organic compounds

4-Fluoro-*N*-[(*E*)-3,4,5-trimethoxybenzylidene]aniline

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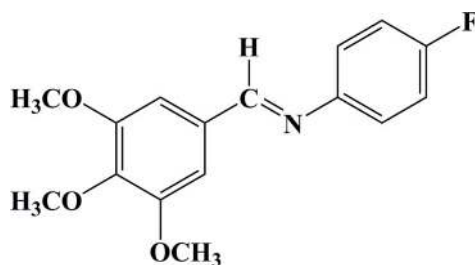Correspondence e-mail: jpodder@phy.buet.ac.bd

The title compound, $C_{16}H_{16}FNO_3$, exists in a *trans* configuration with respect to the C=N bond [1.258 (2) Å]. The central methoxy O atom deviates from the plane of the attached benzene ring by 0.0911 (14) Å. The dihedral angle between the aromatic rings is 47.58 (11)°. The crystal structure features C—H···N and C—H···O interactions.

Related literature

For the uses and biological importance of Schiff base compounds, see: Xia *et al.* (2009); Shah *et al.* (1992); Ünver *et al.* (2004). For related structures, see: Fun *et al.* (2011); Khalaji & Simpson (2009); Balachandar *et al.* (2013).

Experimental

Crystal data

$C_{16}H_{16}FNO_3$	c = 12.9217 (13) Å
M_r = 289.30	β = 105.266 (5)°
Monoclinic, $P2_1$	V = 743.59 (14) Å^3
a = 7.1147 (9) Å	Z = 2
b = 8.3841 (9) Å	Mo $K\alpha$ radiation

μ = 0.10 mm^{-1}	0.40 × 0.35 × 0.30 mm
T = 296 K	

Data collection

Bruker Kappa APEXII CCD diffractometer	5699 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1999) T_{min} = 0.962, T_{max} = 0.971	3358 independent reflections 2468 reflections with $I > 2\sigma(I)$ R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040	1 restraint
$wR(F^2)$ = 0.128	H-atom parameters constrained
S = 1.00	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
3358 reflections	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
190 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···N1^i	0.93	2.57	3.492 (3)	174
C7—H7···O3^{ii}	0.93	2.58	3.504 (3)	173

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+2$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2233).

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supporting information

4-Fluoro-*N*-[(*E*)-3,4,5-trimethoxybenzylidene]aniline

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S1. Comment

Schiff bases are among the most useful ligands in coordination chemistry as they readily form stable complexes with most transition metals (Xia *et al.*, 2009). They are known to exibit potent anti-bacterial, anti-convulsant, anti-inflammatory and anti-cancer activities (Shah *et al.*, 1992). In addition to that, it shows the Non-linear optical properties (Ünver *et al.*, 2004). Therefore, successful application of Schiff bases requires a careful study of their characteristics.

The title compound, $C_{16}H_{16}FNO_3$, exists in a *trans* configuration with respect to the C═N bond [1.258 (2) Å]. The N1═C8 bond length of 1.258 (2) Å is shorter than the N–C bond [1.411 (3) Å], indicating a typical imine double bond. Moreover, the C–N–C angle is 118.93 (17) °. X-ray analysis confirms the molecular structure and atom connectivity as illustrated in Fig. 1.

The dihedral angle formed between the phenyl rings (C1–C6) and (C8–C13) is 47.58 (11) °. The flurine atom F1 is deviated from the phenyl ring (C1–C6) by -0.0243 (23) Å. One of the oxygen atom attached to the phenyl ring (C8–C13) is deviated from the same plane by -0.0911 (14) Å.

The crystal packing is stabilized by C4—H4···N1[i] and C7—H7···O3[ii] inter-molecular interactions. The symmetry codes: (i). 1 - *x*,1/2 + *y*,1 - *z*. (ii). 1 - *x*,1/2 + *y*,2 - *z*.

