

**(*E,E*)-*N*<sup>1</sup>,*N*<sup>2</sup>-Bis(2,6-difluorobenzylidene)ethane-1,2-diamine**

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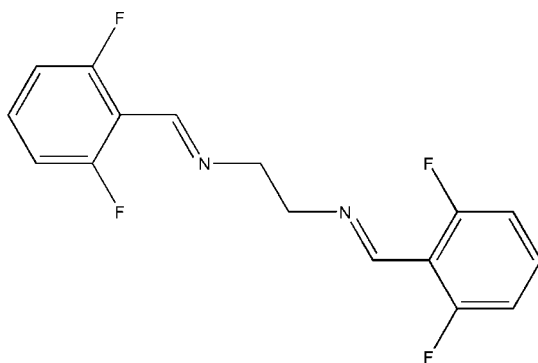
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Key indicators: single-crystal X-ray study; *T* = 150 K; mean  $\sigma(\text{C}-\text{C})$  = 0.003 Å; *R* factor = 0.043; *wR* factor = 0.124; data-to-parameter ratio = 12.0.

The asymmetric unit of the title compound, C<sub>16</sub>H<sub>12</sub>F<sub>4</sub>N<sub>2</sub>, 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···N and  $\pi$ – $\pi$  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*

C<sub>16</sub>H<sub>12</sub>F<sub>4</sub>N<sub>2</sub>  
*M<sub>r</sub>* = 308.28  
Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 7.3304 (10) Å  
*b* = 10.5414 (15) Å  
*c* = 9.2106 (13) Å  
 $\beta$  = 105.487 (2)°  
*V* = 685.89 (17) Å<sup>3</sup>  
*Z* = 2  
Mo *K*α radiation  
 $\mu$  = 0.13 mm<sup>-1</sup>  
*T* = 150 K  
0.34 × 0.30 × 0.10 mm

*Data collection*

Bruker SMART 1K CCD area-detector diffractometer  
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)  
*T*<sub>min</sub> = 0.958, *T*<sub>max</sub> = 0.987  
4573 measured reflections  
1203 independent reflections  
1057 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 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<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.23 e Å<sup>-3</sup>

**Table 1**

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 <sup>i</sup> | 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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**supplementary materials**

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

## (*E,E*)-*N*<sup>1</sup>,*N*<sup>2</sup>-Bis(2,6-difluorobenzylidene)ethane-1,2-diamine

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### 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<sup>II</sup> and Cu<sup>II</sup> 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)<sup>o</sup> out of the plane of the benzene ring. The two halves are parallel by inversion symmetry, but not coplanar, the CH<sub>2</sub>CH<sub>2</sub> 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  $\pi$ - $\pi$  interactions [centroid-centroid distance = 3.6793 (12) Å, interplanar separation = 3.4999 (7) Å, the two planes are strictly parallel by inversion symmetry].

### 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.

### Refinement

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

### Figures

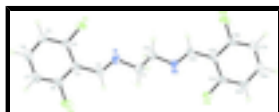


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Symmetry code for suffix A:  $-x + 1, -y + 1, -z + 2$ .

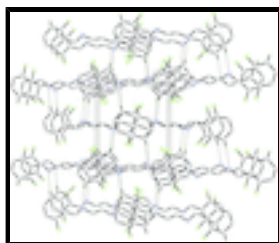


Fig. 2. The crystal packing, viewed down the *a* axis, showing linking of molecules through the intermolecular C—H...N hydrogen bonds to form sheets in the (1 0 -1) plane. The dashed lines represent these intermolecular interactions.

