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(*E,E*)-*N*¹,*N*²-Bis(2,6-difluorobenzylidene)ethane-1,2-diamine

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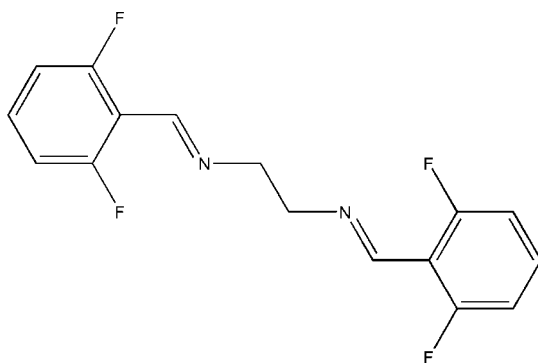
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.124; data-to-parameter ratio = 12.0.

The asymmetric unit of the title compound, $\text{C}_{16}\text{H}_{12}\text{F}_4\text{N}_2$, comprises half of the potentially bidentate Schiff base ligand, with an inversion centre located at the mid-point of the central C—C bond. The crystal packing is stabilized by intermolecular C—H \cdots N and π – π interactions [centroid–centroid distance = 3.6793 (12) Å and interplanar spacing = 3.4999 (7) Å].

Related literature

For background to the synthesis and structural variations of Schiff base ligands and their complexes, see: Granovski *et al.* (1993); Elmali *et al.* (2000).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{F}_4\text{N}_2$
 $M_r = 308.28$
Monoclinic, $P2_1/n$
 $a = 7.3304$ (10) Å
 $b = 10.5414$ (15) Å
 $c = 9.2106$ (13) Å
 $\beta = 105.487$ (2)°
 $V = 685.89$ (17) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 150$ K
 $0.34 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.958$, $T_{\max} = 0.987$
4573 measured reflections
1203 independent reflections
1057 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.124$
 $S = 1.15$
1203 reflections
100 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5}\cdots\text{N1}^i$	0.95	2.53	3.471 (2)	171

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

Data collection: SMART (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2335).

References

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Granovski, A. D., Nivorozhkin, A. L. & Minkin, V. I. (1993). *Coord. Chem. Rev.* **126**, 1–69.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2011). E67, o3107 [doi:10.1107/S1600536811044692]

(*E,E*)-*N*¹,*N*²-Bis(2,6-difluorobenzylidene)ethane-1,2-diamine

Mohammad Khaledi Sardashti, Reza Kia, William Clegg and Ross W. Harrington

S1. Comment

Schiff base ligands are among the most prevalent ligands in the field of coordination chemistry. Metal derivatives of Schiff bases have been studied extensively, and Ni^{II} and Cu^{II} complexes play a major role in both synthetic and structural research (Elmali *et al.*, 2000; Granovski *et al.*, 1993).

The asymmetric unit of the title compound comprises half of the potentially bidentate Schiff base ligand; an inversion centre is located in the middle of the central C—C bond (Fig. 1). Each half of the molecule is essentially planar, the imine segment (C1—N1=C2—C3) being rotated only 7.36 (10)° out of the plane of the benzene ring. The two halves are parallel by inversion symmetry, but not coplanar, the CH₂CH₂ linker unit forming a step between them.

The crystal packing is stabilized by intermolecular C—H⋯N interactions (see Table 1), which generate sheets parallel to (1 0 -1), and by intermolecular π - π interactions [centroid-centroid distance = 3.6793 (12) Å, interplanar separation = 3.4999 (7) Å, the two planes are strictly parallel by inversion symmetry].

S2. Experimental

The title compound was synthesized by mixing 2,4-difluorobenzaldehyde (4 mmol) and ethylenediamine (2 mmol) in chloroform (20 ml). After stirring for 2 h, the solution was filtered and the resulting yellow solid was crystallized from ethanol, giving single crystals suitable for X-ray diffraction.

S3. Refinement

All H atoms were positioned geometrically and constrained to ride on the parent atoms, with C—H = 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

