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Tetracarbonyl{2-[1-(2,6-dimethylphenylimino)ethyl]pyridine-κ^2N,N'}molybdenum(0)

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The title compound, $[Mo(C_{15}H_{16}N_2)(CO)_4]$, was obtained by a one-pot reaction of $Mo(CO)_6$ with 2-acetylpyridine and 2,6-dimethylaniline in ethanol. The complex exhibits a slightly distorted octahedral geometry.

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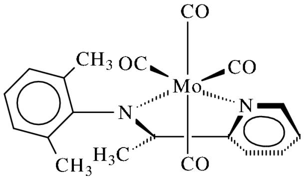
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.060
Data-to-parameter ratio = 15.6

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

Comment

Tetracarbonylmolybdenum complexes with Schiff base ligands form an important class of organometallic species (Mentes, 1999; Mentes *et al.*, 2002; Ali & Burgess, 1993). The spectroscopy, photophysics and photochemistry of α-diimine zerovalent metal complexes have already been reviewed (Stufkens, 1990). α-Diimine metal complexes have been used as catalysts for olefin polymerization (Ittel *et al.*, 2000; Haddleton *et al.*, 1999). The X-ray structures of complexes analogous to the title compound, (I), have been reported (Mentes *et al.*, 1999; Burgess *et al.*, 2000). This paper deals with the crystal structure of a tetracarbonylmolybdenum(0) Schiff base compound, (I), which was grown from tetrahydrofuran (THF) and diethyl ether at room temperature.



(I)

An *ORTEP*-3 (Farrugia, 1997) view of (I) and the atom-numbering scheme are shown in Fig. 1 and selected bond distances and angles are listed in Table 1. In (I), the Mo—C bond lengths of the two *cis* carbonyl groups [Mo—C17 = 1.958 (2) Å and Mo—C19 = 1.965 (2) Å], *trans* to nitrogen in the complex are shorter than those of the two carbonyl groups

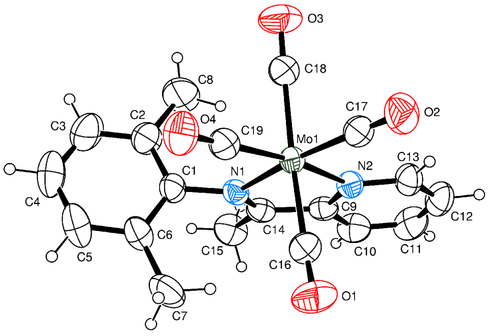


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

[Mo—C16 = 2.047 (2) Å and Mo—C18 = 2.019 (2) Å] which are *trans* to each other, since the nitrogen-donor ligand is a poorer π-acceptor than the carbonyl ligand. There are also two distinct Mo—N bonds in the compound, that to the pyridine N atom and that to the amine N atom [Mo—N2 = 2.2328 (16) Å and Mo—N1 = 2.2380 (19) Å, respectively]. In related complexes, the Mo—N2 bond distance is generally longer than the Mo—N1 bond distance (Burgess *et al.*, 2000) but, in compound (I), the difference, Δ [Δ = (Mo—N_{py})–(Mo—N_{amine})], between the two bond distances is negative. For strongly coordinating Schiff bases Δ is positive and for weaker coordinating Schiff bases Δ is negative. In compound (I), the Schiff base derived from 2-acetylpyridine and 2,6-dimethylaniline, $\Delta = -0.0052$ compared to complexes of the Shiff base components, *viz*. 2-acetylpyridine and either 4-methoxyaniline or 3-fluoro-4-methoxyaniline, in which $\Delta = +0.007$ and $+0.010$, respectively (Burgess *et al.*, 2000). In (I), the chelate bite angle (N1—Mo—N2) subtended at the molybdenum by the Schiff base diimine ligand is 71.44 (7)°. This value compares well with those in the literature for molybdenum(0) complexes (Burgess *et al.*, 2000; Mentes *et al.*, 1999).

Experimental

$Mo(CO)_6$ (0.5 g, 1.89 mmol) with 2-acetylpyridine (0.230 g, 1.9 mmol) and 2,6-dimethylaniline (0.230 g, 1.9 mmol) were refluxed in ethanol (15 ml) for 4 h. The solvent was removed from the dark-violet solution *in vacuo*, and a solid was obtained from a dichloromethane–diethyl ether solution. Dark-violet crystals were obtained by slow evaporation of a THF/diethyl ether solution (yield 61%; m.p. 513 K).

Crystal data

$[Mo(C_{15}H_{16}N_2)(CO)_4]$
$M_r = 432.28$
Triclinic, $P\overline{1}$
$a = 8.8736$ (6) Å
$b = 9.6117$ (7) Å
$c = 12.5601$ (9) Å
$\alpha = 69.480$ (5)°
$\beta = 81.120$ (6)°
$\gamma = 68.915$ (5)°
$V = 935.62$ (11) Å^3
$Z = 2$
$D_x = 1.534$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 16442 reflections
$\theta = 1.7$–29.3°
$\mu = 0.73$ mm^{-1}
$T = 293$ (2) K
Plate, dark violet
0.40 × 0.30 × 0.09 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.786$, $T_{max} = 0.940$
13577 measured reflections
3670 independent reflections
3301 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 26.0°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.060$
$S = 1.05$
3670 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.1218P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.438 (3)	C17—Mo1	1.958 (2)
C9—N2	1.346 (3)	C18—Mo1	2.019 (2)
C13—N2	1.339 (3)	C19—Mo1	1.965 (2)
C14—N1	1.285 (3)	N1—Mo1	2.2380 (17)
C16—Mo1	2.047 (2)	N2—Mo1	2.2328 (16)
C17—Mo1—N1	170.34 (7)	N2—Mo1—N1	71.44 (6)

All H atoms attached to C atoms were refined using the riding-model approximation; C—H = 0.93 Å for aromatic C atoms and C—H = 0.97 Å for methyl H atoms. The U_{iso} values for these H atoms were set at $1.2U_{eq}$ ($1.5U_{eq}$ for methyl) of the parent atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Ali, B. R. & Burgess, J. (1993). *Transition Met. Chem.* **18**, 9–18.
Burgess, J., Fawcett, J., Russell, D. R., Sawbridge, J. & Vu, H. (2000). *Transition Met. Chem.* **25**, 547–551.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Haddleton, D. M., Crossman, M. C., Dana, B. H., Duncalf, D. J., Heming, A. M., Kukulj, D. & Shooter, A. J. (1999). *Macromolecules*, **32**, 2110–2119.
Ittel, S. D., Johnson, L. K. & Brookhart, M. (2000). *Chem. Rev.* **100**, 1169–1204.
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.
Mentes, A., Kemmitt, R. D. W., Fawcett, J. & Russell, D. R. (2002). *J. Organomet. Chem.* **660**, 91–97.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen. Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED*32 (Version 1.04). Stoe &Cie, Darmstadt, Germany.
Stufkens, D. J. (1990). *Coord. Chem. Rev.* **104**, 39–112.