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3-[4-(Dimethylamino)benzylidene-amino]benzonitrile

Hai-Jun Xu,^{a*} Qin-Ying Tan,^a Li-Jing Cui^a and Kun Qian^b^aOrdered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China, and ^bJiangxi University of Traditional Medicine, Nanchang 330047, People's Republic of China

Correspondence e-mail: xuhj@seu.edu.cn

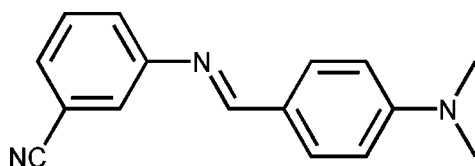
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.110; data-to-parameter ratio = 14.7.

The molecule of the title Schiff base, $\text{C}_{16}\text{H}_{15}\text{N}_3$, is non-planar and displays a *trans* configuration with respect to the $\text{C}=\text{N}$ double bond. The two benzene rings make a dihedral angle of $49.24(3)^\circ$.

Related literature

For general background on Schiff base coordination complexes, see: Garnovskii *et al.* (1993). For a related structure, see: Gong & Xu (2008)



Experimental

Crystal data

$\text{C}_{16}\text{H}_{15}\text{N}_3$
 $M_r = 249.31$
 Monoclinic, $P2_1/c$
 $a = 6.0924(6)$ Å
 $b = 29.127(3)$ Å
 $c = 7.3768(7)$ Å
 $\beta = 92.924(1)^\circ$

$V = 1307.3(2)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293$ K
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.961$, $T_{\max} = 1.000$
 (expected range = 0.947–0.985)

7018 measured reflections
 2559 independent reflections
 2241 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.110$
 $S = 1.05$
 2559 reflections

174 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2436).

References

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supplementary materials

Acta Cryst. (2009). E65, o945 [doi:10.1107/S1600536809011568]

3-[4-(Dimethylamino)benzylideneamino]benzonitrile

H.-J. Xu, Q.-Y. Tan, L.-J. Cui and K. Qian

Comment

Schiff base compounds have attracted great attention and been extensively investigated in terms of their crystallography and coordination chemistry (Garnovskii *et al.*, 1993). As a continuation of our studies on Schiff-base compounds, we here report the synthesis and crystal structure of the title compound (I).

The molecule displays a *trans* configuration with respect to the C=N double bond (Fig. 1). The values of the C—C, C=C, C—N and C=N bond distances in (I) are similar to the corresponding bond lengths in 4-(2-Hydroxybenzylideneamino)benzonitrile (Gong & Xu, 2008). The molecule is nonplanar and the dihedral angle between the planes of the two benzene rings is 49.24 (0.03) °.

Experimental

All chemicals were obtained from commercial sources and directly used without further purification. 3-aminobenzonitrile (1.18 g, 10 mmol) and 4-(dimethylamino)benzaldehyde (1.49 g, 10 mmol) were dissolved in ethanol (20 ml). The mixture was heated to reflux for 6 h, then cooled to room temperature overnight and large amounts of a yellow precipitate were formed. Yellow crystals were obtained by recrystallization from ethanol (yield: 2.04, 82%). ¹H-NMR (CDCl₃, 300 MHz): δ 3.10 (s, 6 H), 6.73–6.76 (d, 2 H), 7.45–7.50 (m, 4 H), 7.84–7.87 (d, 2 H), 8.27 (s, 1 H). ESI-MS: calcd for C₁₆H₁₅N₃ *m/z* 249.31, found 250.18 [*M*+1]. For the X-ray diffraction analysis, suitable single crystals of compound (I) were obtained after two weeks by slow evaporation from an ethanol solution.