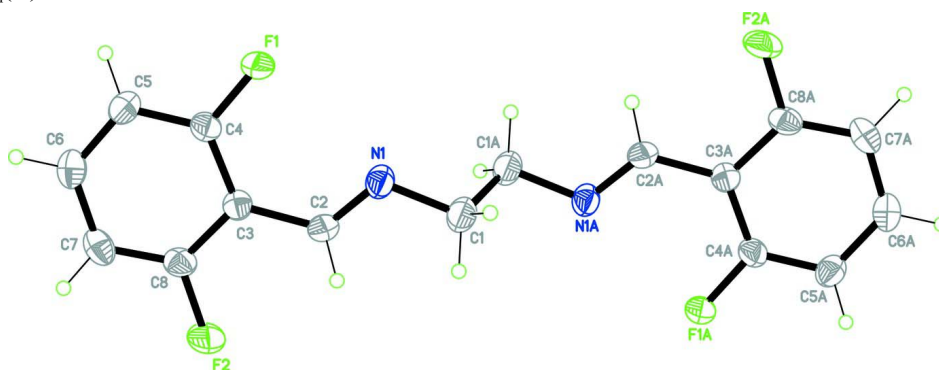
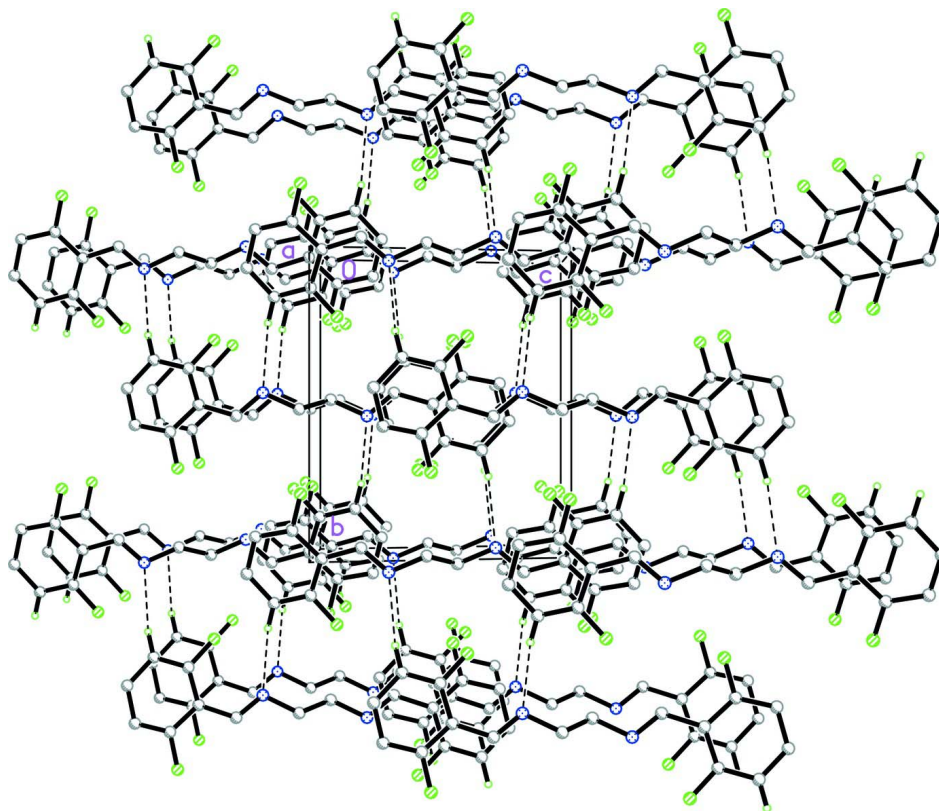


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Symmetry code for suffix A: $-x + 1, -y + 1, -z + 2$.

**Figure 2**

The crystal packing, viewed down the a axis, showing linking of molecules through the intermolecular C—H...N hydrogen bonds to form sheets in the (1 0 - 1) plane. The dashed lines represent these intermolecular interactions.

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Crystal data

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$M_r = 308.28$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.3304$ (10) Å

$b = 10.5414$ (15) Å

$c = 9.2106$ (13) Å

$\beta = 105.487$ (2)°

$V = 685.89$ (17) Å³

$Z = 2$

$F(000) = 316$

$D_x = 1.493$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4055 reflections

$\theta = 2.9$ – 28.3 °

$\mu = 0.13$ mm⁻¹

$T = 150$ K

Plate, yellow

$0.34 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART 1K CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.33 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.958$, $T_{\max} = 0.987$

4573 measured reflections

1203 independent reflections

1057 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 3.0$ °

$h = -8 \rightarrow 8$

$k = -12 \rightarrow 12$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.124$
 $S = 1.15$
 1203 reflections
 100 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.2701P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The low-temperature data were collected with the Oxford Cyrosystems Cryostream 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.24616 (17)	0.28505 (10)	0.59397 (12)	0.0409 (4)
F2	0.29841 (19)	0.71120 (10)	0.45850 (14)	0.0434 (4)
N1	0.4481 (2)	0.46153 (14)	0.79618 (16)	0.0297 (4)
C1	0.5503 (3)	0.51558 (19)	0.9411 (2)	0.0334 (5)
H1A	0.5590	0.6088	0.9317	0.040*
H1B	0.6805	0.4809	0.9717	0.040*
C2	0.3868 (2)	0.53796 (16)	0.6894 (2)	0.0277 (4)
H2	0.4123	0.6256	0.7087	0.033*
C3	0.2783 (2)	0.50109 (15)	0.53673 (18)	0.0244 (4)
C4	0.2091 (2)	0.37948 (16)	0.49141 (19)	0.0272 (4)
C5	0.1031 (2)	0.35046 (18)	0.3480 (2)	0.0311 (5)
H5	0.0594	0.2664	0.3225	0.037*
C6	0.0615 (3)	0.44634 (19)	0.2419 (2)	0.0332 (5)
H6	-0.0123	0.4279	0.1426	0.040*
C7	0.1257 (3)	0.56853 (19)	0.2780 (2)	0.0330 (5)
H7	0.0974	0.6345	0.2052	0.040*
C8	0.2317 (2)	0.59168 (16)	0.4228 (2)	0.0287 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0569 (8)	0.0217 (6)	0.0346 (6)	-0.0067 (5)	-0.0046 (5)	0.0054 (4)
F2	0.0677 (8)	0.0190 (6)	0.0419 (7)	0.0003 (5)	0.0118 (6)	0.0036 (5)

