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2-Fluoro-*N*-(3-nitrobenzylidene)-5-(trifluoromethyl)aniline

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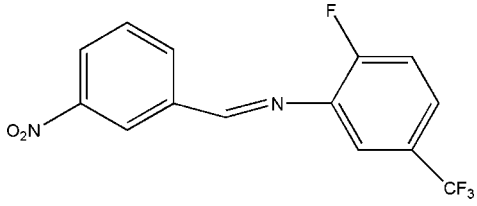
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.049; wR factor = 0.155; data-to-parameter ratio = 11.5.

In the title Schiff base, $C_{14}H_8F_4N_2O$, the dihedral angle between the two rings is 20.92 (4)°. The molecular components are stabilized *via* π–π stacking interactions between adjacent benzene rings of the Schiff base units; the centroid-to-centroid distance is 3.834 (3) Å. The F atoms of the trifluoromethyl group are disordered over two sites in a ratio of *ca* 0.9:0.1.

Related literature

For related literature, see: Alemi & Shaabani (2000); Alizadeh *et al.* (1999); Johnson *et al.* (1996); Kim & Shin (1999); Wang & Zheng (2007).



Experimental

Crystal data

$C_{14}H_8F_4N_2O_2$
M_r = 312.22
Monoclinic, $P2_1/c$
a = 12.863 (3) Å
b = 12.549 (2) Å
c = 8.3211 (16) Å
β = 90.10 (1)°
V = 1343.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 298 (2) K
0.38 × 0.31 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.975
8038 measured reflections
2406 independent reflections
1526 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.155
S = 1.00
2406 reflections
210 parameters
96 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Data collection: *SMART* (Bruker,1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2153).

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supplementary materials

Acta Cryst. (2007). E**63**, o3202 [**doi:10.1107/S1600536807028395**]

2-Fluoro-*N*-(3-nitrobenzylidene)-5-(trifluoromethyl)aniline

M.-H. Yang, G.-B. Yan and Y.-F. Zheng

Comment

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes (Johnson *et al.*, 1996; Alizadeh *et al.*, 1999; Wang & Zheng, 2007). Schiff bases that have solvent-dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optically active) materials (Alemi & Shaabani, 2000). They are also useful in the asymmetric oxidation of methyl phenyl sulfide and are enantioselective (Kim & Shim, 1999). In this paper, we report the synthesis and crystal structure of the title compound.

In the molecular structure of the title compound (Fig. 1), the C8═N2 bond length is 1.270 (3) Å, indicative of a C═N double bond. The C—F, C—O and C—C distances are unremarkable. The F atoms of the trifluoromethyl group were found to be disordered over two positions, the two components being rotated by about 30°.

Tha packing is governed by π···π stacking interactions. The centroid-centroid distance between adjacent benzene rings (at x, y, z and x, 1/2 − y, 1/2 + z) is 3.834 (3) Å, indicating a normal π···π contact.

Experimental

Under nitrogen, a mixture of 3-nitrobenzaldehyde (1.51 g,10 mmol), Na_2SO_4 (3.0 g) and 5-fluoro-2-trifluoromethybeneze-namine (1.79 g, 10 mmol) in absolute ethanol (20 ml) was refluxed for about 12 h to yield a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2 *x* 15 ml) and brine (8 ml). After drying over Na_2SO_4, the solvent was removed under vacuum, and a yellow solid was isolated in 92% yield (3.1 g). Colourless single crystals of the Schiff base suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol (4:1) by slow evaporation of the solvents at room temperature over a period of about one week.

Refinement

All H atoms were placed in calculated positions [Csp^2—H = 0.93 Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The site occupancy factors of atoms F2, F3, F4 and F2', F3' F4' refined to 0.888 (4) and 0.112 (4), respectively.

Figures

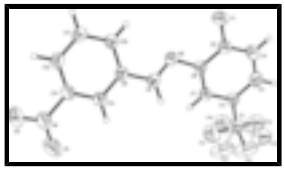


Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Both disorder components are shown.