S2. Experimental

The title compound was prepared by the condensation reaction between of 3,4,5-trimethoxybenzaldehyde (1 mmol, 0.196 g) and 4-Fluoroaniline (1 mmol, 0.172 g), which were taken in equimolar ratio and dissolved in methanol (20 ml). The resulting mixture was stirred at room temperature for overnight. Then filtering, drying the synthesized compound dissolved in a 20 ml of methanol to purify the title material by recrystallization process at least for three times. After 4 days, colourless single crystals were obtained using methanol as solvent by keeping the solution for slow evaporation suitable for single-crystal X-ray structure analysis.

S3. Refinement

The positions of hydrogen atoms were localized from the difference electron density maps and their distances were geometrically constrained. The H atoms bound to the C atoms were treated as riding atoms, with d(C–H) = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl atoms. d(C–H) = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl group. The rotation angles for methyl group is optimized by least squares. During the diffraction experiment, 998 Friedel pairs were merged.
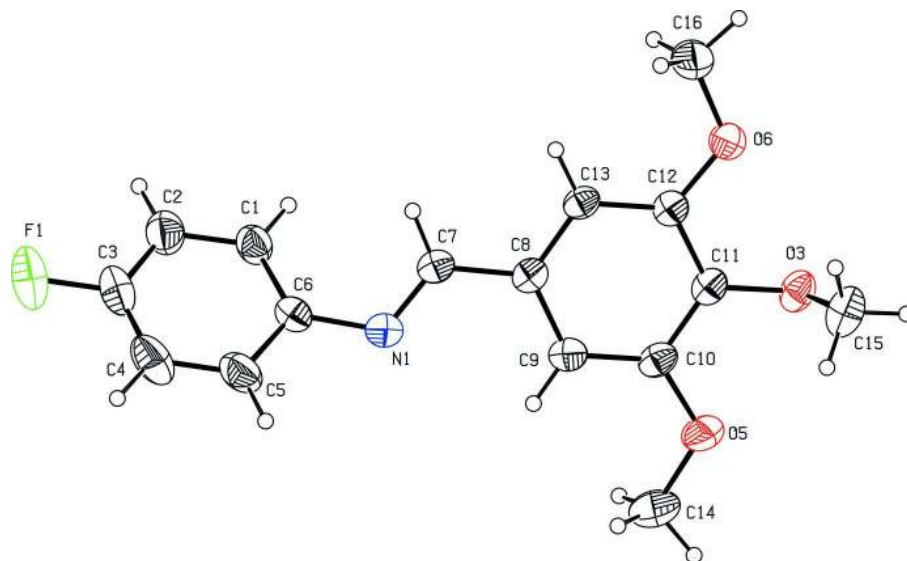
Figure 1

The molecular structure of the title compound with the atom numbering scheme, displacement ellipaoids are drawn at 30% probability level. H atoms are present as small spheres of arbitary radius.
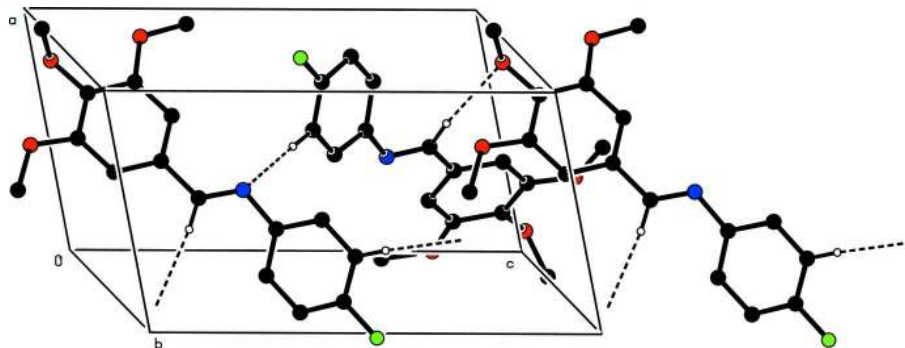
Figure 2

The crystal packing of the title compound, viewed down *a*-axis, showing C4—H4···N1[i] and C7—H7···O3[ii] inter-molecular interactions. The H atoms not involved in the bonding have been excluded for clarity.