N1	0.0339 (8)	0.0273 (8)	0.0247 (8)	0.0032 (6)	0.0022 (6)	-0.0038 (6)
C1	0.0331 (10)	0.0355 (10)	0.0280 (10)	-0.0021 (8)	0.0020 (8)	-0.0042 (8)
C2	0.0331 (9)	0.0210 (9)	0.0287 (9)	-0.0031 (7)	0.0077 (7)	-0.0021 (7)
C3	0.0259 (8)	0.0230 (9)	0.0248 (9)	0.0035 (7)	0.0075 (7)	-0.0012 (7)
C4	0.0306 (9)	0.0228 (9)	0.0271 (9)	0.0030 (7)	0.0059 (7)	0.0027 (7)
C5	0.0312 (9)	0.0292 (10)	0.0308 (10)	-0.0017 (7)	0.0044 (8)	-0.0059 (7)
C6	0.0300 (9)	0.0421 (11)	0.0251 (9)	0.0044 (8)	0.0032 (7)	-0.0014 (8)
C7	0.0370 (10)	0.0349 (10)	0.0278 (10)	0.0101 (8)	0.0098 (8)	0.0082 (8)
C8	0.0357 (10)	0.0198 (9)	0.0324 (10)	0.0042 (7)	0.0124 (8)	0.0007 (7)

Geometric parameters (Å, °)

F1—C4	1.349 (2)	C3—C8	1.392 (2)
F2—C8	1.360 (2)	C3—C4	1.401 (3)
N1—C2	1.258 (2)	C4—C5	1.376 (3)
N1—C1	1.461 (2)	C5—C6	1.382 (3)
C1—C1 ⁱ	1.502 (4)	C5—H5	0.950
C1—H1A	0.990	C6—C7	1.381 (3)
C1—H1B	0.990	C6—H6	0.950
C2—C3	1.471 (2)	C7—C8	1.374 (3)
C2—H2	0.950	C7—H7	0.950
C2—N1—C1	116.98 (16)	F1—C4—C3	118.52 (15)
N1—C1—C1 ⁱ	110.12 (19)	C5—C4—C3	123.79 (16)
N1—C1—H1A	109.6	C4—C5—C6	118.53 (17)
C1 ⁱ —C1—H1A	109.6	C4—C5—H5	120.7
N1—C1—H1B	109.6	C6—C5—H5	120.7
C1 ⁱ —C1—H1B	109.6	C7—C6—C5	120.98 (17)
H1A—C1—H1B	108.1	C7—C6—H6	119.5
N1—C2—C3	124.56 (16)	C5—C6—H6	119.5
N1—C2—H2	117.7	C8—C7—C6	117.89 (18)
C3—C2—H2	117.7	C8—C7—H7	121.1
C8—C3—C4	114.00 (15)	C6—C7—H7	121.1
C8—C3—C2	120.03 (15)	F2—C8—C7	118.23 (17)
C4—C3—C2	125.95 (15)	F2—C8—C3	116.95 (16)
F1—C4—C5	117.69 (16)	C7—C8—C3	124.81 (17)
C2—N1—C1—C1 ⁱ	119.1 (2)	C3—C4—C5—C6	-0.1 (3)
C1—N1—C2—C3	-178.90 (15)	C4—C5—C6—C7	0.4 (3)
N1—C2—C3—C8	-174.14 (17)	C5—C6—C7—C8	-0.1 (3)
N1—C2—C3—C4	7.6 (3)	C6—C7—C8—F2	178.75 (15)
C8—C3—C4—F1	-179.76 (15)	C6—C7—C8—C3	-0.6 (3)
C2—C3—C4—F1	-1.4 (3)	C4—C3—C8—F2	-178.50 (14)
C8—C3—C4—C5	-0.5 (3)	C2—C3—C8—F2	3.0 (2)
C2—C3—C4—C5	177.93 (16)	C4—C3—C8—C7	0.8 (3)
F1—C4—C5—C6	179.17 (15)	C2—C3—C8—C7	-177.67 (16)

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C5—H5···N1 ⁱⁱ	0.95	2.53	3.471 (2)	171

Symmetry code: (ii) $x-1/2, -y+1/2, z-1/2$.