N,N′-Bis(4-chloro-3-fluorobenzyl-idene)ethane-1,2-diamine

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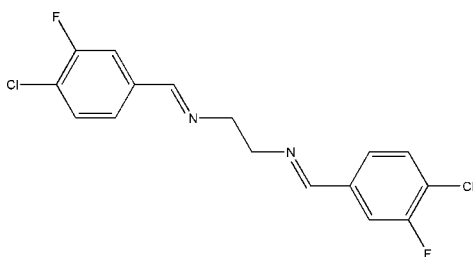
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The asymmetric unit of the title Schiff base compound, $C_{16}H_{12}Cl_2F_2N_2$, contains one half of the centrosymmetric molecule. Molecules related by translation along the a axis form stacks with short intermolecular C···C distances of 3.429 (3) Å. The crystal packing also exhibits short intermolecular Cl···F contacts of 3.087 (1) Å.

Related literature

For a related structure, see Fun & Kia (2008). For general background, see: Pal *et al.* (2005); Calligaris & Randaccio (1987); Hou *et al.* (2001); Ren *et al.* (2002); Allen *et al.* (1987).

Experimental

Crystal data

$C_{16}H_{12}Cl_2F_2N_2$	$V = 720.12$ (3) Å^3
$M_r = 341.18$	$Z = 2$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 4.6542$ (1) Å	$\mu = 0.47$ mm^{-1}
$b = 23.1343$ (6) Å	$T = 100.0$ (1) K
$c = 6.9961$ (2) Å	$0.51 \times 0.05 \times 0.04$ mm
$\beta = 107.063$ (2)°	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	17372 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	2139 independent reflections
	1705 reflections with $I > 2\sigma(I)$
$T_{min} = 0.795$, $T_{max} = 0.983$	$R_{int} = 0.054$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	100 parameters
$wR(F^2) = 0.113$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{max} = 0.99$ e Å^{-3}
2139 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}

Table 1
Selected interatomic distances (Å).

Cl1···F1^i	3.087 (1)	C3···C6^{ii}	3.429 (3)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Bruker (2005). *APEX2, SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Calligaris, M. & Randaccio, L. (1987). *Comprehensive Coordination Chemistry*, Vol. 2, edited by G. Wilkinson, pp. 715–738. London: Pergamon.
Fun, H.-K. & Kia, R. (2008). *Acta Cryst.* E**64**, o1722–o1723.
Hou, B., Friedman, N., Ruhman, S., Sheves, M. & Ottolenghi, M. (2001). *J. Phys. Chem. B*, **105**, 7042–7048.
Pal, S., Barik, A. K., Gupta, S., Hazra, A., Kar, S. K., Peng, S.-M., Lee, G.-H., Butcher, R. J., El Fallah, M. S. & Ribas, J. (2005). *Inorg. Chem.* **44**, 3880–3889.
Ren, S., Wang, R., Komatsu, K., Bonaz-Krause, P., Zyrianov, Y., McKenna, C. E., Csipke, C., Tokes, Z. A. & Lien, E. J. (2002). *J. Med. Chem.* **45**, 410–419.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

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S1. Comment

Schiff bases are one of most prevalent mixed-donor ligands in the field of coordination chemistry. There has been growing interest in Schiff base ligands, mainly because of their wide application in the field of biochemistry, synthesis and catalysis (Pal *et al.*, 2005; Hou *et al.*, 2001; Ren *et al.*, 2002). Many Schiff base complexes have been structurally characterized, but only a relatively small number of free Schiff bases have been characterized (Calligaris & Randaccio, 1987). As an extension of our work (Fun & Kia, 2008) on the structural characterization of Schiff base ligands, the title compound (I) is reported here.

The molecule of the title compound, (I) (Fig. 1), lies across a crystallographic inversion centre and adopts an *E* configuration with respect to the azomethine C=N bond. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and are comparable with those in the related structure (Fun & Kia, 2008). The planar units are parallel by symmetry but extend in opposite directions from the dimethylene bridge. The interesting feature of the crystal structure is the short intermolecular Cl···F interaction (Table 1) with the distance of 3.087 (1) Å, which is shorter than the sum of the van der Waals radii of these atoms. The molecules related by translation along the *a* axis form stacks with short intermolecular C···C distances of 3.429 (3) Å (Table 1).