4-Fluoro-*N*-[(*E*)-3,4,5-trimethoxybenzylidene]aniline

Crystal data

$C_{16}H_{16}FNO_3$	$F(000) = 304$
$M_r = 289.30$	$D_x = 1.292$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 2468 reflections
$a = 7.1147$ (9) Å	$\theta = 2.9$–28.3°
$b = 8.3841$ (9) Å	$\mu = 0.10$ mm^{-1}
$c = 12.9217$ (13) Å	$T = 296$ K
$\beta = 105.266$ (5)°	Block, colourless
$V = 743.59$ (14) Å^3	$0.40 \times 0.35 \times 0.30$ mm
$Z = 2$	

Data collection

Bruker Kappa APEXII CCD diffractometer	5699 measured reflections
Radiation source: fine-focus sealed tube	3358 independent reflections
Graphite monochromator	2468 reflections with $I > 2\sigma(I)$
ω and φ scan	$R_{int} = 0.017$
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	$\theta_{max} = 28.3°$, $\theta_{min} = 2.9°$
$T_{min} = 0.962$, $T_{max} = 0.971$	$h = -6 \rightarrow 9$
	$k = -11 \rightarrow 11$
	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.128$	
$S = 1.00$	H-atom parameters constrained
3358 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0767P)^2 + 0.017P]$ where $P = (F_o^2 + 2F_c^2)/3$
190 parameters	
1 restraint	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.14$ e Å^{-3}
	$\Delta\rho_{min} = -0.13$ e Å^{-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 $F^2 > \sigma(F^2)$ 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	0.9942 (4)	0.5751 (3)	0.51013 (17)	0.1191 (7)
N1	0.4847 (3)	0.2770 (2)	0.70169 (14)	0.0557 (4)
O3	0.0659 (2)	−0.05790 (17)	1.02505 (12)	0.0596 (4)
C4	0.6764 (5)	0.5185 (4)	0.5155 (2)	0.0901 (9)
H4	0.6295	0.5810	0.4548	0.108*
O5	−0.0273 (2)	−0.0800 (2)	0.81378 (13)	0.0745 (5)
O6	0.3531 (2)	0.12728 (18)	1.13224 (11)	0.0608 (4)
C3	0.8696 (5)	0.5003 (3)	0.5578 (2)	0.0763 (7)
C2	0.9455 (4)	0.4108 (4)	0.6458 (2)	0.0737 (7)
H2	1.0795	0.3987	0.6724	0.088*
C6	0.6199 (3)	0.3532 (3)	0.65551 (15)	0.0530 (5)
C7	0.5225 (3)	0.2666 (2)	0.80220 (16)	0.0474 (4)
H7	0.6363	0.3137	0.8430	0.057*
C8	0.3977 (3)	0.1847 (2)	0.85798 (15)	0.0453 (4)
C13	0.4394 (3)	0.1984 (2)	0.96847 (15)	0.0465 (4)
H13	0.5447	0.2598	1.0053	0.056*