2-Fluoro-*N*-(3-Nitrobenzylidene)-5-(trifluoromethyl)aniline

Crystal data

$C_{14}H_8F_4N_2O_2$	F_{000} = 632
M_r = 312.22	D_x = 1.544 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2406 reflections
a = 12.863 (3) Å	θ = 2.3–25.3º
b = 12.549 (2) Å	μ = 0.14 mm^{-1}
c = 8.3211 (16) Å	T = 298 (2) K
β = 90.10 (1)º	Block, colourless
V = 1343.1 (4) Å^3	0.38 × 0.31 × 0.18 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	2406 independent reflections
Radiation source: fine-focus sealed tube	1526 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.087
T = 298(2) K	θ_{max} = 25.3º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→14
T_{min} = 0.948, T_{max} = 0.975	k = −15→15
8038 measured reflections	l = −10→9

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.155	$w = 1/[\sigma^2(F_o^2) + (0.089P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
2406 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
210 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
96 restraints	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.006 (3)

Special details

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

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
F1	0.19004 (12)	0.98237 (12)	−0.08870 (18)	0.0833 (5)	
F2	0.59865 (15)	0.96853 (16)	0.3082 (3)	0.1065 (8)	0.888 (4)
F3	0.50517 (18)	0.8671 (3)	0.4495 (2)	0.1311 (11)	0.888 (4)
F4	0.5854 (2)	0.8061 (2)	0.2409 (4)	0.1325 (11)	0.888 (4)
N2	0.24083 (14)	0.77557 (14)	−0.0569 (2)	0.0564 (5)	
N1	0.1229 (2)	0.30915 (18)	−0.0163 (3)	0.0858 (7)	
O2	0.0583 (2)	0.24421 (15)	−0.0567 (3)	0.1127 (8)	
O1	0.1894 (2)	0.28961 (18)	0.0814 (4)	0.1400 (11)	
C8	0.22886 (17)	0.68459 (18)	0.0073 (3)	0.0560 (6)	
H8	0.2590	0.6716	0.1069	0.067*	
C6	0.29922 (16)	0.85362 (17)	0.0263 (2)	0.0520 (6)	
C14	0.17518 (18)	0.49656 (18)	−0.0107 (3)	0.0579 (6)	
H14	0.2178	0.4809	0.0764	0.069*	
C7	0.38544 (17)	0.83251 (18)	0.1211 (3)	0.0557 (6)	
H7	0.4077	0.7625	0.1341	0.067*	
C5	0.27186 (19)	0.95961 (18)	0.0080 (3)	0.0598 (6)	
C13	0.11706 (18)	0.41783 (17)	−0.0826 (3)	0.0595 (6)	
C2	0.43918 (18)	0.91442 (19)	0.1972 (3)	0.0606 (6)	
C9	0.16923 (16)	0.59942 (17)	−0.0702 (2)	0.0510 (6)	
C11	0.04589 (19)	0.53895 (19)	−0.2696 (3)	0.0639 (6)	
H11	0.0028	0.5537	−0.3565	0.077*	
C12	0.05175 (19)	0.43717 (19)	−0.2105 (3)	0.0641 (6)	
H12	0.0126	0.3825	−0.2557	0.077*	
C3	0.4067 (2)	1.0184 (2)	0.1792 (3)	0.0710 (7)	
