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Pentacarbonyl[*N*,*N*-dimethyl-*N*′-(5-{2-[5-(4-pyridylethynyl)thiophen-2-yl]vinyl}thiophen-2-ylmethylene)benzene-1,4-diamine]tungsten(0)

Ping-Hsin Huang,[a,b,c]* Tai-Hsiang Huang,[b,c] Jiun-Yi Shen[b,c] and Yuh-Sheng Wen[b,c]

[a]Kang-Ning Junior College of Medical Care and Management, Taipei, Taiwan 114, [b]Institute of Chemistry, Academia Sinica, Taipei, Taiwan 115, and [c]Department of Chemistry, National Taiwan Normal University, Taipei, Taiwan

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

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.040
Data-to-parameter ratio = 13.8

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

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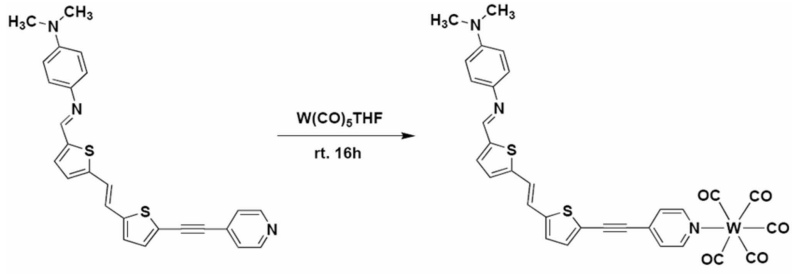
In the title compound, $[W(C_{26}H_{21}N_3S_2)(CO)_5]$, the geometry at the W atom is approximately octahedral, with the *cis* bond angles in the range 86.45 (13)–94.99 (12)°. The bond to the *trans* carbonyl is quite short [W—C = 1.953 (4) Å] and the equatorial W—CO bonds lie in the range 2.010 (4)–2.059 (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 a thienyl entity in the conjugation chain, has great potential as an opto-electronic material (McCullough, 1998).



(I)

The molecular structure of (I) is 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*′-(5-{2-[5-(4-pyridylethynyl)thiophen-2-yl]vinyl}-thiophen-2-ylmethylene)benzene-1,4-diamine ligand. The bond distances and angles involving the W atom are listed in Table 1. The bond to the *trans* carbonyl is quite short [W—C = 1.953 (4) Å] and the equatorial W—CO bonds lie in the range 2.010 (4)–2.059 (4) Å. The *cis* bond angles lie in the range 86.45 (13)–94.99 (12)°. The dihedral angle between the S1/C14–C17 and N6/C7–C11 rings is 7.3 (1)° and that between the S2/C20–C23 and C26–C31 rings is 54.3 (1)°. The two thiophene rings form a dihedral angle of 25.9 (1)°. 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 mixed with *N*,*N*-

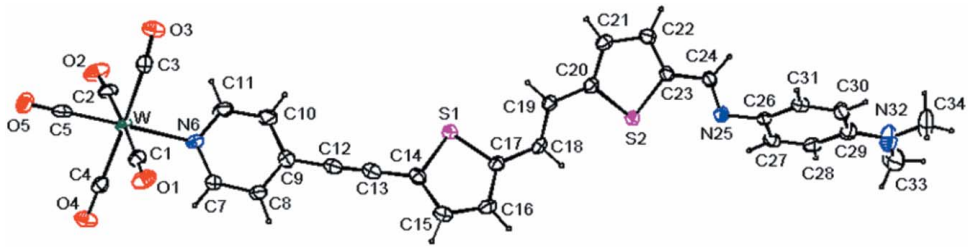


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as small spheres of arbitrary radii.

dimethyl-*N*′-(5-{2-[5-(4-pyridylethynyl)thiophen-2-yl]vinyl}thiophen-2-ylmethylene)benzene-1,4-diamine (PETTIA) (183 mg, 0.24 mmol). The resulting 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 *v*/*v*) mixture as eluant. The title compound was obtained as a red solid in 48% yield. FAB MS: *m*/*e* 763 $(M + 1)^+$. Analysis calculated for $C_{31}H_{21}N_3O_5S_2W$: C 48.72, H 2.77, N 5.50%; found: C 48.24, H 2.68, N 5.36%.

Crystal data

$[W(C_{26}H_{21}N_3S_2)(CO)_5]$
M_r = 763.48
Triclinic, $P\overline{1}$
a = 10.6767 (2) Å
b = 11.9258 (2) Å
c = 13.0623 (2) Å
α = 89.606 (1)°
β = 86.660 (1)°
γ = 63.538 (1)°
V = 1486.08 (5) Å^3
Z = 2
D_x = 1.706 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5729 reflections
θ = 2.4–29.3°
μ = 4.07 mm^{-1}
T = 100.0 (1) K
Prism, red
0.14 × 0.12 × 0.1 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.510, T_{max} = 0.666
10804 measured reflections
5246 independent reflections
4528 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 14$
$l = -13 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.040
S = 0.88
5246 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0123P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

W—C5	1.953 (4)	W—C3	2.048 (4)
W—C1	2.010 (4)	W—C2	2.059 (4)
W—C4	2.025 (3)	W—N6	2.256 (3)
C5—W—C1	89.52 (13)	C4—W—C2	93.54 (13)
C5—W—C4	88.73 (12)	C3—W—C2	88.82 (13)
C1—W—C4	86.45 (13)	C5—W—N6	176.66 (12)
C5—W—C3	87.63 (13)	C1—W—N6	87.36 (11)
C1—W—C3	91.04 (13)	C4—W—N6	92.27 (11)
C4—W—C3	175.60 (13)	C3—W—N6	91.23 (11)
C5—W—C2	88.13 (13)	C2—W—N6	94.99 (12)
C1—W—C2	177.65 (14)		

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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