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Pentacarbonyl{*N*,*N*-dimethyl-*N*′-[4-(pyridin-4-yl-ethynyl-κ*N*)benzylidene]benzene-1,4-diamine}-tungsten(0)

Ping-Hsin Huang[a,b]* and Yuh-Sheng Wen[a]

[a]Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan 115, and [b]Kang-Ning Junior College of Medical Care and Management, Taipei, Taiwan 114

Correspondence e-mail:
pshuang@webmail.knjc.edu.tw

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Key indicators

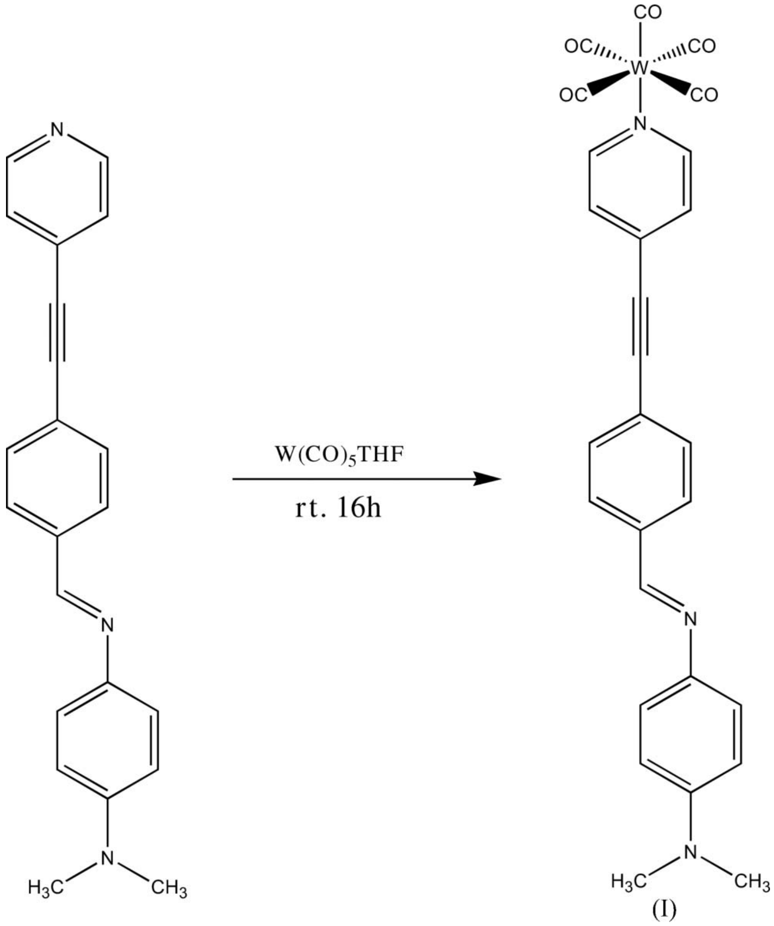
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.005 Å
R factor = 0.022
wR factor = 0.049
Data-to-parameter ratio = 13.9

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

The asymmetric unit of the title compound, [W($C_{22}H_{19}N_3$)-($(CO)_5$], contains two molecules. The geometry at the W atom is approximately octahedral, with the *cis* bond angles in the range 86.11 (13)–92.75 (11)°. The axial carbonyl bonds [W—C = 1.960 (4) and 1.967 (4) Å] are shorter than the equatorial carbonyl bonds, which lie in the range 2.014 (4)–2.047 (4) Å.

Comment

There is widespread interest in π-conjugated molecular frameworks because these materials may be used as molecular wires (Woitellier, 1989) and opto-electronic devices. The title compound, (I), containing a tungsten carbonyl unit as an electron acceptor with an end-capping organic electron donor and benzene ring in the conjugation chain, has great potential as an opto-electronic material (McCullough, 1998).



(I)

The structures of the two indepedent molecules of (I) are shown in Fig. 1. The W atom occupies the center of a slightly distorted octahedral environment, bound to five carbonyl groups and one *N*,*N*-dimethyl-*N*′-[4-(pyridin-4-ylethynyl-*κN*)benzylidene]benzene-1,4-diamine ligand.

