organic compounds

4,4′-Bipyridine–dimethylglyoxime (1/1)

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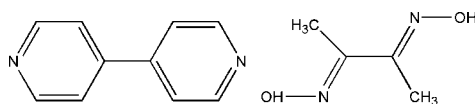
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In the title compound, $C_{10}H_8N_2 \cdot C_4H_8N_2O_2$, both the dimethyl-glyoxime and the 4,4′-bipyridine molecules have crystallographic C_i symmetry. The molecules stack along the a-axis direction with a dihedral angle of 20.4 (8)° between their planes. In the crystal, the components are linked by O—H···N hydrogen bonds into alternating chains along [120] and [1$\bar{2}$0].

Related literature

For the coordination modes of dimethylglyoxime, see: Malinovskii *et al.* (2004); Coropceanu *et al.* (2009). For its use in mediate magnetic interactions, see: Cervera *et al.* (1997).

Experimental

Crystal data

$C_{10}H_8N_2 \cdot C_4H_8N_2O_2$	c = 11.502 (2) Å
M_r = 272.31	β = 99.44 (3)°
Monoclinic, $P2_1/c$	V = 707.6 (2) Å³
a = 8.7247 (17) Å	Z = 2
b = 7.1486 (14) Å	Mo $K\alpha$ radiation

μ = 0.09 mm⁻¹	0.20 × 0.18 × 0.15 mm
T = 298 K	

Data collection

Bruker SMART APEX CCD diffractometer	9684 measured reflections
	1636 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	1265 reflections with $I > 2\sigma(I)$
T_{min} = 0.982, T_{max} = 0.987	R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043	93 parameters
$wR(F^2)$ = 0.133	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.19 e Å⁻³
1636 reflections	$\Delta\rho_{min}$ = −0.13 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^i	0.82	1.94	2.7459 (17)	169

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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*.

References

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Malinovskii, S. T., Bologa, O. A., Coropceanu, E. B., Luboradzki, R. & Gerbeleu, N. V. (2004). *Russ. J. Coord. Chem.* **30**, 339–345.

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supplementary materials

4,4'-Bipyridine-dimethylglyoxime (1/1)

Y. Yang, **Z. Huang**, **H. Lv** and **A. Han**

Comment

Dimethylglyoxime (H_2dmg) with its two oximate group (=N–O–) is a suitable scaffold to construct metal-containing building blocks for extended supramolecular architectures. Several complexes of transition metals with this ligand and its derivatives have been reported (Malinovskii *et al.*, 2004; Coropceanu *et al.*, 2009). Moreover, the NO oxime group has a remarkable efficiency to mediate magnetic interactions when it acts as a bridging ligand (Cervera *et al.*, 1997).

Starting from $Mn(CH_3COO)_2$ and H_2dmg, and using 4,4'-dpy as a bridging ligand, we have aimed to prepare a complex with superior magnetic properties. However, the reaction resulted in a stoichiometric (1:1) molecular complex of dimethylglyoxime-4,4'-bipyridine.

In this structure, the molecules of H_2dmg and 4,4'-dpy are linked through O—H···N hydrogen bonds into alternating chains (Fig. 2).

Experimental

$Mn(CH_3COO)_2$.$4H_2O$ (0.025 g, 0.1 mmol) in 5 ml of water and CH_3COONa(0.016 g, 0.2 mmol) were added to a mixture of H_2dmg (0.024 g, 0.2 mmol) and 4,4'-dpy in 10 ml of methanol. The reaction mixture was boiled in a crucible for ~10 min. The solvent was then evaporated and colorless crystals of the title compound were obtained.

Refinement

Methyl H atoms were placed in calculated position with C—H=0.96 Å, and torsion angles were refined, U_{iso}(H)=$1.5U_{eq}$(C). The position of the O-bound H-atom was determined from a difference Fourier map and then geometrically restrained with O—H=0.82 Å, and U_{iso}(H)=$1.5U_{eq}$(O). Aromatic H atoms were placed in calculated positions with C—H=0.93Å and refined in riding mode with U_{iso}(H)=$1.2U_{eq}$(C).
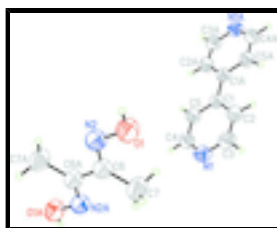
Figures

Fig. 1. Molecular structure showing 50% probability displacement ellipsoids.
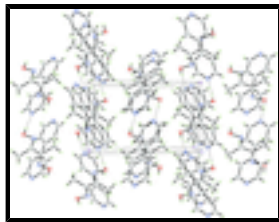
supplementary materials

Fig. 2. Heterosoric stacks of the molecules.

4,4'-Bipyridine–dimethylglyoxime (1/1)

Crystal data

$C_{10}H_8N_2 \cdot C_4H_8N_2O_2$	$F(000) = 288$
$M_r = 272.31$	$707.6(2)$
Monoclinic, $P2_1/c$	$D_x = 1.278$ Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.7247$ (17) Å	Cell parameters from 1636 reflections
$b = 7.1486$ (14) Å	$\theta = 2.4–27.5°$
$c = 11.502$ (2) Å	$\mu = 0.09$ mm^{-1}
$\beta = 99.44$ (3)°	$T = 298$ K
$V = 707.6$ (2) Å^3	Block, colourless
$Z = 2$	$0.20 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD diffractometer	1636 independent reflections
Radiation source: fine-focus sealed tube	1265 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.040$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$h = -11 \rightarrow 11$
$T_{min} = 0.982$, $T_{max} = 0.987$	$k = -9 \rightarrow 9$
9684 measured reflections	$l = -14 \rightarrow 14$

