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2-[(Pyridin-3-ylamino)methyl]phenol

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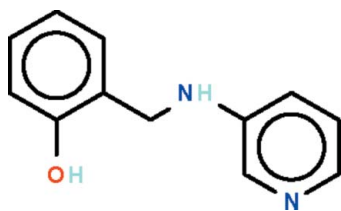
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.137; data-to-parameter ratio = 16.4.

In the title compound, $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}$, the aromatic rings at either ends of the $-\text{CH}_2-\text{NH}-$ link are twisted by 68.79 (7)°. In the crystal, the hydroxy substituent is a hydrogen-bond donor to the N atom of the pyridine ring of an adjacent molecule, and the hydrogen bond generates a chain along the b axis; it is also a hydrogen-bond acceptor to the amino group of another adjacent molecule. The two hydrogen bonds lead to the formation of a layer structure.

Related literature

For the N -salicylidene-3-aminopyridine precursor, see: Csaszar (1990); Kaya & Guelel (2005); Robert *et al.* (2009). For a related structure, see: Xu *et al.* (2011).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}$
 $M_r = 200.24$
 Monoclinic, $P2_1/c$
 $a = 5.8386$ (11) Å
 $b = 13.399$ (3) Å
 $c = 13.169$ (3) Å
 $\beta = 90.519$ (6)°

$V = 1030.1$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293$ K
 $0.21 \times 0.12 \times 0.12$ mm

Data collection

Rigaku R-Axis RAPID IP diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.983$, $T_{\max} = 0.990$

9845 measured reflections
 2358 independent reflections
 1879 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.137$
 $S = 1.08$
 2358 reflections
 144 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.26$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|----------|-------------|-------------|---------------|
| $\text{O1}-\text{H1o}\cdots\text{N2}^i$ | 0.86 (1) | 1.80 (1) | 2.6568 (16) | 175 (2) |
| $\text{N1}-\text{H1n}\cdots\text{O1}^{ii}$ | 0.88 (1) | 2.38 (1) | 3.2296 (17) | 163 (1) |

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSK, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5384).

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supplementary materials

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2-[(Pyridin-3-ylamino)methyl]phenol

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Comment

There are numerous studies on the Schiff bases derived by condensing salicylaldehyde and an aromatic amine. In this study, the azomethine double-bond of *N*-salicylidene-3-aminopyridine (Csaszar, 1990; Kaya & Guelel, 2005; Robert *et al.* 2009) is reduced by sodium borohydride to yield the title secondary amine (Scheme 1). The two aromatic rings at either ends of the –CH₂–NH– link of C₁₂H₁₂N₂O are twisted by 68.79 (7)° (Fig. 1). The hydroxy substituent is hydrogen-bond donor to the N atom of the pyridyl ring of an adjacent molecule, and the hydrogen bond generates a linear chain along the *b*-axis. It is also hydrogen-bond acceptor to the amino group of another adjacent molecule; the two hydrogen bonds lead to the formation of a layer structure (Table 1).

Experimental

A solution of 3-aminopyridine (1 mmol) and salicylaldehyde (1 mmol) in toluene (50 ml) was heated for 10 h. The solvent was removed under vacuum, and the residue was reduced in absolute methanol by sodium borohydride. Colorless crystals were obtained by recrystallization from methanol; yield 80%.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints N–H 0.88±0.01 Å and O–H 0.84±0.01 Å; their temperature factors were refined.

Figures

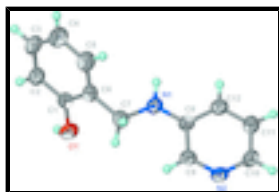


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C₁₂H₁₂N₂O at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2-[(Pyridin-3-ylamino)methyl]phenol

Crystal data

C₁₂H₁₂N₂O

$M_r = 200.24$

Monoclinic, $P2_1/c$

$F(000) = 424$

$D_x = 1.291 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

supplementary materials

Hall symbol: -P 2ybc

$a = 5.8386$ (11) Å

$b = 13.399$ (3) Å

$c = 13.169$ (3) Å

$\beta = 90.519$ (6)°

$V = 1030.1$ (4) Å³

$Z = 4$

Cell parameters from 5377 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 0.08$ mm⁻¹

$T = 293$ K

Prism, colorless

$0.21 \times 0.12 \times 0.12$ mm

Data collection

Rigaku R-Axis RAPID IP
diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scan