Refinement

All H atoms attached to C were positioned geometrically and treated as riding, with C—H = 0.93 (aromatic), 0.93 (methine) or 0.96 Å (methyl) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

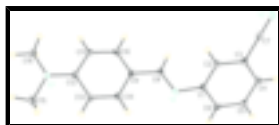


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

3-[4-(Dimethylamino)benzylideneamino]benzonitrile

Crystal data

C₁₆H₁₅N₃

$F_{000} = 528$

supplementary materials

$M_r = 249.31$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.0924$ (6) Å

$b = 29.127$ (3) Å

$c = 7.3768$ (7) Å

$\beta = 92.9240$ (10)°

$V = 1307.3$ (2) Å³

$Z = 4$

$D_x = 1.267$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2559 reflections

$\theta = 2.8$ – 26.0 °

$\mu = 0.08$ mm⁻¹

$T = 293$ K

Block, yellow

$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

φ and ω scans

Absorption correction: multi-scan
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.961$, $T_{\max} = 1.000$

7018 measured reflections

2559 independent reflections

2241 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.064$

$\theta_{\max} = 26.0$ °

$\theta_{\min} = 2.8$ °

$h = -7 \rightarrow 7$

$k = -35 \rightarrow 25$

$l = -9 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.110$

$S = 1.05$

2559 reflections

174 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.1755P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.19$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Extinction correction: none

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|-------------|--------------|----------------------------------|
| N1 | 0.61108 (15) | 0.27979 (3) | 0.59956 (13) | 0.0190 (2) |
| N2 | 0.28428 (16) | 0.47022 (3) | 0.70360 (14) | 0.0263 (3) |
| N3 | 0.14557 (15) | 0.08098 (3) | 0.61116 (13) | 0.0189 (2) |
| C1 | 0.65373 (17) | 0.32723 (4) | 0.58571 (14) | 0.0171 (2) |
| C2 | 0.50894 (17) | 0.36071 (4) | 0.64200 (14) | 0.0169 (2) |
| H2A | 0.3790 | 0.3523 | 0.6941 | 0.020* |
| C3 | 0.56051 (17) | 0.40708 (4) | 0.61955 (14) | 0.0175 (3) |
| C4 | 0.75672 (18) | 0.42049 (4) | 0.54437 (14) | 0.0190 (3) |
| H4A | 0.7889 | 0.4514 | 0.5285 | 0.023* |
| C5 | 0.90207 (18) | 0.38678 (4) | 0.49403 (15) | 0.0207 (3) |
| H5A | 1.0341 | 0.3951 | 0.4452 | 0.025* |
| C6 | 0.85265 (17) | 0.34081 (4) | 0.51572 (15) | 0.0197 (3) |
| H6A | 0.9532 | 0.3186 | 0.4833 | 0.024* |
| C7 | 0.40682 (18) | 0.44200 (4) | 0.66975 (15) | 0.0198 (3) |
| C8 | 0.42252 (17) | 0.26531 (4) | 0.53867 (14) | 0.0174 (3) |
| H8A | 0.3245 | 0.2863 | 0.4847 | 0.021* |
| C9 | 0.35526 (17) | 0.21769 (4) | 0.55017 (14) | 0.0171 (3) |
| C10 | 0.14627 (17) | 0.20363 (4) | 0.48467 (15) | 0.0188 (3) |
| H10A | 0.0515 | 0.2251 | 0.4298 | 0.023* |
| C11 | 0.07713 (17) | 0.15861 (4) | 0.49941 (15) | 0.0187 (3) |
| H11A | -0.0617 | 0.1502 | 0.4526 | 0.022* |
| C12 | 0.21502 (17) | 0.12527 (4) | 0.58471 (14) | 0.0162 (2) |
| C13 | 0.42785 (17) | 0.13953 (4) | 0.64815 (14) | 0.0176 (3) |
| H13A | 0.5238 | 0.1182 | 0.7028 | 0.021* |
| C14 | 0.49460 (17) | 0.18430 (4) | 0.63022 (14) | 0.0175 (2) |
| H14A | 0.6355 | 0.1926 | 0.6722 | 0.021* |
| C15 | 0.30017 (19) | 0.04552 (4) | 0.67163 (16) | 0.0229 (3) |
| H15A | 0.3783 | 0.0553 | 0.7812 | 0.034* |
| H15B | 0.4027 | 0.0401 | 0.5793 | 0.034* |
| H15C | 0.2218 | 0.0177 | 0.6943 | 0.034* |
| C16 | -0.06638 (18) | 0.06583 (4) | 0.53393 (16) | 0.0222 (3) |
| H16A | -0.1766 | 0.0881 | 0.5599 | 0.033* |
| H16B | -0.1035 | 0.0368 | 0.5860 | 0.033* |
| H16C | -0.0590 | 0.0626 | 0.4049 | 0.033* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|-------------|-------------|
| N1 | 0.0189 (5) | 0.0166 (5) | 0.0216 (5) | 0.0000 (4) | 0.0009 (4) | 0.0003 (4) |
| N2 | 0.0295 (5) | 0.0194 (5) | 0.0304 (6) | -0.0017 (4) | 0.0058 (4) | -0.0035 (4) |
| N3 | 0.0188 (5) | 0.0154 (5) | 0.0224 (5) | -0.0007 (4) | 0.0011 (4) | 0.0008 (4) |
| C1 | 0.0179 (5) | 0.0166 (5) | 0.0162 (5) | -0.0016 (4) | -0.0034 (4) | 0.0017 (4) |

