H atoms were placed geometrically and refined using a riding model with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Azimuthal scans were used to derive absorption corrections as the crystal was cut from a larger aggregated mass and the faces could not be indexed satisfactorily. The scans obtained were of high quality, having excellent agreement for ψ angles 180° apart and similar $T_{\rm min}/T_{\rm max}$ for each scan. Poor agreement with the expected absorption correction range is attributed to the high value of μ combined with the large relative uncertainty in measuring the thin plate dimension.

Data collection: *STADI*-4 (Stoe & Cie, 1995*a*). Cell refinement: *STADI*-4. Data reduction: *X-RED* (Stoe & Cie, 1995*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*96.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1440). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 888–890

2,6-Diacetylpyridinebis(*p*-methoxyaniline),† a Terdentate *NNN* Donor Ligand

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Abstract

The title compound, $C_{23}H_{23}N_3O_2$, is a terdentate *NNN* donor ligand. The molecule lies about a crystallographic mirror plane which passes through a C and an N atom of the substituted pyridine ring. The orientation of the exocyclic N atoms in the solid is not what is required for complex formation with metal centres.

Comment

The commercially available *NNN* donor ligand terpyridine is of considerable interest as a planar terdentate ligand, however, its synthesis is laborious and expensive (McWhinnie & Miller, 1969). Our interest in the generation of cheaper alternative terdentate ligands derived from 2,6-diacetylpyridine has been well documented (Alyea & Merrell, 1974; Alyea, Ecott & Merrell, 1982). Complexation of these ligands with transition metal salts of general formula MX_2 has produced five- and six-coordinate species of types MX_2L (M = Ni, Cd; X = Cl) and $[ML_2]X_2$ (M = Ni, Cd; X = ClO_4^-) (Alyea & Merrell, 1974; Alyea, 1983).

The Schiff base condensation of 2,6-diacetylpyridine produced the title compound, (I). The molecule (Fig. 1) lies about a mirror plane which passes through the N1 and C4 atoms. The bond lengths within the ligand are as expected. The double-bond nature of C5—N2 is shown by the bond length of 1.274 (5) Å; the N1—C2 bond length is longer, as expected, at 1.343 (4) Å. The sp^2 nature of the C5 atom is also established by the planarity of the C2, N2, C5, C6 moiety [deviations from −0.003 (1) to 0.007 (4) Å]. The interplanar angle which the C2, N2, C5, C6 plane makes with that defined by N1, C2–C5 is 1.98 (10)°. The interplanar angle between the C2, C5, C6, N2 and N2, C11–C16 planes is 57.20 (15)°. The *p*-methoxy group adopts the usual conformation and lies in the plane of its aromatic ring [torsion angle C13—C14—O1—C17 0.3 (6)°].

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† Alternative name: 2,6-bis[1-(4-methoxyphenylimino)ethyl]pyridine.

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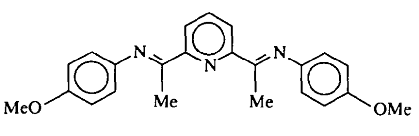

(I)

Examination of the structure with *PLATON* (Spek, 1996*a*) showed that there were no solvent-accessible voids in the crystal lattice. The orientation of the groups bonded to C5 is noteworthy. The $NC_6H_4OCH_3$ group attached to C5 is located so as to minimize steric interactions, with N1—C2—C5—N2 adopting a *trans* conformation [torsion angle −178.7 (4)°]. It is clear that for the three N atoms of the ligand to bond to a metal centre, rotation about the C2—C5 bond must occur. This is exemplified by the structure of [2,6-diacetylpyridine-bis(anilino)](nitrato-*O*,*O*′)(nitrato-*O*)nickel(II), in which the analogous N—C—C—N torsion angles are 0.8 (4)° (Alyea, Ferguson & Restivo, 1975). However, the conformation adopted by ligand (I) here is that which has the least steric hindrance.

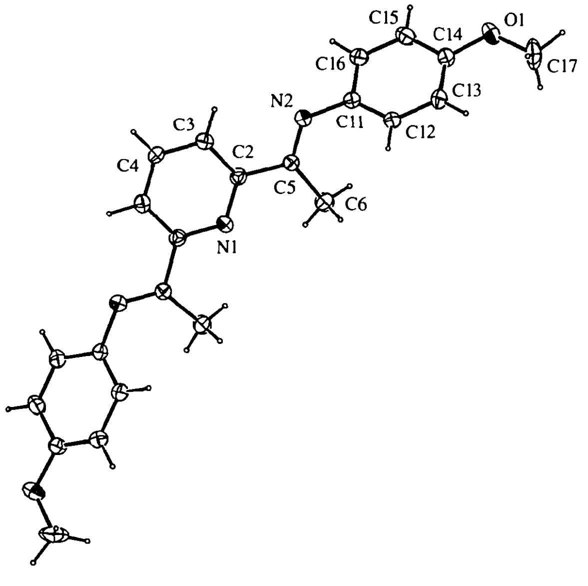

Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. A crystallographic mirror plane runs through the N1 and C4 atoms; the coordinates of the other half of the molecule are obtained from those of the asymmetric unit by the transformation ($1-x$, y, z).

Experimental

To a stirred solution of *p*-methoxyaniline (5.01 g, 40.6 mmol) in absolute ethanol (40 ml), 2,6-diacetylpyridine (3.22 g, 19.6 mmol) was added. Glacial acetic acid (1 ml) was added as a catalyst to the yellow solution, which was then refluxed for 1 h and subsequently stirred overnight. Careful solvent removal *in vacuo* induced the deposition of a yellow precipitate which was collected, washed in hexane and pumped dry. Yellow tabular **a** crystals suitable for X-ray analysis were grown in 65% yield by the slow evaporation of a dichloromethane solution of the complex.