C11	0.1708 (3)	0.0279 (2)	0.96978 (16)	0.0482 (4)
C1	0.8184 (3)	0.3381 (3)	0.69503 (18)	0.0605 (6)
H1	0.8679	0.2774	0.7564	0.073*
C12	0.3258 (3)	0.1214 (2)	1.02398 (15)	0.0468 (4)
C15	−0.0943 (4)	0.0258 (3)	1.0437 (2)	0.0794 (7)
H15A	−0.1612	−0.0411	1.0827	0.119*
H15B	−0.1819	0.0552	0.9763	0.119*
H15C	−0.0488	0.1201	1.0847	0.119*
C5	0.5502 (4)	0.4435 (4)	0.56337 (18)	0.0777 (7)
H5	0.4166	0.4532	0.5338	0.093*
C9	0.2415 (3)	0.0927 (2)	0.80236 (17)	0.0513 (5)
H9	0.2132	0.0838	0.7281	0.062*
C10	0.1283 (3)	0.0142 (2)	0.85863 (16)	0.0513 (5)
C14	−0.0696 (4)	−0.1114 (4)	0.7021 (2)	0.0955 (10)
H14A	−0.1824	−0.1788	0.6812	0.143*
H14B	0.0396	−0.1639	0.6864	0.143*
H14C	−0.0948	−0.0128	0.6632	0.143*
C16	0.5085 (4)	0.2205 (4)	1.1925 (2)	0.0863 (8)
H16A	0.5116	0.2145	1.2671	0.129*
H16B	0.4905	0.3294	1.1691	0.129*
H16C	0.6291	0.1811	1.1824	0.129*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.1440 (16)	0.1259 (17)	0.1116 (12)	−0.0088 (14)	0.0766 (12)	0.0330 (12)
N1	0.0526 (10)	0.0608 (10)	0.0501 (9)	−0.0022 (8)	0.0073 (7)	−0.0009 (8)
O3	0.0550 (8)	0.0458 (8)	0.0784 (10)	−0.0049 (6)	0.0179 (7)	0.0129 (7)
C4	0.115 (3)	0.096 (2)	0.0619 (14)	0.0216 (18)	0.0283 (15)	0.0319 (15)
O5	0.0697 (10)	0.0754 (11)	0.0697 (9)	−0.0290 (9)	0.0030 (8)	−0.0048 (9)
O6	0.0667 (9)	0.0585 (9)	0.0548 (8)	−0.0136 (7)	0.0117 (7)	0.0029 (7)
C3	0.103 (2)	0.0727 (16)	0.0654 (15)	0.0007 (14)	0.0434 (15)	0.0111 (12)
C2	0.0710 (15)	0.0847 (18)	0.0699 (14)	0.0018 (13)	0.0266 (12)	0.0071 (13)
C6	0.0614 (13)	0.0531 (11)	0.0437 (10)	0.0027 (9)	0.0125 (9)	−0.0014 (9)
C7	0.0448 (10)	0.0406 (10)	0.0540 (11)	0.0010 (8)	0.0077 (8)	−0.0028 (8)
C8	0.0416 (9)	0.0364 (9)	0.0562 (11)	0.0053 (7)	0.0097 (8)	0.0019 (8)
C13	0.0425 (9)	0.0379 (9)	0.0558 (11)	−0.0014 (7)	0.0071 (8)	−0.0019 (8)
C11	0.0445 (10)	0.0345 (9)	0.0638 (11)	0.0031 (8)	0.0111 (9)	0.0074 (8)
C1	0.0630 (14)	0.0647 (13)	0.0543 (12)	0.0057 (10)	0.0164 (10)	0.0139 (10)
C12	0.0481 (10)	0.0363 (9)	0.0546 (11)	0.0033 (8)	0.0110 (8)	0.0039 (8)
C15	0.0658 (15)	0.0681 (16)	0.113 (2)	−0.0018 (12)	0.0385 (14)	0.0099 (14)
C5	0.0791 (15)	0.099 (2)	0.0506 (12)	0.0123 (15)	0.0093 (11)	0.0191 (13)
C9	0.0511 (10)	0.0456 (10)	0.0522 (10)	0.0007 (8)	0.0047 (8)	−0.0010 (8)
C10	0.0454 (10)	0.0378 (10)	0.0650 (12)	−0.0016 (8)	0.0049 (9)	0.0012 (9)
C14	0.0864 (19)	0.102 (2)	0.0858 (18)	−0.0330 (17)	0.0016 (16)	−0.0256 (17)
C16	0.097 (2)	0.097 (2)	0.0620 (14)	−0.0355 (17)	0.0170 (14)	−0.0132 (14)