supplementary materials

| | | | | | | |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C2 | 0.0150 (5) | 0.0187 (6) | 0.0170 (5) | -0.0024 (4) | 0.0005 (4) | 0.0002 (4) |
| C3 | 0.0190 (5) | 0.0182 (6) | 0.0151 (5) | -0.0014 (4) | -0.0010 (4) | -0.0016 (4) |
| C4 | 0.0211 (5) | 0.0184 (6) | 0.0172 (5) | -0.0060 (4) | -0.0022 (4) | 0.0005 (4) |
| C5 | 0.0161 (5) | 0.0262 (6) | 0.0196 (6) | -0.0049 (5) | 0.0002 (4) | 0.0005 (5) |
| C6 | 0.0149 (5) | 0.0231 (6) | 0.0209 (5) | 0.0023 (4) | -0.0010 (4) | -0.0014 (4) |
| C7 | 0.0225 (6) | 0.0170 (6) | 0.0199 (5) | -0.0056 (5) | 0.0009 (4) | -0.0009 (4) |
| C8 | 0.0165 (5) | 0.0171 (6) | 0.0187 (5) | 0.0033 (4) | 0.0027 (4) | 0.0015 (4) |
| C9 | 0.0171 (5) | 0.0170 (5) | 0.0174 (6) | 0.0012 (4) | 0.0039 (4) | 0.0001 (4) |
| C10 | 0.0164 (5) | 0.0180 (6) | 0.0221 (6) | 0.0042 (4) | 0.0013 (4) | 0.0018 (4) |
| C11 | 0.0147 (5) | 0.0196 (6) | 0.0219 (6) | -0.0004 (4) | 0.0014 (4) | -0.0001 (4) |
| C12 | 0.0181 (5) | 0.0162 (5) | 0.0148 (5) | 0.0015 (4) | 0.0046 (4) | -0.0003 (4) |
| C13 | 0.0186 (5) | 0.0176 (5) | 0.0167 (5) | 0.0041 (4) | 0.0013 (4) | 0.0013 (4) |
| C14 | 0.0162 (5) | 0.0189 (5) | 0.0175 (5) | 0.0008 (4) | 0.0012 (4) | -0.0007 (4) |
| C15 | 0.0292 (6) | 0.0158 (6) | 0.0233 (6) | 0.0000 (5) | -0.0039 (5) | 0.0009 (4) |
| C16 | 0.0198 (6) | 0.0184 (6) | 0.0285 (6) | -0.0020 (4) | 0.0035 (5) | -0.0007 (5) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|--------------|-------------|
| N1—C8 | 1.2833 (14) | C8—H8A | 0.9300 |
| N1—C1 | 1.4105 (14) | C9—C10 | 1.3998 (15) |
| N2—C7 | 1.1466 (15) | C9—C14 | 1.4013 (15) |
| N3—C12 | 1.3746 (14) | C10—C11 | 1.3834 (16) |
| N3—C15 | 1.4526 (14) | C10—H10A | 0.9300 |
| N3—C16 | 1.4531 (14) | C11—C12 | 1.4109 (15) |
| C1—C2 | 1.3924 (15) | C11—H11A | 0.9300 |
| C1—C6 | 1.3984 (15) | C12—C13 | 1.4181 (15) |
| C2—C3 | 1.3986 (15) | C13—C14 | 1.3743 (15) |
| C2—H2A | 0.9300 | C13—H13A | 0.9300 |
| C3—C4 | 1.3988 (15) | C14—H14A | 0.9300 |
| C3—C7 | 1.4434 (16) | C15—H15A | 0.9600 |
| C4—C5 | 1.3854 (16) | C15—H15B | 0.9600 |
| C4—H4A | 0.9300 | C15—H15C | 0.9600 |
| C5—C6 | 1.3836 (16) | C16—H16A | 0.9600 |
| C5—H5A | 0.9300 | C16—H16B | 0.9600 |
| C6—H6A | 0.9300 | C16—H16C | 0.9600 |
| C8—C9 | 1.4501 (15) | | |
| C8—N1—C1 | 117.49 (9) | C14—C9—C8 | 121.43 (10) |
| C12—N3—C15 | 120.76 (9) | C11—C10—C9 | 121.69 (10) |
| C12—N3—C16 | 120.20 (9) | C11—C10—H10A | 119.2 |
| C15—N3—C16 | 116.99 (9) | C9—C10—H10A | 119.2 |
| C2—C1—C6 | 119.09 (10) | C10—C11—C12 | 120.71 (10) |
| C2—C1—N1 | 122.86 (10) | C10—C11—H11A | 119.6 |
| C6—C1—N1 | 118.04 (10) | C12—C11—H11A | 119.6 |
| C1—C2—C3 | 119.44 (10) | N3—C12—C11 | 121.91 (9) |
| C1—C2—H2A | 120.3 | N3—C12—C13 | 120.73 (10) |
| C3—C2—H2A | 120.3 | C11—C12—C13 | 117.33 (10) |
| C2—C3—C4 | 121.21 (10) | C14—C13—C12 | 121.08 (10) |
| C2—C3—C7 | 119.83 (10) | C14—C13—H13A | 119.5 |
| C4—C3—C7 | 118.94 (10) | C12—C13—H13A | 119.5 |

