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N-[3,5-Bis(trifluoromethyl)phenyl]salicylaldimine

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Key indicators

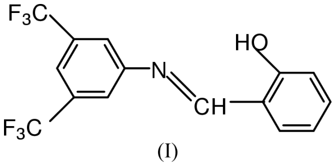
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.063
wR factor = 0.191
Data-to-parameter ratio = 9.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{15}H_9F_6NO$, is non-planar with a dihedral angle between the two aromatic rings of 29.57 (2)°. The central N=C bond distance of 1.276 (4) Å is typical for a double bond. There are intramolecular O—H···N and weak intermolecular C—H···F hydrogen bonds.

Comment

Solvent extraction is now widely accepted as a process for the recovery of metal ions from aqueous solution in hydrometallurgical and environmental applications (El Aamrani *et al.*, 1999), with a new interest in the processing of heavy metals by chelation in combination with supercritical fluid (SCF) extraction processing. We have designed novel Schiff base-type chelating agents by attaching CO_2-philic fluoroalkyl groups, and synthesized and characterized these compounds for supercritical CO_2 extraction of heavy metals. In this study, we report the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The C8—N1 and C7—C6 bond lengths are 1.417 (4) and 1.439 (4) Å, respectively (Table 1), and agree with the corresponding distances in 2-[(4-hydroxyphenyl)iminomethyl]thiophene [1.422 (2) and 1.447 (3) Å; Kazak *et al.*, 2000] and *trans*-[4-dimethylaminophenyl)iminomethyl]-*N*-methylpyridinium *para*-toluenesulfonate [1.413 (3) and 1.461 Å; Coe *et al.*, 2001]. The N1=C7 bond length of 1.276 (4) Å is typical of a double bond, similar to the corresponding bond lengths in *N*-(2,4-dinitrophenyl)–*N*-methylhydrazone [1.279 (2) Å; Aygün *et al.*, 1998], 2-salicylideneamino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carbonitrile [1.281 (2) Å; Elerman & Elmalı, 1998] and 2-[(4-hydroxyphenyl)iminomethyl]thiophene [1.282 (2) Å; Kazak *et al.*, 2000]. The O1—C5 distance of 1.360 (4) Å is close to the value of 1.349 (6) Å in 3-*tert*-butyl-2-hydroxy-5-methoxyazobenzene (Işık *et al.*, 1998). The dihedral angle between the rings formed by atoms C1–C6 and C8–C13 is 29.57 (2)°.

There is an intramolecular O1—H1···N1 hydrogen bond and a weak intermolecular C7—H7···F1*a*^{i} hydrogen bond (Table 2). Both CF_3 groups show rotational disorder.

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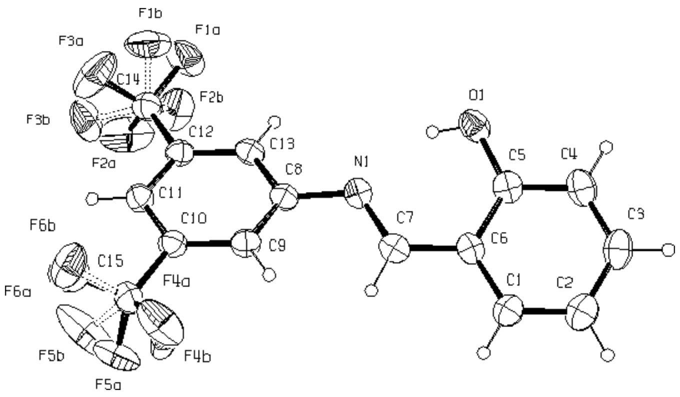

Figure 1
An *ORTEP*III (Burnett & Johnson, 1996) drawing of (I), showing the atomic numbering scheme. Displacement ellipsoids for non-H atoms are shown at the 30% probability level. The site-occupancy factors are 0.64 (1) and 0.36 (1) for F1*a*—F6*a* and F1*b*—F6*b*, respectively.

