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4,4'-[Ethylenebis(nitrilomethylidene)]-dibenzonitrile

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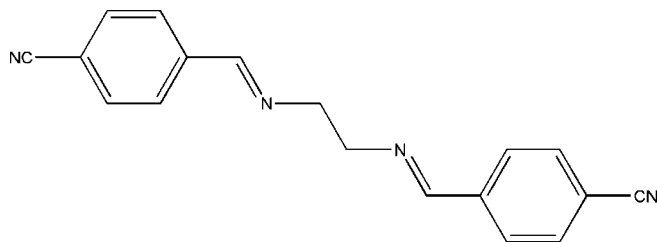
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.047; wR factor = 0.146; data-to-parameter ratio = 25.5.

The molecule of the title Schiff base compound, $\text{C}_{18}\text{H}_{14}\text{N}_4$, lies across a crystallographic inversion centre and adopts an E configuration with respect to the azomethine ($\text{C}=\text{N}$) bonds. The imino groups are coplanar with the aromatic rings with a maximum deviation of 0.1574 (12) Å for the N atom. Within the molecule, the planar units are parallel, but extend in opposite directions from the dimethylene bridge. In the crystal structure, pairs of intermolecular $\text{C}-\text{H}\cdots\text{N}$ hydrogen bonds link neighbouring molecules into centrosymmetric dimers with $R_2^2(10)$ ring motifs. An interesting feature of the crystal structure is the short intermolecular $\text{C}\cdots\text{C}$ interaction with a distance of 3.3821 (13) Å, which is shorter than the sum of the van der Waals radius of a carbon atom.

Related literature

For bond-length data, see Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures see, for example: Fun & Kia (2008); Fun, Kargar & Kia (2008); Fun, Kia & Kargar (2008). For information on Schiff base complexes and their applications, see, for example, Pal *et al.* (2005); Calligaris & Randaccio, (1987). Hou *et al.* (2001); Ren *et al.* (2002). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{14}\text{N}_4$
 $M_r = 286.33$
 Triclinic, $P\bar{1}$
 $a = 4.6843$ (2) Å
 $b = 6.9872$ (3) Å
 $c = 11.6208$ (5) Å
 $\alpha = 78.147$ (3)°
 $\beta = 87.462$ (3)°
 $\gamma = 74.081$ (2)°
 $V = 357.94$ (3) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 100$ K
 $0.45 \times 0.29 \times 0.06$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.964$, $T_{\max} = 0.995$
 7927 measured reflections
 2551 independent reflections
 2034 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.146$
 $S = 1.08$
 2551 reflections
 100 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C4}-\text{H4A}\cdots\text{N2}^i$ | 0.93 | 2.60 | 3.4702 (12) | 156 |

Symmetry code: (i) $-x + 3, -y + 1, -z + 1$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; 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, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2733).

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4,4'-[Ethylenebis(nitrilomethylidyne)]dibenzonitrile

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Comment

Schiff bases are among the most prevalent mixed-donor ligands in the field of coordination chemistry in which there has been growing interest, mainly because of their wide application in the areas such as 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 had their X-ray structures reported (Calligaris & Randaccio, 1987). As an extension of our work (Fun, Kargar & Kia 2008; Fun, Kia & Kargar 2008) on the structural characterization of Schiff base ligands, the title compound (I), is reported here.

The molecule of the title compound, (Fig. 1), lies across a crystallographic inversion centre and adopts an *E* configuration with respect to the azomethine (C=N) bond. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable with the values found in related structures (Fun & Kia 2008; Fun, Kargar & Kia 2008; Fun, Kia & Kargar 2008). The two planar units are parallel but extend in opposite directions from the dimethylene bridge. The interesting feature of the crystal structure is the short intermolecular C3...C6 interactions [symmetry code: 1 + x, y, z] with a distance of 3.3821 (13) Å, which is shorter than the sum of the van der Waals radius of carbon atom. In the crystal structure, pairs of intermolecular C—H...N hydrogen bonds link neighbouring molecules into dimer with $R^2_2(10)$ ring motif (Bernstein *et al.*, 1995) (Table 1, Fig. 2).

Experimental

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

Refinement

All of the hydrogen atoms were positioned geometrically with C—H = 0.95 or 0.97 Å and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

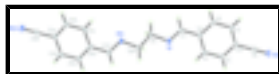


Fig. 1. The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms. The suffix A corresponds to symmetry code $[-x + 1, -y, -z]$.

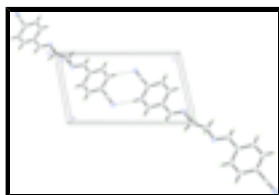


Fig. 2. The crystal packing of (I), viewed approximately down the *a*-axis, showing dimer formation by $R^2_2(10)$ ring motif. Intermolecular interactions are shown as dashed lines.