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
$wR(F^2) = 0.133$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.1229P]$ where $P = (F_o^2 + 2F_c^2)/3$
1636 reflections	$(\Delta/\sigma)_{max} = 0.021$
93 parameters	$\Delta\rho_{max} = 0.19$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.13$ e Å^{-3}

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 ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
C1	0.02851 (14)	0.09220 (19)	0.48302 (12)	0.0464 (3)
O1	0.72158 (14)	0.26180 (17)	0.14282 (10)	0.0699 (4)
H1	0.7740	0.1714	0.1296	0.105*
N1	0.14118 (15)	0.43625 (18)	0.41432 (12)	0.0600 (4)
N2	0.62500 (14)	0.31488 (17)	0.03926 (12)	0.0556 (4)
C5	−0.04603 (17)	0.1905 (2)	0.38632 (14)	0.0557 (4)
H5	−0.1364	0.1429	0.3421	0.067*
C6	0.55111 (16)	0.4662 (2)	0.05327 (13)	0.0518 (4)
C3	0.21126 (19)	0.3451 (2)	0.50901 (16)	0.0652 (5)
H3	0.2997	0.3982	0.5527	0.078*
C2	0.16017 (18)	0.1766 (2)	0.54594 (15)	0.0591 (4)
H2	0.2137	0.1190	0.6130	0.071*
C4	0.01328 (18)	0.3583 (2)	0.35553 (15)	0.0605 (4)
H4	−0.0391	0.4208	0.2899	0.073*
C7	0.5661 (2)	0.5712 (3)	0.16681 (15)	0.0701 (5)
H7A	0.5414	0.4897	0.2275	0.105*
H7B	0.4958	0.6754	0.1578	0.105*
H7C	0.6707	0.6158	0.1881	0.105*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0428 (7)	0.0475 (7)	0.0486 (7)	0.0018 (5)	0.0062 (5)	−0.0049 (6)
O1	0.0727 (8)	0.0700 (8)	0.0608 (7)	0.0253 (6)	−0.0076 (6)	−0.0006 (5)
N1	0.0600 (8)	0.0521 (7)	0.0671 (8)	−0.0065 (6)	0.0081 (6)	−0.0017 (6)
N2	0.0527 (7)	0.0553 (7)	0.0560 (7)	0.0087 (5)	0.0001 (5)	0.0000 (5)
C5	0.0513 (8)	0.0572 (8)	0.0550 (8)	−0.0064 (6)	−0.0020 (6)	0.0017 (7)
C6	0.0473 (7)	0.0539 (8)	0.0529 (8)	0.0050 (6)	0.0042 (6)	−0.0030 (6)
C3	0.0583 (9)	0.0607 (9)	0.0723 (11)	−0.0125 (7)	−0.0024 (8)	−0.0045 (8)
C2	0.0548 (8)	0.0562 (9)	0.0614 (9)	−0.0035 (7)	−0.0049 (7)	0.0018 (7)
C4	0.0621 (9)	0.0570 (9)	0.0596 (9)	−0.0021 (7)	0.0020 (7)	0.0063 (7)
C7	0.0760 (11)	0.0732 (11)	0.0567 (9)	0.0180 (9)	−0.0026 (8)	−0.0097 (8)

Geometric parameters (Å, °)

C1—C5	1.384 (2)	C6—C6ⁱⁱ	1.474 (3)
C1—C2	1.391 (2)	C6—C7	1.493 (2)
C1—C1ⁱ	1.484 (3)	C3—C2	1.376 (2)
O1—N2	1.3941 (17)	C3—H3	0.9300
O1—H1	0.8200	C2—H2	0.9300
N1—C4	1.329 (2)	C4—H4	0.9300
N1—C3	1.329 (2)	C7—H7A	0.9600
N2—C6	1.2831 (18)	C7—H7B	0.9600
C5—C4	1.376 (2)	C7—H7C	0.9600
C5—H5	0.9300		
C5—C1—C2	115.92 (14)	C2—C3—H3	118.2
C5—C1—C1ⁱ	121.91 (15)	C3—C2—C1	120.06 (15)
C2—C1—C1ⁱ	122.16 (16)	C3—C2—H2	120.0
N2—O1—H1	109.5	C1—C2—H2	120.0
C4—N1—C3	116.54 (14)	N1—C4—C5	123.68 (15)
C6—N2—O1	111.59 (12)	N1—C4—H4	118.2
C4—C5—C1	120.16 (14)	C5—C4—H4	118.2
C4—C5—H5	119.9	C6—C7—H7A	109.5
C1—C5—H5	119.9	C6—C7—H7B	109.5
N2—C6—C6ⁱⁱ	114.82 (16)	H7A—C7—H7B	109.5
N2—C6—C7	124.04 (14)	C6—C7—H7C	109.5
C6ⁱⁱ—C6—C7	121.13 (16)	H7A—C7—H7C	109.5
N1—C3—C2	123.61 (14)	H7B—C7—H7C	109.5
N1—C3—H3	118.2		
C2—C1—C5—C4	1.8 (2)	N1—C3—C2—C1	0.1 (3)
C1ⁱ—C1—C5—C4	−177.95 (15)	C5—C1—C2—C3	−1.7 (2)
O1—N2—C6—C6ⁱⁱ	178.74 (15)	C1ⁱ—C1—C2—C3	178.06 (16)
O1—N2—C6—C7	−0.5 (2)	C3—N1—C4—C5	−1.3 (2)
C4—N1—C3—C2	1.4 (3)	C1—C5—C4—N1	−0.3 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1ⁱⁱⁱ	0.82	1.94	2.7459 (17)	169.

Symmetry codes: (iii) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

Fig. 2