# organic compounds

# $N^1,N^2$-Bis(2-pyridyl)formamidine

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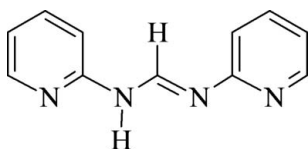Correspondence e-mail: jdchen@cycu.edu.tw

In the crystal structure of the title compound, $C_{11}H_{10}N_4$, the dihedral angle between the two pyridyl rings is 36.1 (1)°. The molecules are connected *via* two strong N—H···N and two weak C—H···N hydrogen bonds into dimers, which are located on centers of inversion. This compound adopts the *s–trans–anti–s–cis* conformation in the solid state.

## Related literature

For similar structures, see: Liang *et al.* (2003); Yang *et al.* (2000); Radak *et al.* (2001); Cotton *et al.* (1998). For the synthesis, see: Roberts (1949).



## Experimental

### Crystal data

$C_{11}H_{10}N_4$  
$M_r = 198.23$  
Monoclinic, $P2/n$  
$a = 11.0411$ (14) Å  
$b = 4.3904$ (5) Å  
$c = 20.789$ (3) Å  
$\beta = 98.725$ (2)°

$V = 996.1$ (2) Å$^3$  
$Z = 4$  
Mo $K\alpha$ radiation  
$\mu = 0.09$ mm$^{-1}$  
$T = 298$ K  
$0.44 \times 0.12 \times 0.08$ mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
Absorption correction: empirical (using intensity measurements) (*SADABS*; Bruker, 1997)  
$T_{min} = 0.983$, $T_{max} = 0.995$

3628 measured reflections  
1697 independent reflections  
1251 reflections with $I > 2\sigma(I)$  
$R_{int} = 0.041$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$  
$wR(F^2) = 0.143$  
$S = 1.13$  
1697 reflections  
141 parameters

H atoms treated by a mixture of independent and constrained refinement  
$\Delta\rho_{max} = 0.14$ e Å$^{-3}$  
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3N···N2$^i$ | 0.90 (3) | 2.14 (3) | 3.044 (3) | 175 (2) |
| C8—H8$A$···N1$^i$ | 0.93 | 2.51 | 3.388 (4) | 157 |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* and *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2134).

## References

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**supplementary materials**

# $N^1,N^2$-Bis(2-pyridyl)formamidine

## C.-J. Wu, C.-W. Su, C.-W. Yeh, J.-D. Chen and J.-C. Wang

## Comment

The title compound and its anion have been used as bridging ligands in coordination chemistry (Liang *et al.*, 2003; Yang *et al.*, 2000; Radak *et al.*, 2001; Cotton *et al.*, 1998). In the present work, the structure of the title compound (Fig. 1) has been determined to explore its ligand conformation. In the crystal structure of the title compound the molecule is in a *s-trans-anti-s-cis* conformation. This conformation is different from that in the Re complex, which is *s-cis-syn-s-cis*(Liang *et al.*, 2003).

Thus, the conformation of the free ligand has been changed upon coordination to the metal center. The molecules are connected via two strong N—H—N and two weak C—H—N hydrogen bonds into dimers, which are located on centres of inversion (Fig. 2),

## Experimental

The title compound was prepared according to a published procedure (Roberts, 1949). 2-Aminopyridine (11.28 g, 0.12 mol) and triethyl orthoformate (8.88 g, 0.06 mol) were placed in a flask under nitrogen. The mixture was then refluxed for 8 h to give a brown solid. Dichloromethane was then added to dissolve the solid and then hexanes added to induce the precipitate. The precipitate was filtered and dried under vacuum to give a light yellow solid with a yield of 82 %. Crystals suitable for X-ray crystallography were obtained by dissolving the product in dichloromethane, followed by slow evaporation of the solvent.

## Refinement

Pyridyl and methine H atoms were positioned with ideal geometry and were refined isotropic with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ using a riding model. The amine H atom was found in fourier difference map and refined isotropically.
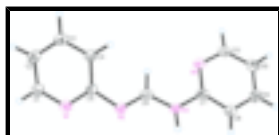
## Figures



Fig. 1. : An *ORTEP* diagram showing the structure of the title compound with labeling and displacement ellipsoids drawn at the 30 % probability level.
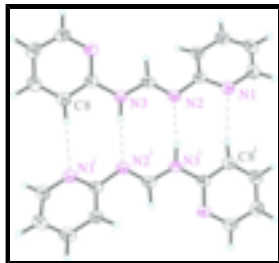
Fig. 2. : View onto the dimers formed by intermolecular hydrogen bonding, which is shown as dashed lines. Symmetry code: (i) -x+1, -y, -z+1.