H3	0.4421	1.0730	0.2312	0.085*	
C1	0.53030 (17)	0.88854 (16)	0.2968 (2)	0.0805 (8)	
F2'	0.5717 (13)	0.9541 (12)	0.4066 (16)	0.1065 (8)	0.112 (4)
F3'	0.5223 (16)	0.7972 (9)	0.379 (2)	0.1311 (11)	0.112 (4)
F4'	0.6153 (9)	0.8669 (16)	0.210 (2)	0.1325 (11)	0.112 (4)
C10	0.10359 (16)	0.61967 (18)	−0.2007 (2)	0.0571 (6)	
H10	0.0988	0.6885	−0.2417	0.069*	
C4	0.3218 (2)	1.04161 (19)	0.0841 (3)	0.0727 (7)	
H4	0.2990	1.1115	0.0719	0.087*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0829 (11)	0.0742 (10)	0.0930 (10)	0.0090 (7)	−0.0250 (8)	0.0103 (7)
F2	0.0812 (14)	0.1240 (17)	0.1144 (18)	−0.0439 (11)	−0.0324 (12)	0.0193 (13)
F3	0.1160 (16)	0.198 (3)	0.0790 (14)	−0.0485 (18)	−0.0326 (12)	0.0497 (15)
F4	0.1058 (17)	0.113 (2)	0.178 (2)	0.0311 (15)	−0.0713 (16)	−0.0253 (18)
N2	0.0582 (12)	0.0519 (11)	0.0590 (11)	−0.0046 (9)	−0.0073 (9)	−0.0036 (9)
N1	0.0954 (18)	0.0547 (14)	0.1074 (18)	0.0008 (13)	−0.0218 (15)	−0.0023 (12)
O2	0.146 (2)	0.0564 (12)	0.1361 (19)	−0.0302 (13)	−0.0375 (16)	0.0007 (11)
O1	0.149 (2)	0.0699 (14)	0.201 (3)	−0.0046 (14)	−0.087 (2)	0.0301 (15)
C8	0.0571 (13)	0.0566 (14)	0.0544 (13)	−0.0035 (11)	−0.0078 (10)	−0.0025 (10)
C6	0.0537 (13)	0.0506 (13)	0.0518 (12)	−0.0084 (10)	−0.0010 (10)	−0.0002 (10)
C14	0.0574 (14)	0.0555 (14)	0.0607 (13)	0.0021 (11)	−0.0121 (11)	−0.0035 (11)
C7	0.0568 (14)	0.0504 (13)	0.0598 (13)	−0.0035 (10)	−0.0040 (11)	−0.0001 (10)
C5	0.0587 (14)	0.0585 (15)	0.0623 (13)	0.0015 (11)	−0.0075 (11)	0.0033 (11)
C13	0.0611 (14)	0.0440 (13)	0.0733 (15)	−0.0002 (11)	−0.0051 (12)	−0.0033 (11)
C2	0.0615 (14)	0.0598 (15)	0.0605 (13)	−0.0123 (12)	−0.0051 (11)	−0.0012 (11)
C9	0.0493 (12)	0.0505 (13)	0.0532 (12)	−0.0028 (10)	−0.0030 (9)	−0.0047 (10)
C11	0.0617 (15)	0.0664 (16)	0.0634 (14)	−0.0064 (12)	−0.0172 (11)	−0.0024 (12)
C12	0.0639 (15)	0.0569 (14)	0.0715 (15)	−0.0093 (12)	−0.0088 (12)	−0.0113 (12)
C3	0.0803 (18)	0.0568 (15)	0.0758 (16)	−0.0196 (13)	−0.0098 (14)	−0.0060 (12)
C1	0.0770 (18)	0.0820 (19)	0.0824 (19)	−0.0195 (16)	−0.0213 (14)	0.0061 (15)
F2'	0.0812 (14)	0.1240 (17)	0.1144 (18)	−0.0439 (11)	−0.0324 (12)	0.0193 (13)
F3'	0.1160 (16)	0.198 (3)	0.0790 (14)	−0.0485 (18)	−0.0326 (12)	0.0497 (15)
F4'	0.1058 (17)	0.113 (2)	0.178 (2)	0.0311 (15)	−0.0713 (16)	−0.0253 (18)
C10	0.0580 (14)	0.0556 (14)	0.0579 (13)	−0.0042 (11)	−0.0051 (11)	0.0017 (10)
C4	0.0902 (19)	0.0472 (14)	0.0806 (17)	−0.0046 (13)	−0.0061 (15)	0.0025 (12)

