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Pentacarbonyl(di-2-pyridylamine)-tungsten(0)

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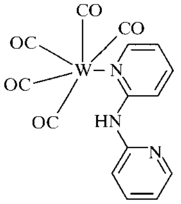
In the title molecular complex, (I), the W atom is in an octahedral environment with four equatorial carbonyl ligands and a fifth in an axial position *trans* to the monodentate dipyridylamine ligand. The long dimension of this last bisects the angle between two of the equatorial carbonyl groups and while the non-bonded pyridyl N atom is directed away from the W atom, the bridging amine group is directed towards it. Thus, in addition to the N atom to which it is attached, the amino H has two nearest neighbour C atoms of equatorial carbonyl groups but does not participate in hydrogen bonding in any real or usual sense. The W—C bond distance for the axial carbonyl group is notably less than those of the equatorial groups.

Comment

For the most part, the bond distances and angles of the title compound, (I), are in no way remarkable. On the other hand, W1—C5 of the axial carbonyl group [1.967(7) Å] is appreciably shorter than the W—C bond lengths of the equatorial groups which are in the range 2.020 (7)–2.063 (8) Å. There is, however, no comparable distinction in the carbonyl C—O bond lengths. The orientation of the dipyridylamine ligand at 90 ° to the equatorial plane of the complex with the non-bonding pyridyl N directed away from the metal atom and the bridging NH group directed towards it is most likely determined by steric effects. The long dimension of the dipyridyl ligand bisects the angle between the C3—O3 and C4—O4 equatorial carbonyl groups. As a result, in addition to the Natom to which it is attached, the amino H atom has two nearest neighbour non-H atoms [C3 and C4 at 2.30 (6) and 2.78 (6) Å respectively]. For convenience, the molecular geometry in the vicinity of the amino H atom is reported in the manner appropriate to the description of hydrogen bonds, but the distances and angles reported in this way suggest that hydrogen bonding in any real or usual sense is entirely absent.



(I)

Experimental

The title compound was prepared by the addition of dipyridylamine to a solution of [W(CO)$_5$(thf)] in tetrahydrofuran, followed by removal of the solvent under reduced pressure. The resulting yellow solid was recrystallized from deaerated toluene. [W(CO)$_5$(thf)] was prepared by photolysis of W(CO)$_6$ in argon-purged tetrahydrofuran.

Crystal data

[W(C$_{10}$H$_9$N$_3$)(CO)$_5$]	D_x = 2.032 Mg m^{-3}
M_r = 495.10	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 14 reflections
a = 12.812 (11) Å	θ = 10.20–12.45°
b = 7.185 (7) Å	μ = 7.166 mm^{-1}
c = 17.835 (15) Å	T = 298 (2) K
β = 99.61 (7)°	Block, green–yellow
V = 1619 (3) Å^3	0.52 × 0.26 × 0.26 mm
Z = 4	

Data collection

Nicolet $P3$ diffractometer	$R_{\rm int}$ = 0.041
θ–2θ scans	$\theta_{\rm max}$ = 27.56°
Absorption correction: ψ scan (North *et al.*, 1968)	h = 0 → 16
$T_{\rm min}$ = 0.071, $T_{\rm max}$ = 0.151	k = 0 → 9
3904 measured reflections	l = −23 → 22
3748 independent reflections	2 standard reflections every 50 reflections random variation: ±2%
2799 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.3862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{\rm max}$ = 0.001
$wR(F^2)$ = 0.082	$\Delta\rho_{\rm max}$ = 0.99 e Å^{-3}
S = 1.016	$\Delta\rho_{\rm min}$ = −0.60 e Å^{-3}
3748 reflections	Extinction correction: *SHELXL*97
222 parameters	Extinction coefficient: 0.0038 (2)
H atoms treated by a mixture of independent and constrained refinement	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···C3	0.92 (6)	2.30 (6)	3.154 (8)	154 (5)
N1—H1···C4	0.92 (6)	2.78 (6)	3.317 (9)	119 (5)

H atoms were initially placed in calculated positions and while H1 of the amino group was refined isotropically in the normal manner, the remaining H atoms were refined with a riding model.

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Data collection: *Nicolet P*3 *Software* (Nicolet, 1980); cell refinement: *Nicolet P*3 *Software* (Nicolet, 1980); data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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