S2. Experimental

The synthetic method has been described earlier (Fun & Kia, 2008). Single crystals suitable for *X*-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

S3. Refinement

All of the hydrogen atoms were positioned geometrically with C—H = 0.93 or 0.97 Å and refined in riding mode with U_{iso} (H) = 1.2 U_{eq} (C). The highest residual peak of 0.99 e. Å^{-3} is located 0.25 Å from atom H4A.
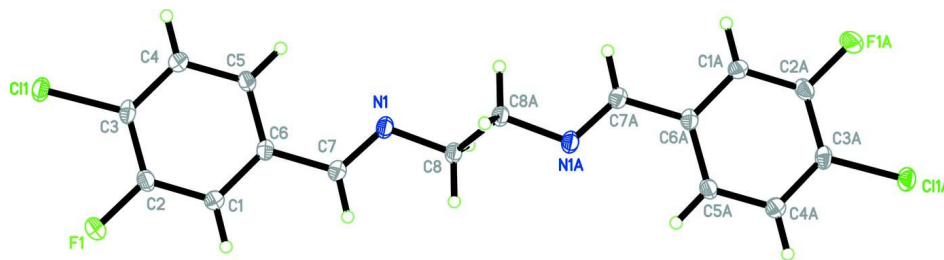

Figure 1

The molecular structure of (I) with atom labels and 50% probability displacement ellipsoids [symmetry code: (A) -*x*, -*y*, -*z* + 1].

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Crystal data

$C_{16}H_{12}Cl_2F_2N_2$	$F(000) = 348$
$M_r = 341.18$	$D_x = 1.573$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3479 reflections
$a = 4.6542$ (1) Å	$\theta = 3.2–30.0°$
$b = 23.1343$ (6) Å	$\mu = 0.47$ mm^{-1}
$c = 6.9961$ (2) Å	$T = 100$ K
$\beta = 107.063$ (2)°	Needle, colourless
$V = 720.12$ (3) Å^3	$0.51 \times 0.05 \times 0.04$ mm
$Z = 2$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	17372 measured reflections
Radiation source: fine-focus sealed tube	2139 independent reflections
Graphite monochromator	1705 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.054$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$\theta_{max} = 30.2°$, $\theta_{min} = 1.8°$
	$h = -6\rightarrow6$
	$k = -32\rightarrow32$
$T_{min} = 0.795$, $T_{max} = 0.983$	$l = -9\rightarrow9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	
$wR(F^2) = 0.113$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.6198P]$ where $P = (F_o^2 + 2F_c^2)/3$
2139 reflections	
100 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 0.99$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Experimental. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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	$U_{iso}*/U_{eq}$
Cl1	0.77503 (10)	0.17566 (2)	−0.30608 (7)	0.01931 (14)
F1	0.6479 (3)	0.24323 (5)	0.0088 (2)	0.0285 (3)
N1	0.0845 (4)	0.04180 (7)	0.2962 (2)	0.0165 (3)