## $N^1,N^2$-Bis(2-pyridyl)formamidine

*Crystal data*

| | |
|---|---|
| $C_{11}H_{10}N_4$ | $F_{000} = 416$ |
| $M_r = 198.23$ | $D_x = 1.322$ Mg m$^{-3}$ |
| Monoclinic, $P2/n$ | Mo $K\alpha$ radiation <br> $\lambda = 0.71073$ Å |
| $a = 11.0411$ (14) Å | Cell parameters from 1493 reflections |
| $b = 4.3904$ (5) Å | $\theta = 2.0–25.1°$ |
| $c = 20.789$ (3) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\beta = 98.725$ (2)° | $T = 298$ K |
| $V = 996.1$ (2) Å$^3$ | Column, colorless |
| $Z = 4$ | $0.44 \times 0.12 \times 0.08$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1697 independent reflections |
| Radiation source: fine-focus sealed tube | 1251 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.041$ |
| $T = 298$ K | $\theta_{max} = 25.1°$ |
| $\varphi$ and $\omega$ scans | $\theta_{min} = 2.0°$ |
| Absorption correction: empirical (using intensity measurements) (*SADABS*; Bruker, 1997) | $h = -13 \rightarrow 8$ |
| $T_{min} = 0.983$, $T_{max} = 0.995$ | $k = -5 \rightarrow 4$ |
| 3628 measured reflections | $l = -23 \rightarrow 24$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.059$ | $w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.3516P]$ <br> where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.143$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.13$ | $\Delta\rho_{max} = 0.14$ e Å$^{-3}$ |

| | |
|---|---|
| 1697 reflections | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |
| 141 parameters | Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.024 (4) |
| Secondary atom site location: difference Fourier map | |

*Special details*

**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 $F^2$, convention-al R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2sigma(F^2)$ is used only for calculat-ing 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.7611 (2) | −0.1912 (6) | 0.64055 (11) | 0.0633 (7) |
| N2 | 0.66575 (17) | −0.0267 (5) | 0.53928 (10) | 0.0502 (6) |
| N3 | 0.58806 (19) | 0.2810 (5) | 0.45290 (10) | 0.0482 (6) |
| N4 | 0.71668 (18) | 0.5763 (5) | 0.39863 (10) | 0.0536 (6) |
| C1 | 0.8583 (3) | −0.3057 (8) | 0.67908 (14) | 0.0731 (9) |
| H1B | 0.8510 | −0.3453 | 0.7223 | 0.088* |
| C2 | 0.9685 (3) | −0.3685 (8) | 0.65886 (15) | 0.0708 (9) |
| H2B | 1.0342 | −0.4453 | 0.6876 | 0.085* |
| C3 | 0.9785 (3) | −0.3143 (7) | 0.59503 (15) | 0.0675 (8) |
| H3A | 1.0516 | −0.3553 | 0.5796 | 0.081* |
| C4 | 0.8802 (2) | −0.1991 (7) | 0.55388 (13) | 0.0559 (7) |
| H4B | 0.8856 | −0.1632 | 0.5103 | 0.067* |
| C5 | 0.7723 (2) | −0.1370 (6) | 0.57845 (12) | 0.0483 (6) |
| C6 | 0.6813 (2) | 0.1693 (6) | 0.49522 (12) | 0.0480 (6) |
| H6A | 0.7604 | 0.2361 | 0.4926 | 0.058* |
| C7 | 0.6029 (2) | 0.4915 (6) | 0.40425 (12) | 0.0471 (6) |
| C8 | 0.5000 (2) | 0.6016 (6) | 0.36432 (13) | 0.0558 (7) |
| H8A | 0.4219 | 0.5384 | 0.3699 | 0.067* |
| C9 | 0.5161 (3) | 0.8048 (7) | 0.31657 (14) | 0.0632 (8) |
| H9A | 0.4487 | 0.8817 | 0.2891 | 0.076* |
| C10 | 0.6329 (3) | 0.8952 (7) | 0.30926 (14) | 0.0636 (8) |
| H10A | 0.6462 | 1.0314 | 0.2768 | 0.076* |
| C11 | 0.7286 (2) | 0.7769 (7) | 0.35157 (13) | 0.0596 (8) |
| H11A | 0.8073 | 0.8405 | 0.3473 | 0.072* |
| H3N | 0.512 (3) | 0.203 (6) | 0.4525 (12) | 0.058 (8)* |