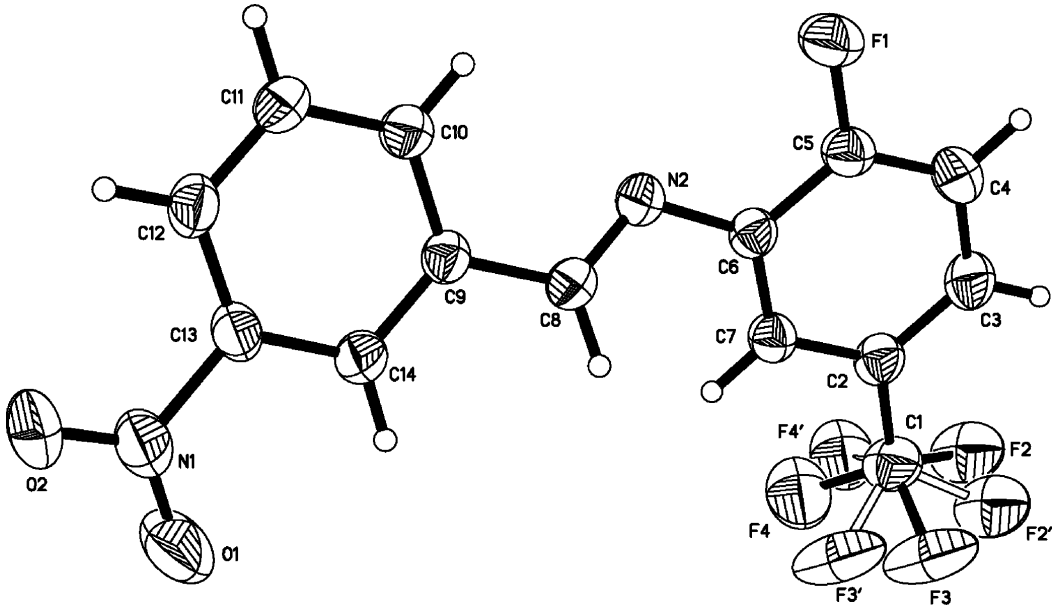
Geometric parameters (Å, °)

F1—C5	1.355 (3)	C7—H7	0.9300
F2—C1	1.3377 (10)	C5—C4	1.368 (3)
F3—C1	1.3381 (10)	C13—C12	1.378 (3)
F4—C1	1.3380 (10)	C2—C3	1.378 (4)
N2—C8	1.270 (3)	C2—C1	1.472 (3)
N2—C6	1.415 (3)	C9—C10	1.399 (3)
N1—O1	1.205 (3)	C11—C12	1.371 (3)
N1—O2	1.212 (3)	C11—C10	1.380 (3)
N1—C13	1.473 (3)	C11—H11	0.9300
C8—C9	1.465 (3)	C12—H12	0.9300
C8—H8	0.9300	C3—C4	1.380 (4)
C6—C5	1.384 (3)	C3—H3	0.9300
C6—C7	1.387 (3)	C1—F2'	1.3395 (11)
C14—C13	1.376 (3)	C1—F4'	1.3398 (11)
C14—C9	1.385 (3)	C1—F3'	1.3399 (11)
C14—H14	0.9300	C10—H10	0.9300
C7—C2	1.391 (3)	C4—H4	0.9300