The bond distances and angles involving the W atoms are listed in Table 1. The W—C$_{axial}$ bonds are shorter [W—C = 1.960 (4) and 1.967 (4) Å] than the equatorial W—CO bonds which are in the range 2.014 (4)–2.042 (3) [angles 86.11 (13)–92.75 (11)°]. The dihedral angle between the C19–C24 and N11/C12–C16 rings is 23.58 (1)° and that between the N11/C12–C16 and C26–C31 rings is 2.21 (1)° [10.03 (1) and 36.91 (1)° for the other molecue]. The two benzene rings form a dihedral angle of 21.65 (1)° [46.80 (1)° for the other molecule]. No significant hydrogen-bonding interactions are observed in the crystal structure.

Experimental

A tetrahydrofuran (THF) solution (100 ml) of W(CO)$_5$(THF) prepared from W(CO)$_6$ (100 mg, 0.28 mmol) was transferred to a flask containing *N*,*N*-dimethyl-N′-[4-(pyridin-4-ylethynyl-*κN*)benzylidene]benzene-1,4-diamine (78 mg, 0.24 mmol). The solution was stirred at room temperature for 16 h, and the solvent was removed under vacuum. The residue was chromatographed through silica gel using a THF/hexane (1:10) mixture as eluant. The compound was obtained as a red solid in 48% yield. FAB–MS: *m*/*e* 650 $(M + 1)^+$. Analysis calculated for $C_{27}H_{19}N_3O_5W$: C 49.94, H 2.95, N 6.47%; found: C 49.50, H 2.88, N 6.38%.

Crystal data

$[W(C_{22}H_{19}N_3)(CO)_5]$
$M_r = 649.30$
Triclinic, $P\overline{1}$
$a = 12.2258$ (2) Å
$b = 12.5046$ (2) Å
$c = 17.3157$ (3) Å
$\alpha = 102.595$ (1)°
$\beta = 95.362$ (1)°
$\gamma = 93.249$ (1)°
$V = 2564.09$ (7) Å^3
$Z = 4$
$D_x = 1.682$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 4.55$ mm^{-1}
$T = 100.0$ (1) K
Plate, red
0.3 × 0.25 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.493$, $T_{max} = 0.986$ (expected range = 0.348–0.695)
39209 measured reflections
9009 independent reflections
7221 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.049$
$S = 0.99$
9009 reflections
650 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0204P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.94$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00043 (5)

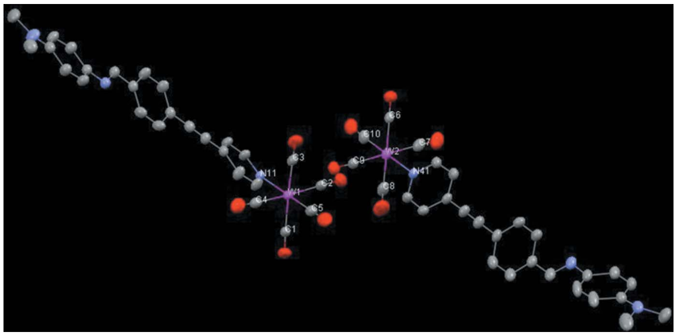


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme employed.

Table 1
Selected geometric parameters (Å, °).

W1—C5	1.960 (4)	W2—C10	1.967 (4)
W1—C3	2.014 (4)	W2—C8	2.028 (4)
W1—C4	2.032 (4)	W2—C7	2.035 (4)
W1—C2	2.037 (4)	W2—C9	2.037 (4)
W1—C1	2.042 (3)	W2—C6	2.047 (4)
W1—N11	2.273 (2)	W2—N41	2.267 (2)
C5—W1—C3	86.11 (13)	C4—W1—C1	91.34 (13)
C5—W1—C4	89.60 (14)	C2—W1—C1	91.86 (12)
C3—W1—C4	87.78 (14)	C5—W1—N11	177.46 (11)
C5—W1—C2	89.67 (14)	C3—W1—N11	92.18 (11)
C3—W1—C2	88.97 (13)	C4—W1—N11	88.46 (11)
C4—W1—C2	176.70 (12)	C2—W1—N11	92.18 (11)
C5—W1—C1	88.92 (13)	C1—W1—N11	92.75 (11)
C3—W1—C1	174.96 (12)		

H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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