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Pentacarbonyl(*N*,*N*-dimethylbenzylamine)-tungsten

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The title compound, $[W(C_9H_{13}N)(CO)_5]$, was prepared by irradiation of $W(CO)_6$ in tetrahydrofuran in the presence of *N*,*N*-dimethylbenzylamine. The geometry at the W atom is approximately octahedral, with the *cis* bond angles in the range 86.3 (3)–95.6 (2)°. The bond to the tertiary amine is long [2.371 (5) Å] and, as might be expected, the bond to the *trans* carbonyl is quite short [W—C = 1.964 (7) Å]. The remaining W—CO bonds lie in the range 2.033 (6)–2.049 (6) Å. Similar bonding patterns have been observed in related $W(CO)_5$(amine) complexes,

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.011 Å
R factor = 0.033
wR factor = 0.074
Data-to-parameter ratio = 20.2

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

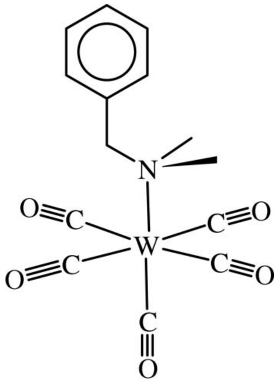
Comment

(*N*,*N*-Dimethylbenzylamine)pentacarbonyltungsten was prepared by irradiation of $W(CO)_6$ in tetrahydrofuran (THF) in the presence of the amine. Presumably, the reaction proceeds *via* an intermediate THF complex (Aroney *et al.*, 1994, and references therein):

$$W(CO)_6 + THF \rightarrow W(CO)_5(THF) + CO$$

$$W(CO)_5(THF) + C_9H_{13}N \rightarrow W(CO)_5(C_9H_{13}N) + THF.$$

Although a number of cyclometallated complexes of tungsten with this ligand have been reported previously (van der Schaaf *et al.*, 1993), no carbonyl complex has been structurally characterized. Also, in our hands, no cyclometallated complex was isolated.



(I)

The structure of (*N*,*N*-dimethylbenzylamine)pentacarbonyltungsten, (I), is shown in Fig. 1. The geometry at the W atom is approximately octahedral, with the *cis* bond angles in the range 86.3 (3)–95.6 (2)°. The bond to the tertiary amine is long [2.371 (5) Å] and, as might be expected, the bond to the *trans* carbonyl is quite short [W1—C10 = 1.964 (7) Å]. The remaining W—CO bonds lie in the range 2.033 (6)–

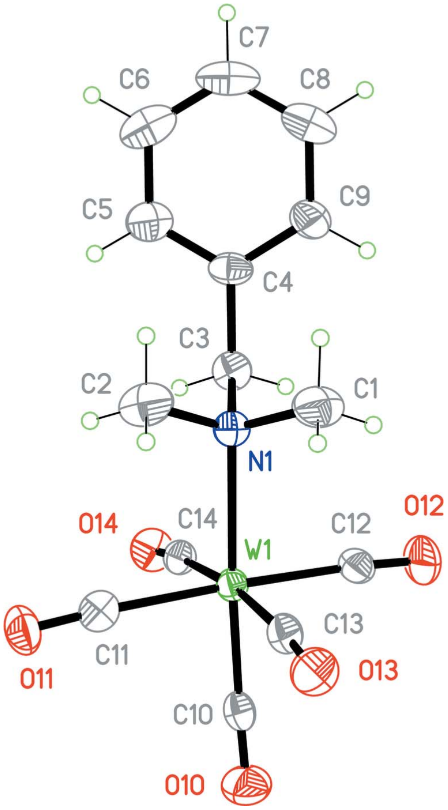


Figure 1
Perspective view of the the complex, with displacement ellipsoids drawn at the 50% probability level.

2.049 (6) Å. Similar bonding patterns have been observed in related *W*(CO)$_5$(amine) complexes [see, for example, Long *et al.* (2002) and Moralejo *et al.* (1991)]. There are no obvious π–π or edge-to-face interactions.