4,4'-[Ethylenebis(nitrilomethyldyne)]dibenzonitrile

Crystal data

| | |
|--------------------------------|---|
| $C_{18}H_{14}N_4$ | $Z = 1$ |
| $M_r = 286.33$ | $F_{000} = 150$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.328 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation |
| $a = 4.6843 (2) \text{ \AA}$ | $\lambda = 0.71073 \text{ \AA}$ |
| $b = 6.9872 (3) \text{ \AA}$ | Cell parameters from 3276 reflections |
| $c = 11.6208 (5) \text{ \AA}$ | $\theta = 3.1\text{--}36.3^\circ$ |
| $\alpha = 78.147 (3)^\circ$ | $\mu = 0.08 \text{ mm}^{-1}$ |
| $\beta = 87.462 (3)^\circ$ | $T = 100 \text{ K}$ |
| $\gamma = 74.081 (2)^\circ$ | Plate, colourless |
| $V = 357.94 (3) \text{ \AA}^3$ | $0.45 \times 0.29 \times 0.06 \text{ mm}$ |

Data collection

| | |
|--|--|
| Bruker SMART APEXII CCD area-detector diffractometer | 2551 independent reflections |
| Radiation source: fine-focus sealed tube | 2034 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.023$ |
| $T = 100 \text{ K}$ | $\theta_{\text{max}} = 32.5^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 3.1^\circ$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | $h = -6 \rightarrow 7$ |
| $T_{\text{min}} = 0.964$, $T_{\text{max}} = 0.995$ | $k = -10 \rightarrow 10$ |
| 7927 measured reflections | $l = -17 \rightarrow 17$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | H-atom parameters constrained |
| $wR(F^2) = 0.146$ | $w = 1/[\sigma^2(F_o^2) + (0.0885P)^2 + 0.0252P]$ |
| $S = 1.08$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2551 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 100 parameters | $\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$ |
| | Extinction correction: none |

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|-------------|----------------------------------|
| N1 | 0.59056 (17) | 0.17735 (11) | 0.08275 (6) | 0.01615 (18) |
| N2 | 1.42099 (19) | 0.74379 (12) | 0.37951 (7) | 0.0216 (2) |
| C1 | 0.8714 (2) | 0.45474 (13) | 0.15338 (7) | 0.01522 (19) |
| H1A | 0.7912 | 0.4927 | 0.0776 | 0.018* |
| C2 | 1.0275 (2) | 0.57187 (13) | 0.19178 (8) | 0.01621 (19) |
| H2A | 1.0552 | 0.6873 | 0.1416 | 0.019* |
| C3 | 1.14366 (19) | 0.51580 (13) | 0.30666 (7) | 0.01489 (19) |
| C4 | 1.1063 (2) | 0.34172 (13) | 0.38257 (7) | 0.01654 (19) |
| H4A | 1.1845 | 0.3047 | 0.4586 | 0.020* |
| C5 | 0.9504 (2) | 0.22467 (13) | 0.34268 (8) | 0.01632 (19) |
| H5A | 0.9235 | 0.1088 | 0.3927 | 0.020* |
| C6 | 0.83369 (19) | 0.27878 (13) | 0.22843 (7) | 0.01374 (18) |
| C7 | 0.67371 (19) | 0.14854 (13) | 0.18937 (7) | 0.01451 (18) |
| H7A | 0.6322 | 0.0424 | 0.2440 | 0.017* |
| C8 | 0.4324 (2) | 0.03967 (13) | 0.05429 (7) | 0.01563 (19) |
| H8A | 0.2243 | 0.1108 | 0.0394 | 0.019* |
| H8B | 0.4460 | -0.0732 | 0.1202 | 0.019* |
| C9 | 1.2999 (2) | 0.64058 (13) | 0.34745 (7) | 0.01641 (19) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|-------------|-------------|
| N1 | 0.0164 (4) | 0.0181 (3) | 0.0170 (3) | -0.0075 (3) | -0.0010 (3) | -0.0063 (3) |
| N2 | 0.0229 (4) | 0.0232 (4) | 0.0216 (4) | -0.0100 (3) | -0.0030 (3) | -0.0054 (3) |
| C1 | 0.0149 (4) | 0.0185 (4) | 0.0139 (4) | -0.0058 (3) | -0.0021 (3) | -0.0047 (3) |
| C2 | 0.0167 (4) | 0.0168 (4) | 0.0169 (4) | -0.0068 (3) | -0.0011 (3) | -0.0040 (3) |
| C3 | 0.0127 (4) | 0.0173 (4) | 0.0168 (4) | -0.0051 (3) | -0.0004 (3) | -0.0067 (3) |
| C4 | 0.0169 (4) | 0.0191 (4) | 0.0151 (4) | -0.0059 (3) | -0.0028 (3) | -0.0047 (3) |
| C5 | 0.0175 (4) | 0.0166 (4) | 0.0159 (4) | -0.0063 (3) | -0.0015 (3) | -0.0029 (3) |
| C6 | 0.0120 (4) | 0.0157 (4) | 0.0149 (4) | -0.0040 (3) | -0.0001 (3) | -0.0056 (3) |