C1	0.4139 (4)	0.17032 (8)	0.1460 (3)	0.0170 (4)
H1A	0.3865	0.1959	0.2417	0.020*
C2	0.5544 (4)	0.18819 (8)	0.0072 (3)	0.0178 (4)
C3	0.6002 (4)	0.15091 (8)	−0.1355 (3)	0.0162 (4)
C4	0.5045 (4)	0.09402 (8)	−0.1403 (3)	0.0166 (4)
H4A	0.5381	0.0684	−0.2340	0.020*
C5	0.3581 (4)	0.07550 (8)	−0.0045 (3)	0.0158 (4)
H5A	0.2890	0.0376	−0.0100	0.019*
C6	0.3139 (4)	0.11314 (8)	0.1400 (3)	0.0146 (3)
C7	0.1624 (4)	0.09406 (8)	0.2865 (3)	0.0153 (4)
H7A	0.1227	0.1210	0.3742	0.018*
C8	−0.0692 (4)	0.02721 (8)	0.4438 (3)	0.0154 (4)
H8A	−0.2811	0.0208	0.3775	0.019*
H8B	−0.0510	0.0590	0.5372	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0208 (2)	0.0198 (2)	0.0212 (2)	−0.00002 (17)	0.01230 (17)	0.00367 (18)
F1	0.0397 (7)	0.0162 (6)	0.0378 (8)	−0.0075 (5)	0.0242 (6)	−0.0041 (5)
N1	0.0177 (7)	0.0177 (8)	0.0162 (8)	−0.0006 (6)	0.0082 (6)	0.0024 (6)
C1	0.0184 (8)	0.0155 (9)	0.0192 (9)	−0.0001 (7)	0.0088 (7)	−0.0015 (7)
C2	0.0175 (8)	0.0138 (8)	0.0241 (10)	−0.0015 (6)	0.0093 (7)	0.0016 (7)
C3	0.0135 (8)	0.0185 (9)	0.0185 (9)	0.0003 (7)	0.0077 (7)	0.0048 (7)
C4	0.0168 (8)	0.0165 (9)	0.0171 (9)	0.0003 (7)	0.0061 (7)	−0.0004 (7)
C5	0.0173 (8)	0.0131 (8)	0.0185 (9)	−0.0008 (6)	0.0073 (7)	0.0017 (7)
C6	0.0139 (7)	0.0152 (8)	0.0155 (8)	0.0006 (6)	0.0057 (6)	0.0027 (7)
C7	0.0147 (8)	0.0169 (9)	0.0152 (9)	0.0003 (6)	0.0057 (7)	0.0012 (7)
C8	0.0164 (8)	0.0155 (8)	0.0165 (9)	0.0003 (6)	0.0081 (7)	0.0020 (7)

Geometric parameters (Å, º)

Cl1—C3	1.7274 (19)	C4—C5	1.389 (3)
F1—C2	1.345 (2)	C4—H4A	0.9300
N1—C7	1.270 (2)	C5—C6	1.395 (3)
N1—C8	1.458 (2)	C5—H5A	0.9300
C1—C2	1.383 (3)	C6—C7	1.472 (3)
C1—C6	1.399 (3)	C7—H7A	0.9300
C1—H1A	0.9300	C8—C8[i]	1.524 (4)
C2—C3	1.383 (3)	C8—H8A	0.9700
C3—C4	1.387 (3)	C8—H8B	0.9700
Cl1···F1[ii]	3.087 (1)	C3···C6[iii]	3.429 (3)
C7—N1—C8	117.74 (17)	C4—C5—H5A	119.7
C2—C1—C6	118.90 (18)	C6—C5—H5A	119.7
C2—C1—H1A	120.5	C5—C6—C1	119.56 (17)
C6—C1—H1A	120.5	C5—C6—C7	121.36 (16)

F1—C2—C3	118.59 (17)	C1—C6—C7	119.08 (17)
F1—C2—C1	119.72 (17)	N1—C7—C6	121.70 (18)
C3—C2—C1	121.70 (17)	N1—C7—H7A	119.2
C2—C3—C4	119.55 (17)	C6—C7—H7A	119.2
C2—C3—Cl1	119.73 (14)	N1—C8—C8ⁱ	109.64 (18)
C4—C3—Cl1	120.72 (15)	N1—C8—H8A	109.7
C3—C4—C5	119.63 (18)	C8ⁱ—C8—H8A	109.7
C3—C4—H4A	120.2	N1—C8—H8B	109.7
C5—C4—H4A	120.2	C8ⁱ—C8—H8B	109.7
C4—C5—C6	120.65 (17)	H8A—C8—H8B	108.2
C6—C1—C2—F1	178.99 (16)	C4—C5—C6—C1	1.0 (3)
C6—C1—C2—C3	−0.5 (3)	C4—C5—C6—C7	−179.29 (16)
F1—C2—C3—C4	−179.76 (16)	C2—C1—C6—C5	0.2 (3)
C1—C2—C3—C4	−0.2 (3)	C2—C1—C6—C7	−179.57 (16)
F1—C2—C3—Cl1	0.1 (2)	C8—N1—C7—C6	−178.90 (15)
C1—C2—C3—Cl1	179.65 (14)	C5—C6—C7—N1	4.9 (3)
C2—C3—C4—C5	1.4 (3)	C1—C6—C7—N1	−175.37 (17)
Cl1—C3—C4—C5	−178.50 (14)	C7—N1—C8—C8ⁱ	−133.5 (2)
C3—C4—C5—C6	−1.7 (3)		

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