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.983$, $T_{\max} = 0.990$

9845 measured reflections

2358 independent reflections

1879 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.0$ °

$h = -7 \rightarrow 7$

$k = -17 \rightarrow 17$

$l = -17 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.137$

$S = 1.08$

2358 reflections

144 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.0952P]$

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

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

$\Delta\rho_{\max} = 0.26$ e Å⁻³

$\Delta\rho_{\min} = -0.18$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| O1 | 1.17862 (17) | 0.30493 (7) | 0.38420 (7) | 0.0517 (3) |
| H1O | 1.249 (3) | 0.2486 (9) | 0.3790 (12) | 0.073 (5)* |
| N1 | 0.6391 (2) | 0.42170 (9) | 0.32008 (9) | 0.0512 (3) |
| H1N | 0.534 (2) | 0.3813 (10) | 0.3444 (11) | 0.059 (4)* |
| N2 | 0.61025 (18) | 0.63154 (8) | 0.14353 (8) | 0.0493 (3) |
| C1 | 1.0374 (2) | 0.30367 (9) | 0.46539 (9) | 0.0396 (3) |
| C2 | 1.0673 (2) | 0.23629 (11) | 0.54458 (10) | 0.0499 (3) |
| H2 | 1.1882 | 0.1911 | 0.5429 | 0.060* |
| C3 | 0.9202 (3) | 0.23577 (13) | 0.62516 (10) | 0.0591 (4) |
| H3 | 0.9407 | 0.1897 | 0.6773 | 0.071* |

| | | | | |
|-----|------------|--------------|--------------|------------|
| C4 | 0.7428 (3) | 0.30289 (14) | 0.62928 (11) | 0.0669 (5) |
| H4 | 0.6418 | 0.3022 | 0.6835 | 0.080* |
| C5 | 0.7166 (3) | 0.37110 (12) | 0.55208 (11) | 0.0597 (4) |
| H5 | 0.5982 | 0.4173 | 0.5557 | 0.072* |
| C6 | 0.8605 (2) | 0.37334 (9) | 0.46929 (9) | 0.0431 (3) |
| C7 | 0.8270 (2) | 0.44915 (10) | 0.38681 (11) | 0.0484 (3) |
| H7A | 0.7961 | 0.5138 | 0.4169 | 0.058* |
| H7B | 0.9664 | 0.4547 | 0.3477 | 0.058* |
| C8 | 0.5640 (2) | 0.48446 (9) | 0.24474 (9) | 0.0416 (3) |
| C9 | 0.6833 (2) | 0.57012 (10) | 0.21638 (10) | 0.0449 (3) |
| H9 | 0.8200 | 0.5850 | 0.2499 | 0.054* |
| C10 | 0.4163 (2) | 0.61131 (11) | 0.09441 (11) | 0.0545 (4) |
| H10 | 0.3659 | 0.6543 | 0.0434 | 0.065* |
| C11 | 0.2877 (2) | 0.52808 (12) | 0.11717 (11) | 0.0586 (4) |
| H11 | 0.1522 | 0.5152 | 0.0819 | 0.070* |
| C12 | 0.3611 (2) | 0.46471 (11) | 0.19197 (10) | 0.0528 (4) |
| H12 | 0.2755 | 0.4083 | 0.2077 | 0.063* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|-------------|------------|-------------|-------------|-------------|
| O1 | 0.0596 (6) | 0.0440 (5) | 0.0517 (6) | 0.0094 (4) | 0.0165 (5) | 0.0042 (4) |
| N1 | 0.0588 (7) | 0.0428 (6) | 0.0519 (6) | -0.0104 (5) | -0.0083 (5) | 0.0094 (5) |
| N2 | 0.0515 (6) | 0.0442 (6) | 0.0522 (6) | -0.0019 (5) | 0.0093 (5) | 0.0090 (5) |
| C1 | 0.0424 (6) | 0.0385 (6) | 0.0380 (6) | 0.0015 (5) | 0.0016 (5) | -0.0046 (5) |
| C2 | 0.0494 (7) | 0.0527 (8) | 0.0476 (7) | 0.0131 (6) | 0.0022 (6) | 0.0037 (6) |
| C3 | 0.0645 (9) | 0.0721 (10) | 0.0408 (7) | 0.0146 (7) | 0.0030 (6) | 0.0107 (6) |
| C4 | 0.0663 (9) | 0.0880 (12) | 0.0467 (8) | 0.0213 (8) | 0.0168 (7) | 0.0033 (7) |
| C5 | 0.0570 (8) | 0.0670 (10) | 0.0551 (8) | 0.0241 (7) | 0.0097 (7) | -0.0013 (7) |
| C6 | 0.0448 (6) | 0.0413 (7) | 0.0432 (6) | 0.0053 (5) | -0.0032 (5) | -0.0040 (5) |
| C7 | 0.0465 (7) | 0.0396 (7) | 0.0589 (8) | 0.0025 (5) | -0.0045 (6) | 0.0019 (6) |
| C8 | 0.0473 (6) | 0.0377 (6) | 0.0400 (6) | -0.0011 (5) | 0.0038 (5) | -0.0009 (5) |
| C9 | 0.0403 (6) | 0.0434 (7) | 0.0511 (7) | -0.0022 (5) | 0.0045 (5) | 0.0029 (5) |
| C10 | 0.0592 (8) | 0.0578 (8) | 0.0465 (7) | 0.0034 (6) | 0.0008 (6) | 0.0120 (6) |
| C11 | 0.0558 (8) | 0.0701 (10) | 0.0498 (8) | -0.0095 (7) | -0.0094 (6) | 0.0057 (7) |
| C12 | 0.0584 (8) | 0.0518 (8) | 0.0481 (7) | -0.0169 (6) | -0.0026 (6) | 0.0037 (6) |