| | | | |
|------------|-------------|---------------|-------------|
| C5—C4—C3 | 118.64 (10) | C13—C14—C9 | 121.54 (10) |
| C5—C4—H4A | 120.7 | C13—C14—H14A | 119.2 |
| C3—C4—H4A | 120.7 | C9—C14—H14A | 119.2 |
| C6—C5—C4 | 120.59 (10) | N3—C15—H15A | 109.5 |
| C6—C5—H5A | 119.7 | N3—C15—H15B | 109.5 |
| C4—C5—H5A | 119.7 | H15A—C15—H15B | 109.5 |
| C5—C6—C1 | 120.95 (10) | N3—C15—H15C | 109.5 |
| C5—C6—H6A | 119.5 | H15A—C15—H15C | 109.5 |
| C1—C6—H6A | 119.5 | H15B—C15—H15C | 109.5 |
| N2—C7—C3 | 177.67 (13) | N3—C16—H16A | 109.5 |
| N1—C8—C9 | 123.00 (10) | N3—C16—H16B | 109.5 |
| N1—C8—H8A | 118.5 | H16A—C16—H16B | 109.5 |
| C9—C8—H8A | 118.5 | N3—C16—H16C | 109.5 |
| C10—C9—C14 | 117.61 (10) | H16A—C16—H16C | 109.5 |
| C10—C9—C8 | 120.95 (10) | H16B—C16—H16C | 109.5 |

Fig. 1