C8—N2—C6	118.75 (19)	C11—C12—H12	120.8
O1—N1—O2	122.5 (3)	C13—C12—H12	120.8
O1—N1—C13	118.5 (2)	C2—C3—C4	120.2 (2)
O2—N1—C13	118.9 (2)	C2—C3—H3	119.9
N2—C8—C9	122.3 (2)	C4—C3—H3	119.9
N2—C8—H8	118.8	F2—C1—F4	104.9 (2)
C9—C8—H8	118.8	F2—C1—F3	104.06 (17)
C5—C6—C7	116.7 (2)	F4—C1—F3	107.6 (2)
C5—C6—N2	118.4 (2)	F2—C1—F2'	39.6 (7)
C7—C6—N2	124.8 (2)	F4—C1—F2'	120.1 (9)
C13—C14—C9	119.0 (2)	F3—C1—F2'	64.7 (7)
C13—C14—H14	120.5	F2—C1—F4'	69.8 (8)
C9—C14—H14	120.5	F4—C1—F4'	39.0 (8)
C6—C7—C2	121.0 (2)	F3—C1—F4'	132.1 (10)
C6—C7—H7	119.5	F2'—C1—F4'	99.8 (8)
C2—C7—H7	119.5	F2—C1—F3'	131.1 (8)
F1—C5—C4	118.7 (2)	F4—C1—F3'	63.7 (9)
F1—C5—C6	117.7 (2)	F3—C1—F3'	47.4 (8)
C4—C5—C6	123.5 (2)	F2'—C1—F3'	102.0 (8)
C14—C13—C12	122.7 (2)	F4'—C1—F3'	99.6 (8)
C14—C13—N1	118.3 (2)	F2—C1—C2	113.42 (19)
C12—C13—N1	118.9 (2)	F4—C1—C2	113.36 (18)
C3—C2—C7	120.0 (2)	F3—C1—C2	112.74 (19)
C3—C2—C1	120.8 (2)	F2'—C1—C2	124.4 (9)
C7—C2—C1	119.3 (2)	F4'—C1—C2	112.9 (9)
C14—C9—C10	118.70 (19)	F3'—C1—C2	114.6 (9)
C14—C9—C8	119.6 (2)	C11—C10—C9	120.9 (2)
C10—C9—C8	121.7 (2)	C11—C10—H10	119.6
C12—C11—C10	120.4 (2)	C9—C10—H10	119.6
C12—C11—H11	119.8	C5—C4—C3	118.6 (2)
C10—C11—H11	119.8	C5—C4—H4	120.7
C11—C12—C13	118.3 (2)	C3—C4—H4	120.7
C6—N2—C8—C9	179.72 (18)	C14—C13—C12—C11	1.0 (4)
C8—N2—C6—C5	−147.5 (2)	N1—C13—C12—C11	179.1 (2)
C8—N2—C6—C7	35.2 (3)	C7—C2—C3—C4	−0.9 (4)
C5—C6—C7—C2	1.8 (3)	C1—C2—C3—C4	179.3 (2)
N2—C6—C7—C2	179.17 (19)	C3—C2—C1—F2	−26.3 (3)
C7—C6—C5—F1	177.73 (19)	C7—C2—C1—F2	153.9 (2)
N2—C6—C5—F1	0.2 (3)	C3—C2—C1—F4	−145.8 (3)
C7—C6—C5—C4	−3.4 (3)	C7—C2—C1—F4	34.4 (3)
N2—C6—C5—C4	179.1 (2)	C3—C2—C1—F3	91.7 (3)
C9—C14—C13—C12	−0.9 (4)	C7—C2—C1—F3	−88.2 (3)
C9—C14—C13—N1	−179.0 (2)	C3—C2—C1—F2'	17.6 (10)
O1—N1—C13—C14	−11.1 (4)	C7—C2—C1—F2'	−162.2 (10)
O2—N1—C13—C14	165.8 (2)	C3—C2—C1—F4'	−103.2 (10)
O1—N1—C13—C12	170.7 (3)	C7—C2—C1—F4'	77.0 (10)
O2—N1—C13—C12	−12.4 (4)	C3—C2—C1—F3'	143.7 (10)
C6—C7—C2—C3	0.2 (3)	C7—C2—C1—F3'	−36.1 (11)

C6—C7—C2—C1	179.99 (18)	C12—C11—C10—C9	0.2 (3)
C13—C14—C9—C10	0.5 (3)	C14—C9—C10—C11	−0.1 (3)
C13—C14—C9—C8	178.6 (2)	C8—C9—C10—C11	−178.3 (2)
N2—C8—C9—C14	167.7 (2)	F1—C5—C4—C3	−178.4 (2)
N2—C8—C9—C10	−14.2 (3)	C6—C5—C4—C3	2.7 (4)
C10—C11—C12—C13	−0.6 (3)	C2—C3—C4—C5	−0.5 (4)

Fig. 1


F1
C11
C10
C5
N2
C4
C12
C6
C9
C8
C3
C13
C7
C14
C2
F4'
C1
F2
N1
O2
F4
F2'
O1
F3'
F3