## (*E,E*)-*N*<sup>1</sup>,*N*<sup>2</sup>-Bis(2,6-difluorobenzylidene)ethane-1,2-diamine

### Crystal data

|                                 |   |
|---------------------------------|---|
| $C_{16}H_{12}F_4N_2$            | $F(000) = 316$  |
| $M_r = 308.28$                  | $D_x = 1.493 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1/n$            | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: $-P 2yn$           | Cell parameters from 4055 reflections                   |
| $a = 7.3304 (10) \text{ \AA}$   | $\theta = 2.9\text{--}28.3^\circ$                       |
| $b = 10.5414 (15) \text{ \AA}$  | $\mu = 0.13 \text{ mm}^{-1}$                            |
| $c = 9.2106 (13) \text{ \AA}$   | $T = 150 \text{ K}$                                     |
| $\beta = 105.487 (2)^\circ$     | Plate, yellow   |
| $V = 685.89 (17) \text{ \AA}^3$ | $0.34 \times 0.30 \times 0.10 \text{ mm}$               |
| $Z = 2$                         |   |

### Data collection

|  |  |
|--|--|
| Bruker SMART 1K CCD area-detector diffractometer         | 1203 independent reflections   |
| Radiation source: fine-focus sealed tube graphite        | 1057 reflections with $I > 2\sigma(I)$                                 |
| Detector resolution: $8.33 \text{ pixels mm}^{-1}$       | $R_{\text{int}} = 0.031$   |
| $\omega$ scans   | $\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 3.0^\circ$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | $h = -8 \rightarrow 8$   |
| $T_{\text{min}} = 0.958$ , $T_{\text{max}} = 0.987$      | $k = -12 \rightarrow 12$   |
| 4573 measured reflections                                | $l = -10 \rightarrow 10$   |

### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.124$               | H-atom parameters constrained                                  |
| $S = 1.15$                      | $w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.2701P]$              |
| 1203 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 100 parameters                  | $(\Delta/\sigma)_{\text{max}} < 0.001$                         |
| 0 restraints                    | $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$            |
|                                 | $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$           |

### 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 ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| 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 ( $\text{\AA}^2$ )*

|    | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|----|-------------|-------------|-------------|-------------|-------------|-------------|
| 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)  |

## supplementary materials

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### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|                          |              |             |              |
|--------------------------|--------------|-------------|--------------|
| 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 <sup>i</sup>       | 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 <sup>i</sup>    | 110.12 (19)  | C5—C4—C3    | 123.79 (16)  |
| N1—C1—H1A                | 109.6        | C4—C5—C6    | 118.53 (17)  |
| C1 <sup>i</sup> —C1—H1A  | 109.6        | C4—C5—H5    | 120.7        |
| N1—C1—H1B                | 109.6        | C6—C5—H5    | 120.7        |
| C1 <sup>i</sup> —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 <sup>i</sup> | 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 codes: (i)  $-x+1, -y+1, -z+2$ .

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

| $D-H\cdots A$                          | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| C5—H5 <sup>iii</sup> —N1 <sup>ii</sup> | 0.95  | 2.53        | 3.471 (2)   | 171           |

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

Fig. 1

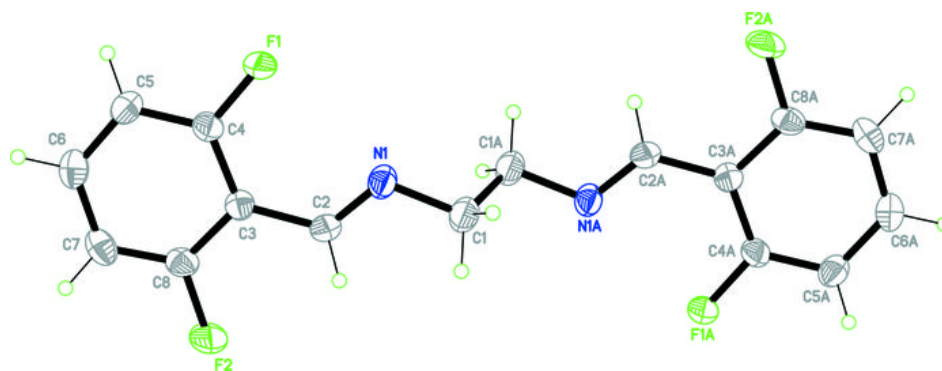


Fig. 2