Crystal data

$C_{23}H_{23}N_3O_2$
$M_r = 373.44$
Orthorhombic
$Cmc2_1$
$a = 40.266\ (14)$ Å
$b = 7.0440\ (13)$ Å
$c = 7.1203\ (13)$ Å
$V = 2019.6\ (9)$ Å^3
$Z = 4$
$D_x = 1.228$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.10–19.12°$
$\mu = 0.080$ mm^{-1}
$T = 294\ (1)$ K
Tabular **a**
0.39 × 0.39 × 0.19 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1221 measured reflections
1221 independent reflections
706 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 27.08°$
$h = 0 \rightarrow 51$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 9$
3 standard reflections frequency: 60 min intensity variation: 0.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.144$
$S = 0.940$
1221 reflections
133 parameters
H atoms riding (C—H 0.93–0.96 Å)
$w = 1/[\sigma^2(F_o^2) + (0.0855P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.165$ e Å^{-3}
$\Delta\rho_{\min} = -0.180$ e Å^{-3}
Extinction correction: *SHELXL93*
Extinction coefficient: 0.0015 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	1/2	0.2449 (6)	0.0000 (7)	0.0360 (11)
C2	0.47132 (9)	0.2592 (5)	0.0952 (5)	0.0341 (10)
C3	0.47059 (8)	0.2863 (6)	0.2903 (6)	0.0418 (10)
C4	1/2	0.3001 (9)	0.3871 (8)	0.047 (2)
C5	0.43970 (8)	0.2444 (6)	−0.0156 (5)	0.0340 (9)
C6	0.44234 (10)	0.2071 (7)	−0.2215 (6)	0.0503 (11)
N2	0.41299 (7)	0.2623 (5)	0.0787 (4)	0.0443 (10)
C11	0.38132 (9)	0.2576 (6)	−0.0060 (5)	0.0397 (10)
C12	0.37186 (9)	0.3808 (6)	−0.1455 (6)	0.0460 (11)
C13	0.33973 (9)	0.3800 (6)	−0.2165 (7)	0.0478 (10)
C14	0.31687 (9)	0.2518 (6)	−0.1498 (6)	0.0431 (10)
C15	0.32615 (9)	0.1293 (6)	−0.0076 (6)	0.0502 (11)
C16	0.35782 (9)	0.1335 (6)	0.0650 (7)	0.0477 (11)
O1	0.28476 (6)	0.2358 (5)	−0.2114 (5)	0.0618 (10)
C17	0.27426 (11)	0.3610 (9)	−0.3566 (9)	0.085 (2)

Table 2. *Selected geometric parameters (Å, °)*

N1—C2	1.343 (4)	C5—C6	1.493 (6)
C2—C5	1.502 (5)	N2—C11	1.411 (5)
C5—N2	1.274 (5)		
N2—C5—C6	126.5 (3)	C6—C5—C2	117.9 (3)
N2—C5—C2	115.6 (3)		
N1—C2—C5—N2	−178.7 (4)	C13—C14—O1—C17	0.3 (6)

Molecule (I) crystallized in the orthorhombic system; space groups *Cmc*2₁, *Cmcm* or *C*2*cm* allowed by the systematic absences. With $Z = 4$, space group *Cmcm* requires that the molecule have either *mm* or 2/*m* symmetry; this is not possible and this space group option was rejected. Of the two remaining space groups (both of which require that the molecule have crystallographic *m* symmetry), only the *Cmc*2₁ choice afforded a solution. The molecule is achiral (has mirror symmetry) and with only C, H, N and O atoms (which have very small anomalous dispersion components) we deemed it very unlikely that a correct determination of the direction of the polar axis in the *Cmc*2₁ space group would be possible. As a consequence of this decision, we measured one unique octant of data to a $\theta_{\max}$ of 27.5° with Mo $K\alpha$ radiation and obtained a reflection/parameter ratio of 9.18. As anticipated, the refinement did not allow a determination of the chiral axis direction.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD*-4-*PC Software*. Data reduction: *DATRD*2 in *NRCVAX*96 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *NRCVAX*96 and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *NRCVAX*96, *ORTEP*II (Johnson, 1976), *PLATON* (Spek, 1996*a*) and *PLUTON* (Spek 1996*b*). Software used to prepare material for publication: *NRCVAX*96, *SHELXL*93 and *WordPerfect* macro *PREPCIF* (Ferguson, 1996).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1460). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 890–892

N-(6-Amino-3,4-dihydro-3-methyl-5-nitroso-4-oxopyrimidin-2-yl)glycine–Water (1/2)

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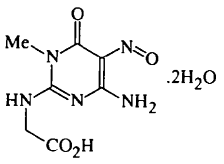
Abstract

The title compound, $C_7H_9N_5O_4.2H_2O$, makes use of the two waters of crystallization to form a hydrogen-bonded network which utilizes all available N—H and O—H groups as hydrogen-bond donors. The shortest intermolecular O—H···O bond is 2.491 (3) Å.

Comment

The title compound, (I), is of potential biological interest since it is an adduct of the amino acid glycine and a substituted uracil nucleobase. In addition, this type of compound can coordinate with transition metal ions such as Cd^{II} and Hg^{II}, whose complexes we are currently investigating.



(I)

A view of the asymmetric unit is shown in Fig. 1 and details of the geometry are given in Table 1. The dimensions of the N2—C2—N1—C6—N6 frag-