Experimental

W(CO)$_6$ (0.351 g, 1.0 mmol) and *N*,*N*-dimethylbenzylamine (0.30 ml, 2.0 mmol) were dissolved in sodium-dried THF (20 ml). The mixture was stirred under N$_2$ and irradiated with UV light for 4 h, yielding a yellow solution. The progress of the reaction was monitored by following the CO stretching band at 1975 cm^{-1} by IR. The volume of the solvent was reduced under vacuum and n-hexane added to induce crystallization (yield 0.078 g, 17%). The sample was not pure and did not give satisfactory microanalysis. The EI mass spectrum of the complex showed a cluster corresponding to the parent ion W(C$_6$H$_5$CH$_2$N(CH$_3$)$_2$)(CO)$_5$ centered at m/e 459 and the isotope pattern matched that predicted from theory. Clusters corresponding to sequential loss of CO groups were observed at m/e of 431 [W(C$_6$H$_5$CH$_2$N(CH$_3$)$_2$)(CO)$_4$], 403 [W(C$_6$H$_5$CH$_2$N(CH$_3$)$_2$(CO)$_3$] and 375 [W(C$_6$H$_5$CH$_2$N(CH$_3$)$_2$)(CO)$_2$]. Clusters at m/e 345, 317 and 135 were assigned to W(C$_6$H$_5$CH$_2$N)(CO)$_2$, W(C$_6$H$_5$CH$_2$N)(CO) and [C$_6$H$_5$CH$_2$N(CH$_3$)$_2$ + H$^+$], respectively. Clusters corresponding to W(CO)$_6$, W(CO)$_5$, W(CO)$_4$, W(CO)$_3$, W(CO)$_2$, W(CO) and W were also observed. The W(CO)$_6$ was most likely present as an impurity in the sample. ^{1}H NMR (CDCl$_3$): 2.78 (*s*, 6H, CH$_3$), 4.22 (*s*, 2H, CH$_2$), 7.25–7.34 (*m*, 5H, aromatic). ^{13}C NMR: 55.3 (CH$_3$), 73.6 (CH$_2$), 128.5 (aromatic C$_3$, C$_5$), 129.0 (aromatic C$_4$), 132.0 (aromatic, C$_2$, C$_6$), 191, 199, 202 (carbonyl). IR (KBr, cm^{-1}): 3425 (*m*), 1952 (*m*), 1060 (w), 932 (*m*), 853 (*m*) 774 (*m*), 592 (*s*).

Crystal data

[W(C$_9$H$_{13}$N)(CO)$_5$]
M_r = 459.10
Orthorhombic, *Pbca*
a = 13.7829 (11) Å
b = 12.5247 (10) Å
c = 18.2985 (14) Å
V = 3158.8 (4) Å^3
Z = 8
D_x = 1.931 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 4675 reflections
θ = 2.5–28.1°
μ = 7.33 mm^{-1}
T = 150 (2) K
Plate, yellow
0.28 × 0.20 × 0.05 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω rotation with narrow-frame scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min}$ = 0.233, $T_{\max}$ = 0.711
18147 measured reflections

3870 independent reflections
2534 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.059
$\theta_{\max}$ = 29.0°
$h = -13 \rightarrow 18$
$k = -16 \rightarrow 16$
$l = -24 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.074
S = 1.03
3870 reflections
192 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0125P)^2 + 14.4005P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 1.10 e Å^{-3}
$\Delta\rho_{\min}$ = −0.85 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

W1—N1	2.371 (5)	O11—C11	1.144 (8)
W1—C10	1.964 (7)	O12—C12	1.152 (8)
W1—C11	2.041 (6)	O13—C13	1.156 (7)
W1—C12	2.034 (6)	O14—C14	1.142 (7)
W1—C13	2.033 (6)	N1—C1	1.492 (9)
W1—C14	2.048 (6)	N1—C2	1.496 (9)
O10—C10	1.157 (9)	N1—C3	1.514 (8)
N1—W1—C10	176.8 (2)	C13—W1—C14	172.6 (3)
N1—W1—C11	93.9 (2)	W1—N1—C1	110.0 (4)
N1—W1—C12	89.8 (2)	W1—N1—C2	111.7 (4)
N1—W1—C13	91.5 (2)	W1—N1—C3	109.1 (4)
N1—W1—C14	95.6 (2)	C1—N1—C2	107.4 (5)
C10—W1—C11	88.4 (3)	C1—N1—C3	109.3 (5)
C10—W1—C12	88.0 (3)	C2—N1—C3	109.3 (5)
C10—W1—C13	86.3 (3)	N1—C3—C4	115.8 (6)
C10—W1—C14	86.7 (3)	W1—C10—O10	177.2 (6)
C11—W1—C12	174.8 (3)	W1—C11—O11	176.4 (6)
C11—W1—C13	90.5 (2)	W1—C12—O12	174.0 (6)
C11—W1—C14	86.8 (2)	W1—C13—O13	173.9 (6)
C12—W1—C13	93.1 (3)	W1—C14—O14	174.5 (5)
C12—W1—C14	89.1 (3)		

H atoms bonded to C atoms were inserted at calculated positions and refined using a riding model. The constrained C—H distances were 0.95, 0.98 and 0.99 Å for aryl, methyl, and methylene H atoms, respectively. The H atoms of methylene and aryl groups were refined with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) and those of the methyl groups with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). The highest residual electron-density peak is 0.88 Å from the W atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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