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

| | | | |
|--------|-------------|---------|-------------|
| O1—C1 | 1.3562 (15) | C4—H4 | 0.9300 |
| O1—H1O | 0.86 (1) | C5—C6 | 1.383 (2) |
| N1—C8 | 1.3695 (16) | C5—H5 | 0.9300 |
| N1—C7 | 1.4472 (17) | C6—C7 | 1.4986 (17) |
| N1—H1N | 0.88 (1) | C7—H7A | 0.9700 |
| N2—C10 | 1.3269 (18) | C7—H7B | 0.9700 |
| N2—C9 | 1.3312 (16) | C8—C12 | 1.3936 (18) |
| C1—C2 | 1.3892 (18) | C8—C9 | 1.3953 (18) |
| C1—C6 | 1.3933 (17) | C9—H9 | 0.9300 |
| C2—C3 | 1.371 (2) | C10—C11 | 1.379 (2) |

supplementary materials

| | | | |
|-------------|--------------|----------------|--------------|
| C2—H2 | 0.9300 | C10—H10 | 0.9300 |
| C3—C4 | 1.373 (2) | C11—C12 | 1.366 (2) |
| C3—H3 | 0.9300 | C11—H11 | 0.9300 |
| C4—C5 | 1.374 (2) | C12—H12 | 0.9300 |
| C1—O1—H1O | 110.3 (12) | C1—C6—C7 | 121.29 (12) |
| C8—N1—C7 | 121.31 (11) | N1—C7—C6 | 111.17 (11) |
| C8—N1—H1N | 114.9 (10) | N1—C7—H7A | 109.4 |
| C7—N1—H1N | 117.6 (10) | C6—C7—H7A | 109.4 |
| C10—N2—C9 | 119.43 (11) | N1—C7—H7B | 109.4 |
| O1—C1—C2 | 121.78 (11) | C6—C7—H7B | 109.4 |
| O1—C1—C6 | 118.47 (11) | H7A—C7—H7B | 108.0 |
| C2—C1—C6 | 119.73 (12) | N1—C8—C12 | 120.65 (11) |
| C3—C2—C1 | 120.58 (12) | N1—C8—C9 | 122.81 (11) |
| C3—C2—H2 | 119.7 | C12—C8—C9 | 116.54 (12) |
| C1—C2—H2 | 119.7 | N2—C9—C8 | 122.96 (12) |
| C2—C3—C4 | 120.35 (13) | N2—C9—H9 | 118.5 |
| C2—C3—H3 | 119.8 | C8—C9—H9 | 118.5 |
| C4—C3—H3 | 119.8 | N2—C10—C11 | 121.55 (12) |
| C3—C4—C5 | 119.01 (14) | N2—C10—H10 | 119.2 |
| C3—C4—H4 | 120.5 | C11—C10—H10 | 119.2 |
| C5—C4—H4 | 120.5 | C12—C11—C10 | 119.44 (13) |
| C4—C5—C6 | 122.24 (13) | C12—C11—H11 | 120.3 |
| C4—C5—H5 | 118.9 | C10—C11—H11 | 120.3 |
| C6—C5—H5 | 118.9 | C11—C12—C8 | 120.07 (12) |
| C5—C6—C1 | 118.05 (12) | C11—C12—H12 | 120.0 |
| C5—C6—C7 | 120.66 (11) | C8—C12—H12 | 120.0 |
| O1—C1—C2—C3 | -179.14 (13) | C5—C6—C7—N1 | -77.50 (16) |
| C6—C1—C2—C3 | 1.9 (2) | C1—C6—C7—N1 | 102.91 (14) |
| C1—C2—C3—C4 | -0.9 (2) | C7—N1—C8—C12 | -169.54 (13) |
| C2—C3—C4—C5 | -0.7 (3) | C7—N1—C8—C9 | 10.6 (2) |
| C3—C4—C5—C6 | 1.2 (3) | C10—N2—C9—C8 | -0.4 (2) |
| C4—C5—C6—C1 | -0.2 (2) | N1—C8—C9—N2 | -179.66 (12) |
| C4—C5—C6—C7 | -179.76 (14) | C12—C8—C9—N2 | 0.5 (2) |
| O1—C1—C6—C5 | 179.63 (12) | C9—N2—C10—C11 | 0.2 (2) |
| C2—C1—C6—C5 | -1.41 (19) | N2—C10—C11—C12 | 0.0 (2) |
| O1—C1—C6—C7 | -0.77 (17) | C10—C11—C12—C8 | 0.1 (2) |
| C2—C1—C6—C7 | 178.19 (11) | N1—C8—C12—C11 | 179.81 (14) |
| C8—N1—C7—C6 | 174.01 (12) | C9—C8—C12—C11 | -0.3 (2) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|------------|-------------|-------------|---------------|
| O1—H1 _o \cdots N2 ⁱ | 0.862 (14) | 1.797 (14) | 2.6568 (16) | 175.0 (16) |
| N1—H1 _n \cdots O1 ⁱⁱ | 0.881 (13) | 2.376 (12) | 3.2296 (17) | 163.2 (12) |

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

Fig. 1

