2.609 (5)	148
C10—H10···O2^{i}	0.93	2.52	3.277 (5)	139

Symmetry code: (i) *x*−1, *y*+1, *z*.