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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1235). Services for accessing these data are described at the back of the journal.

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6,6'-Dimethyl-2,2'-bipyridyl

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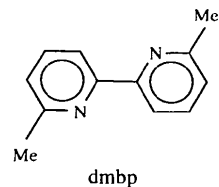
Abstract

In the solid state, the novel ligand 6,6'-dimethyl-2,2'-bipyridyl (dmbp), $C_{12}H_{12}N_2$, is a planar centrosymmetric molecule in which the pyridyl N atoms have a *transoid* arrangement, by virtue of the symmetry.

Comment

Square-planar complexes of Pt^{II} containing the title dmbp ligand, e.g. $[Pt(dmbp)Cl_2]$, have aroused much interest recently due to their unusual redox and physical properties (Zuleta *et al.*, 1990; Miskowski *et al.*, 1993).

Knowledge of the solid-state structure of this ligand will enable further understanding of its chemical behaviour and coordination ability.



The molecule crystallizes in the space group $P2_1/c$, lying on a centre of symmetry, with the N atoms of the pyridyl rings *trans* to each other around the central bond. This conformation of the uncoordinated molecule contrasts with the *cisoid* arrangement necessary when it acts as a chelating ligand. The molecule is planar, with only the methyl H atoms deviating significantly from the plane. Bond lengths and angles are as expected for this type of system and compare favourably with those reported for other bipyridyl molecules (Nakatsu *et al.*, 1972; Troyanov *et al.*, 1989).

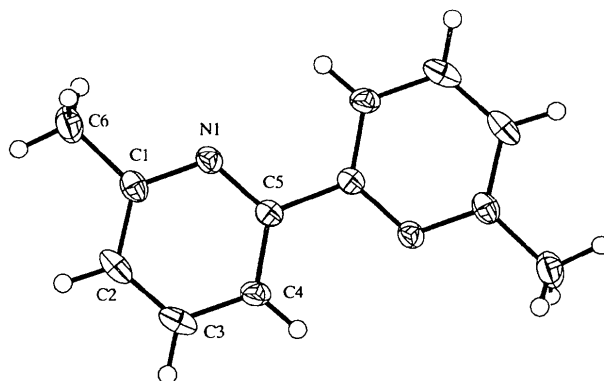


Fig. 1. The molecular structure of dmbp shown with 50% probability ellipsoids.

Experimental

The title compound was prepared according to the method of Badger & Sasse (1956, 1963), with modifications (Case, 1966; Burstal, 1938; Parks *et al.*, 1973; Newcome *et al.*, 1981; Rodde & Breitmaier, 1987) in order to improve yield. 2-Picoline (Aldrich) was refluxed (96 h) over freshly degassed Raney-nickel catalyst (dried under vacuum for 3 h) using a Soxhlet apparatus. NaOH was added to the alloy at 353–363 K over a period of 15 min. After removal of unreacted 2-picoline by distillation, the crude product was dissolved in ethanol, heated to boiling point and filtered over decolourising charcoal. The resulting yellow solution was evaporated to dryness and sublimed at 373 K. Recrystallization from ethanol produced clear prismatic crystals, which were characterized by NMR using a Bruker AMX360 ($DMSO-d_6$); δ 2.52 (s, py-CH₃, 6H), 7.25 (d, 5,5'-py-H, $J = 7.5$ Hz, 2H), 7.76 (t, 4,4'-py-H, $J = 7.7$ Hz, 2H) and 8.14 (d, 3,3'-py-H, $J = 7.8$ Hz, 2H).

Crystal data

C₁₂H₁₂N₂M_r = 184.24

Monoclinic

P2₁/c

a = 6.775 (5) Å

b = 11.146 (3) Å

c = 7.055 (4) Å

β = 111.72 (6)°

V = 494.9 (4) Å³

Z = 2

D_x = 1.241 Mg m⁻³D_m not measured

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 50 reflections

θ = 3.24–24.89°

μ = 0.075 mm⁻¹

T = 150 (2) K

Prism

0.35 × 0.32 × 0.11 mm

Colourless

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1167). Services for accessing these data are described at the back of the journal.

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Data collection

Delft Instruments FAST-TV area-detector diffractometer

Scan method: Darr *et al.* (1993)

Absorption correction: none
 1612 measured reflections
 742 independent reflections

613 reflections with

I > 2σ(I)

R_{int} = 0.100θ_{max} = 24.89°

h = -7 → 8

k = -12 → 6

l = -8 → 7

Refinement

Refinement on F²R[F² > 2σ(F²)] = 0.051wR(F²) = 0.135

S = 1.065

742 reflections

65 parameters

H atoms riding

w = 1/[σ²(F_o²) + (0.0766P)²]where P = (F_o² + 2F_c²)/3(Δ/σ)_{max} = 0.010Δρ_{max} = 0.213 e Å⁻³Δρ_{min} = -0.266 e Å⁻³

Extinction correction: none

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C1	1.338 (2)	C2—C3	1.383 (3)
N1—C5	1.353 (2)	C3—C4	1.377 (2)
C1—C2	1.390 (3)	C4—C5	1.386 (2)
C1—C6	1.503 (3)	C5—C5 ⁱ	1.485 (3)
C1—N1—C5	118.37 (15)	C4—C3—C2	118.94 (18)
N1—C1—C2	122.28 (16)	C3—C4—C5	119.26 (16)
N1—C1—C6	116.12 (18)	N1—C5—C4	122.08 (15)
C2—C1—C6	121.60 (17)	N1—C5—C5 ⁱ	116.50 (18)
C3—C2—C1	119.06 (17)	C4—C5—C5 ⁱ	121.41 (17)

Symmetry code: (i) -x, 2 - y, -z.

The absence of any intensity decay was confirmed by comparing intensities of equivalent reflections at the beginning and end of data collection. The methyl groups were allowed torsional freedom during the course of the refinement. H atoms were included in calculated positions (riding model), with U_{iso} set at 1.2 (CH) and 1.5 (CH₃) times the U_{eq} of the parent atoms.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1989). Cell refinement: *REFINE* in *MADNES*. Data reduction: *ABSMAD* (Karaulov, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SNOOPI* (Davies, 1983). Software used to prepare material for publication: *SHELXL93*.

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5,15-Bis(3,5-di-*tert*-butylphenyl)-10,20-bis-(trimethylsilylethynyl)porphyrin

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Abstract

In the title compound, C₅₈H₇₀N₄Si₂, the acetylene groups lie in the plane of the porphyrin π system. The angle between the plane of each phenyl ring and the plane of the porphyrin is 62.39 (4)°. There are no π–π

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