supplementary materials

| | | | | | | |
|----|------------|------------|------------|-------------|-------------|-------------|
| C7 | 0.0142 (4) | 0.0159 (4) | 0.0153 (4) | -0.0059 (3) | 0.0003 (3) | -0.0050 (3) |
| C8 | 0.0159 (4) | 0.0183 (4) | 0.0162 (4) | -0.0085 (3) | -0.0008 (3) | -0.0058 (3) |
| C9 | 0.0158 (4) | 0.0182 (4) | 0.0159 (4) | -0.0047 (3) | -0.0012 (3) | -0.0046 (3) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------------|-------------|--------------------------|--------------|
| N1—C7 | 1.2745 (11) | C4—C5 | 1.3893 (12) |
| N1—C8 | 1.4585 (11) | C4—H4A | 0.9300 |
| N2—C9 | 1.1551 (11) | C5—C6 | 1.3962 (12) |
| C1—C2 | 1.3821 (11) | C5—H5A | 0.9300 |
| C1—C6 | 1.4031 (12) | C6—C7 | 1.4730 (11) |
| C1—H1A | 0.9300 | C7—H7A | 0.9300 |
| C2—C3 | 1.4017 (12) | C8—C8 ⁱ | 1.5246 (16) |
| C2—H2A | 0.9300 | C8—H8A | 0.9700 |
| C3—C4 | 1.3966 (12) | C8—H8B | 0.9700 |
| C3—C9 | 1.4389 (11) | | |
| C7—N1—C8 | 117.00 (7) | C6—C5—H5A | 119.6 |
| C2—C1—C6 | 120.14 (8) | C5—C6—C1 | 119.61 (8) |
| C2—C1—H1A | 119.9 | C5—C6—C7 | 118.94 (7) |
| C6—C1—H1A | 119.9 | C1—C6—C7 | 121.45 (8) |
| C1—C2—C3 | 119.68 (8) | N1—C7—C6 | 121.78 (8) |
| C1—C2—H2A | 120.2 | N1—C7—H7A | 119.1 |
| C3—C2—H2A | 120.2 | C6—C7—H7A | 119.1 |
| C4—C3—C2 | 120.80 (8) | N1—C8—C8 ⁱ | 109.60 (9) |
| C4—C3—C9 | 119.58 (8) | N1—C8—H8A | 109.8 |
| C2—C3—C9 | 119.61 (7) | C8 ⁱ —C8—H8A | 109.8 |
| C5—C4—C3 | 118.95 (8) | N1—C8—H8B | 109.8 |
| C5—C4—H4A | 120.5 | C8 ⁱ —C8—H8B | 109.8 |
| C3—C4—H4A | 120.5 | H8A—C8—H8B | 108.2 |
| C4—C5—C6 | 120.81 (8) | N2—C9—C3 | 178.76 (9) |
| C4—C5—H5A | 119.6 | | |
| C6—C1—C2—C3 | -1.03 (13) | C4—C5—C6—C7 | 179.13 (7) |
| C1—C2—C3—C4 | 0.65 (13) | C2—C1—C6—C5 | 1.07 (13) |
| C1—C2—C3—C9 | -178.50 (7) | C2—C1—C6—C7 | -178.76 (7) |
| C2—C3—C4—C5 | -0.28 (13) | C8—N1—C7—C6 | -179.67 (7) |
| C9—C3—C4—C5 | 178.86 (7) | C5—C6—C7—N1 | -172.24 (8) |
| C3—C4—C5—C6 | 0.32 (14) | C1—C6—C7—N1 | 7.59 (14) |
| C4—C5—C6—C1 | -0.71 (14) | C7—N1—C8—C8 ⁱ | -131.42 (10) |

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

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| C4—H4A \cdots N2 ⁱⁱ | 0.93 | 2.60 | 3.4702 (12) | 156 |

Symmetry codes: (ii) $-x+3, -y+1, -z+1$.

Fig. 1

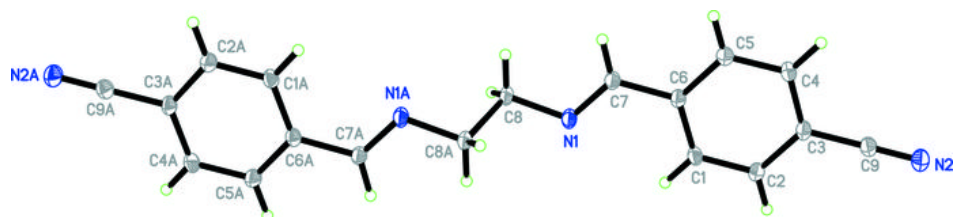


Fig. 2

