Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,6-Bis[1-(phenylimino)ethyl]pyridine

Ayfer Mentes,[a] John Fawcett[b]* and Raymond D. W. Kemmitt[b]

[a]Department of Chemistry, Art and Science Faculty, Zonguldak Karaelmas University, Zonguldak 67100, Turkey, and [b]Department of Chemistry, University of Leicester, Leicester LE1 7RH, England

Correspondence e-mail: jxf@leicester.ac.uk

Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.151
Data-to-parameter ratio = 8.4

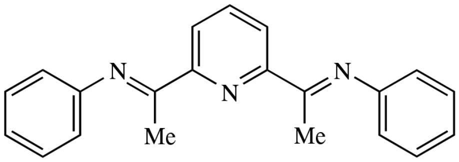
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_{21}H_{19}N_3$ (BIP1), has been determined. The molecule lies on a crystallographic mirror plane. The terminal phenyl groups are rotated 60.2 (2)° from the plane of the ethylidenepyridine system.

Received 28 March 2001
Accepted 5 April 2001
Online 12 April 2001

Comment

The title compound, (I) or BIP1, was previously described (not a crystal structure dteremination; Blake *et al.*, 1989). Platinum complexes of the type *fac*-[PtXMe$_3$(BIP1)] (X = Cl, Br, I) have been recently reported (Orrell *et al.*, 1998) and the crystal structure of the iodine derivative, *fac*-[PtIMe$_3$(BIP1)], was determined to confirm the proposed stereochemistry.



(I)

Pentacoordinate complexes of iron, [{[ArN=C(Me)]$_2$-C$_5$H$_3$N}FeCl$_2$] (Ar = alkyl substituted aryl ring), have been used for the isospecific polymerization of propylene (Small & Brookhart, 1999).

Our interest in tetracarbonyl complexes of molybdenum and tungsten with chelating nitrogen-donor ligands (Mentes, 1999; Mentes *et al.*, 1999) lead us to investigate *in situ* reactions of 2,6-diacetylpyridine with aniline in the presence of [Mo(CO)$_4$(pip)$_2$] (pip is piperidine), from which reaction crystals of BIP1 were obtained.

The molecular structure of BIP1 is shown in Fig. 1 and selected bond distances and angles are given in Table 1. Atoms C1 and N1 lie on a crystallographic mirror plane which is orthogonal to the ring. The terminal phenyl groups are rotated by 60.2 (2)° from the plane of the ethylidenepyridine system. The bond length of the symmetric imine group (C4—N2) of 1.266 (4) Å is similar to that in *fac*-[PtIMe$_3$(BIP1)], 1.277 (8) (coordinated to Pt) and 1.291 (9) Å (uncoordinated), and also in [FeCl$_2$[(BIP2)] {BIP2 is 2-[1-(2-*tert*-butylphenylimino)ethyl]-6-[1-(2,6-diisopropylphenylimino)ethyl]pyridine} 1.273 (3) and 1.284 (3) Å. The C=N bond lengths in [Mo(CO)$_4${C$_5$H$_4$NC(CH$_3$)=NCH$_3$}] and [Mo(CO)$_4${C$_5$H$_4$-NC(C$_6$H$_5$)=NCH$_3$}] are 1.289 (5) and 1.283 (4) Å, respectively.

Experimental

The Schiff base compound (BIP1) was isolated by refluxing 2,6-diacetylpyridine (0.489 g, 3 mmol) and excess of aniline (5 ml,

DOI: 101107/S1600536801005943

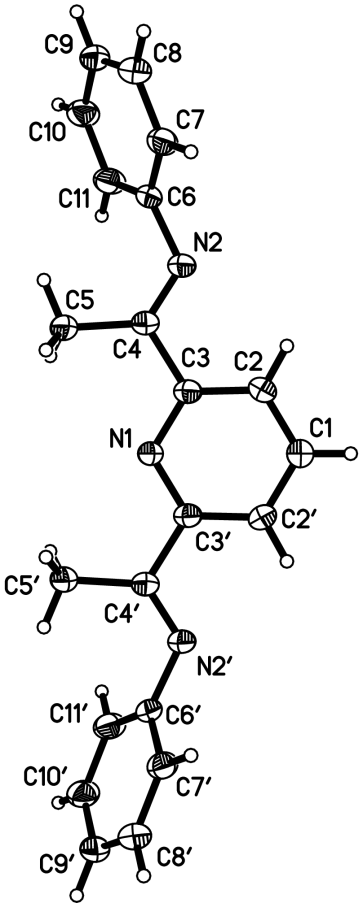


Figure 1

The molecular structure of BIP1 showing the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level. H atoms are shown as spheres of arbitrary radii. Primed atoms are generated by the symmetry operation $-x, y, z$.

55 mmol) in propan-2-ol (10 ml) for 2 h. *cis*-[Mo(CO)$_4$(pip)$_2$] (1.134 g, 3 mmol) was added to the reaction. The reaction mixture was cooled to room temperature and the solvent removed by rotary evaporation. The intermediate (BIP1) was isolated and recrystallized as pale-yellow crystals from an acetone–methanol (50:50) mixture.

Crystal data

$C_{21}H_{19}N_3$
$M_r = 313.39$
Orthorhombic, *Cmc*2$_1$
$a = 33.805$ (5) Å
$b = 7.1247$ (9) Å
$c = 7.2074$ (10) Å
$V = 1735.9$ (4) Å^3
$Z = 4$
$D_x = 1.199$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 33 reflections
$\theta = 5.3$–12.4°
$\mu = 0.07$ mm^{-1}
$T = 290$ (2) K
Block, yellow
0.46 × 0.39 × 0.31 mm

Data collection

Bruker *P*4 diffractometer
ω scans
1096 measured reflections
849 independent reflections
663 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\rm max} = 25.0°$

$h = -1 \rightarrow 40$
$k = 0 \rightarrow 8$
$l = -8 \rightarrow 1$
3 standard reflections
every 100 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.151$
$S = 1.07$
849 reflections
101 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.9470P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0054 (19)

Table 1

Selected bond lengths (Å).

N1—C3	1.340 (4)	C3—C4	1.497 (5)
N2—C4	1.266 (4)	C4—C5	1.504 (7)
N2—C6	1.424 (3)		

The absolute configuration could not be determined from the Flack parameter and the structure was refined using merged Friedel pairs. Since there were insufficient data to refine all atom positional and anisotropic displacement parameters, the phenyl ring was refined as a rigid group with C—C bond distances and C—C—C idealized.

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

References

Blake, A. J., Lavery, A. J., Hyde, T. J. & Schroder, M. (1989). *J. Chem. Soc. Dalton Trans.* pp. 965–970.
Fait, J. (1991). *XSCANS User's Manual.* Siemens Analytical X-ray instruments Inc., Madison, Wisconsin, USA.
Mentes, A. (1999). *Transition Met. Chem.* **24**, 77–80.
Mentes, A., Fawcett, J., Kemmitt, R. D. W. & Russell, D. R. (1999). *Acta Cryst.* C**55**, 48–50.
Orrell, K. G., Osborne, A. G., Eik, V., Silva, M. W., Hursthouse, D. E., Malik, K. M. A. & Vassilev, N. G. (1998). *J. Organomet. Chem.* **555**, 35–47.
Small, B. L. & Brookhart, M. (1999). *Macromolecules*, **32**, 2120–2130.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.