Geometric parameters (Å, °)

F1—C3	1.359 (3)	C8—C13	1.384 (3)
N1—C7	1.258 (2)	C8—C9	1.388 (3)
N1—C6	1.411 (3)	C13—C12	1.375 (3)
O3—C11	1.366 (2)	C13—H13	0.9300
O3—C15	1.412 (3)	C11—C12	1.384 (2)
C4—C3	1.347 (4)	C11—C10	1.392 (3)
C4—C5	1.370 (4)	C1—H1	0.9300
C4—H4	0.9300	C15—H15A	0.9600
O5—C10	1.359 (2)	C15—H15B	0.9600
O5—C14	1.419 (3)	C15—H15C	0.9600
O6—C12	1.362 (2)	C5—H5	0.9300
O6—C16	1.409 (3)	C9—C10	1.386 (3)
C3—C2	1.351 (4)	C9—H9	0.9300
C2—C1	1.378 (3)	C14—H14A	0.9600
C2—H2	0.9300	C14—H14B	0.9600
C6—C1	1.375 (3)	C14—H14C	0.9600
C6—C5	1.388 (3)	C16—H16A	0.9600
C7—C8	1.455 (3)	C16—H16B	0.9600
C7—H7	0.9300	C16—H16C	0.9600
C7—N1—C6	118.93 (17)	O6—C12—C13	125.09 (17)
C11—O3—C15	113.85 (16)	O6—C12—C11	114.78 (16)
C3—C4—C5	119.0 (2)	C13—C12—C11	120.13 (17)
C3—C4—H4	120.5	O3—C15—H15A	109.5
C5—C4—H4	120.5	O3—C15—H15B	109.5
C10—O5—C14	118.18 (19)	H15A—C15—H15B	109.5
C12—O6—C16	117.81 (17)	O3—C15—H15C	109.5
C4—C3—C2	122.9 (3)	H15A—C15—H15C	109.5
C4—C3—F1	118.8 (2)	H15B—C15—H15C	109.5
C2—C3—F1	118.3 (3)	C4—C5—C6	120.6 (3)
C3—C2—C1	117.9 (2)	C4—C5—H5	119.7
C3—C2—H2	121.0	C6—C5—H5	119.7
C1—C2—H2	121.0	C10—C9—C8	119.28 (18)
C1—C6—C5	117.9 (2)	C10—C9—H9	120.4
C1—C6—N1	123.28 (19)	C8—C9—H9	120.4
C5—C6—N1	118.7 (2)	O5—C10—C9	124.98 (18)
N1—C7—C8	123.50 (17)	O5—C10—C11	114.75 (18)
N1—C7—H7	118.3	C9—C10—C11	120.27 (17)
C8—C7—H7	118.3	O5—C14—H14A	109.5
C13—C8—C9	120.36 (17)	O5—C14—H14B	109.5
C13—C8—C7	118.60 (16)	H14A—C14—H14B	109.5
C9—C8—C7	121.03 (17)	O5—C14—H14C	109.5
C12—C13—C8	120.21 (16)	H14A—C14—H14C	109.5
C12—C13—H13	119.9	H14B—C14—H14C	109.5
C8—C13—H13	119.9	O6—C16—H16A	109.5
O3—C11—C12	120.42 (17)	O6—C16—H16B	109.5

O3—C11—C10	119.75 (17)	H16A—C16—H16B	109.5
C12—C11—C10	119.73 (17)	O6—C16—H16C	109.5
C6—C1—C2	121.5 (2)	H16A—C16—H16C	109.5
C6—C1—H1	119.2	H16B—C16—H16C	109.5
C2—C1—H1	119.2		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···N1[i]	0.93	2.57	3.492 (3)	174
C7—H7···O3[ii]	0.93	2.58	3.504 (3)	173

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