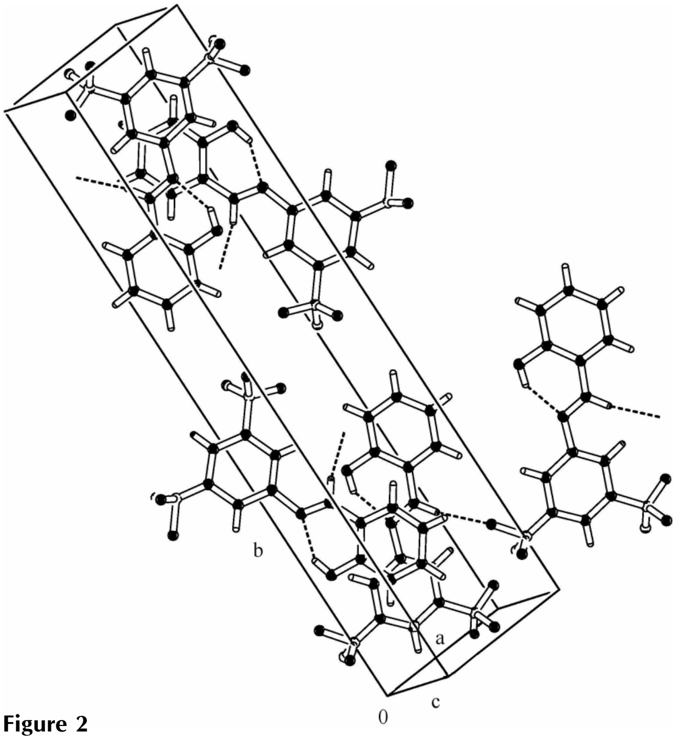

Figure 2
PLATON/*PLUTON* plot (Spek, 1997), showing part of the hydrogen-bond network and the stacking of molecules. The disordered F atoms with 36% occupancy have been omitted for clarity.

Experimental

An ethanol solution of 3,5-bis(trifluoromethyl)aminobenzene (2.29 g/20 ml ethanol) was added dropwise, at room temperature, to a solution of salicylaldehyde (1.22 g, 0.01 mol) in ethanol (20 ml), and large amounts of a yellow precipitate were formed. The mixture was stirred for 30 min at this temperature and filtered under vacuum. Spectroscopically pure (I) was obtained by crystallization from toluene as pale-yellow needles (yield: 93%, m.p.: 369–371 K); elemental analysis, calculated for $C_{15}H_9F_6N$: C 54.05, H 2.70, N 4.20%; found: C 54.34, H 2.78, N 4.19%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.9–7.1 (*m*, 7H, Ar—H), 8.5 (*s*, 1H, H—C═N), 12.2 (*s*, 1H, OH). IR (KBr) 3600–3300 (*br*, Ph—OH), 2950 (H—CN), 1650 (C═N), 1600 (Ar—CH). UV–vis ($CHCl_3$): λ = 240 (ε: 2000), 276 (max., ε: 2927), 346 nm (ε: 1835).

Crystal data

$C_{15}H_9F_6NO$
$M_r = 333.23$
Monoclinic, $P2_1/a$
$a = 7.509$ (1) Å
$b = 26.634$ (2) Å
$c = 7.854$ (1) Å
$\beta = 117.34$ (1)°
$V = 1395.3$ (3) Å^3
$Z = 4$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 35 reflections
$\theta = 5$–25°
$\mu = 0.16$ mm^{-1}
$T = 180$ (2) K
Plate, yellow
0.30 × 0.30 × 0.02 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
9212 measured reflections
2508 independent reflections
1400 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\rm max} = 25.2°$
$h = -4 \rightarrow 9$
$k = -30 \rightarrow 31$
$l = -9 \rightarrow 9$
1 standard reflection
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.191$
$S = 1.06$
2508 reflections
264 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0908P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.062$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.011 (3)

Table 1
Selected geometric parameters (Å, °).

C12—C11	1.375 (4)	N1—C8	1.417 (4)
C12—C13	1.379 (5)	C1—C6	1.381 (4)
O1—C5	1.360 (4)	C6—C7	1.439 (4)
N1—C7	1.276 (4)	C8—C9	1.377 (4)
C11—C12—C13	120.7 (3)	C1—C6—C7	120.8 (3)
C11—C12—C14	120.0 (3)	C5—C6—C7	120.6 (3)
C13—C12—C14	119.3 (3)	N1—C7—C6	123.5 (3)
C7—N1—C8	119.5 (3)	C9—C8—N1	123.6 (3)
O1—C5—C4	117.8 (3)	C13—C8—N1	117.1 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.88	2.604 (4)	147
C7—H7···F1*a*$^{\rm i}$	0.93	2.48	3.351 (8)	156

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

The disordered trifluoromethyl F atoms were refined anisotropically, with geometrical and displacement parameter restraints for the two sets of positions; the refined occupancy factors were 0.639 (14) and 0.361 (14).

Data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999), *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1997).

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