# supplementary materials

*Atomic displacement parameters ($Å^2$)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0500 (13) | 0.0826 (18) | 0.0570 (14) | −0.0025 (12) | 0.0077 (11) | 0.0050 (13) |
| N2 | 0.0373 (11) | 0.0612 (14) | 0.0520 (12) | −0.0022 (10) | 0.0069 (9) | −0.0015 (11) |
| N3 | 0.0370 (11) | 0.0514 (13) | 0.0562 (13) | −0.0033 (10) | 0.0070 (10) | 0.0006 (11) |
| N4 | 0.0438 (12) | 0.0592 (14) | 0.0597 (13) | −0.0022 (10) | 0.0144 (10) | −0.0004 (11) |
| C1 | 0.0608 (18) | 0.097 (2) | 0.0590 (17) | 0.0002 (18) | 0.0027 (14) | 0.0105 (17) |
| C2 | 0.0492 (16) | 0.084 (2) | 0.075 (2) | 0.0047 (16) | −0.0052 (14) | 0.0032 (18) |
| C3 | 0.0455 (15) | 0.079 (2) | 0.079 (2) | 0.0036 (15) | 0.0121 (14) | −0.0029 (18) |
| C4 | 0.0445 (14) | 0.0701 (19) | 0.0538 (15) | 0.0014 (13) | 0.0100 (12) | −0.0024 (14) |
| C5 | 0.0407 (13) | 0.0499 (15) | 0.0541 (15) | −0.0058 (11) | 0.0061 (11) | −0.0039 (12) |
| C6 | 0.0383 (13) | 0.0518 (15) | 0.0547 (14) | −0.0026 (12) | 0.0100 (11) | −0.0094 (13) |
| C7 | 0.0435 (14) | 0.0472 (14) | 0.0515 (14) | 0.0005 (12) | 0.0106 (11) | −0.0090 (13) |
| C8 | 0.0458 (15) | 0.0579 (17) | 0.0631 (16) | 0.0007 (13) | 0.0067 (12) | −0.0030 (14) |
| C9 | 0.0622 (18) | 0.0610 (18) | 0.0649 (18) | 0.0097 (15) | 0.0051 (14) | 0.0005 (15) |
| C10 | 0.0713 (19) | 0.0610 (19) | 0.0617 (17) | 0.0038 (15) | 0.0207 (15) | 0.0031 (15) |
| C11 | 0.0535 (16) | 0.0632 (19) | 0.0660 (18) | −0.0029 (14) | 0.0214 (14) | −0.0008 (15) |

*Geometric parameters ($Å$, °)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.337 (4) | C3—C4 | 1.372 (4) |
| N1—C5 | 1.337 (3) | C3—H3A | 0.9300 |
| N2—C6 | 1.287 (3) | C4—C5 | 1.392 (3) |
| N2—C5 | 1.411 (3) | C4—H4B | 0.9300 |
| N3—C6 | 1.342 (3) | C6—H6A | 0.9300 |
| N3—C7 | 1.398 (3) | C7—C8 | 1.388 (3) |
| N3—H3N | 0.90 (3) | C8—C9 | 1.366 (4) |
| N4—C7 | 1.332 (3) | C8—H8A | 0.9300 |
| N4—C11 | 1.337 (3) | C9—C10 | 1.380 (4) |
| C1—C2 | 1.374 (4) | C9—H9A | 0.9300 |
| C1—H1B | 0.9300 | C10—C11 | 1.370 (4) |
| C2—C3 | 1.369 (4) | C10—H10A | 0.9300 |
| C2—H2B | 0.9300 | C11—H11A | 0.9300 |
| C1—N1—C5 | 117.5 (2) | C4—C5—N2 | 122.7 (2) |
| C6—N2—C5 | 116.7 (2) | N2—C6—N3 | 122.6 (2) |
| C6—N3—C7 | 123.6 (2) | N2—C6—H6A | 118.7 |
| C6—N3—H3N | 118.9 (17) | N3—C6—H6A | 118.7 |
| C7—N3—H3N | 117.1 (17) | N4—C7—C8 | 123.2 (3) |
| C7—N4—C11 | 116.5 (2) | N4—C7—N3 | 117.6 (2) |
| N1—C1—C2 | 124.1 (3) | C8—C7—N3 | 119.2 (2) |
| N1—C1—H1B | 118.0 | C9—C8—C7 | 118.5 (3) |
| C2—C1—H1B | 118.0 | C9—C8—H8A | 120.8 |
| C3—C2—C1 | 117.8 (3) | C7—C8—H8A | 120.8 |
| C3—C2—H2B | 121.1 | C8—C9—C10 | 119.6 (3) |
| C1—C2—H2B | 121.1 | C8—C9—H9A | 120.2 |
| C2—C3—C4 | 119.7 (3) | C10—C9—H9A | 120.2 |

| C2—C3—H3A | 120.1 | C11—C10—C9 | 117.6 (3) |
| C4—C3—H3A | 120.1 | C11—C10—H10A | 121.2 |
| C3—C4—C5 | 118.9 (3) | C9—C10—H10A | 121.2 |
| C3—C4—H4B | 120.6 | N4—C11—C10 | 124.6 (3) |
| C5—C4—H4B | 120.6 | N4—C11—H11A | 117.7 |
| N1—C5—C4 | 122.0 (2) | C10—C11—H11A | 117.7 |
| N1—C5—N2 | 115.2 (2) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| N3—H3N⋯N2[i] | 0.90 (3) | 2.14 (3) | 3.044 (3) | 175 (2) |
| C8—H8A⋯N1[i] | 0.93 | 2.51 | 3.388 (4) | 157 |

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

**Fig. 1**

**Fig. 2**