ORGANOMETALLICS
Organometallics, 1998, 17(9), 1655-1662, DOI:10.1021/om9708992

X-ray Data Collection for $Tp'(CO)_2\overline{WCHCHC(O)Ph}$ (15).

A single crystal of **15** was mounted on a glass rod coated with epoxy. Diffraction data were collected on a Riggaku diffractometer. Centered reflections found in the region $30°<2T<40°$ were refined to indicate a monoclinic unit cell. Unit cell and collection parameters are given in Table 2.2. Only data with $I>2.5\sigma(I)$ were used in structure solution and refinement. The data were collected for Lorentz-polarization effects during the final stages of refinement.

Solution and Refinement of $Tp'(CO)_2\overline{WCHCHC(O)Ph}$ (15).

The space group C_c was confirmed and the position of the tungsten atoms were deduced from the three-dimensional Patterson functions. The positions of the remaining 36 non-hydrogen atoms were determined through subsequent Fourier and difference Fourier calculations. A molecule of CH_2Cl_2 per molecule of **15** was found in the unit cell. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were calculated by using a C-H distance of 0.96 Å and an isotropic thermal parameter calculated from the anisotropic values for the atoms to which they were attached. The final residuals were calculated using 174 parameters refined against 1824 data.

Supporting Information

Table 2.2. Crystallographic Data and collection parameters for $Tp'(CO)_2W\overline{CHCHC(O)Ph}$ (15).

formula	$BC_{26}H_{29}N_6O_3W \cdot CH_2Cl_2$
mol wt	753.95
cryst syst	Monoclinic
space group	C_c
a, Å	11.3968(22)
b, Å	18.403(7)
c, Å	14.5479(15)
β, deg	103.550(11)
V,	2966.3(13) Å^3
Z	4
D_{calc}, mg m^{-3}	1.688
F(000)	1486.71
cryst dimens, mm	0.50 x 0.32 x 0.08
temp, °C	-145
radiation,	MoKα(0.71073)
2θ range, deg	30<2θ<40
μ, mm^{-1}	4.20
scan mode	omega
total no. of data	1977
total no. of unique data	1974
no. of obs data (I>2.0σ(I))	1858
R_f	0.036
R_w	0.044
GoF	1.58

Table 2.3. Complete List of Bond Distances (Å) for Tp'(CO)$_2$WCHCHC(O)Ph (15).

W(1)-C(1)	1.984(12)	N(22)-C(23)	1.366(17)
W(1)-C(2)	1.928(14)	C(23)-C(24)	1.396(19)
W(1)-O(3)	2.031(9)	C(23)-C(26)	1.485(21)
W(1)-C(6)	2.117(14)	C(24)-C(25)	1.370(20)
W(1)-N(11)	2.208(9)	C(24)-H(24)	0.960(14)
W(1)-N(21)	2.243(11)	C(25)-C(27)	1.490(21)
W(1)-N(31)	2.233(11)	C(26)-H(26a)	0.960(15)
W(1)-H(6)	2.7555(11)	C(26)-H(26b)	0.960(15)
Cl(1)-Cl(2)	2.903(7)	C(26)-H(26c)	0.960(15)
Cl(1)-C(51)	1.855(18)	C(27)-H(27a)	0.960(15)
Cl(2)-C(51)	1.701(18)	C(27)-H(27b)	0.960(15)
Cl(2)-H(51a)	2.200(6)	C(27)-H(27c)	0.960(16)
Cl(2)-H(51b)	2.202(5)	N(31)-N(32)	1.377(15)
C(1)-O(1)	1.151(16)	N(31)-C(35)	1.328(18)
C(2)-O(2)	1.184(17)	N(32)-C(33)	1.341(18)
O(3)-C(4)	1.328(17)	C(33)-C(34)	1.398(19)
C(4)-C(5)	1.328(20)	C(33)-C(36)	1.483(21)
C(4)-C(41)	1.505(19)	C(34)-C(35)	1.410(20)
C(5)-C(6)	1.443(18)	C(34)-H(34)	0.960(13)
C(5)-H(5)	0.960(14)	C(35)-C(37)	1.482(22)
C(6)-H(6)	0.960(14)	C(36)-H(36a)	0.960(15)
B(1)-N(12)	1.537(19)	C(36)-H(36b)	0.960(14)
B(1)-N(22)	1.528(19)	C(36)-H(36c)	0.960(15)
B(1)-N(32)	1.571(19)	C(37)-H(37a)	0.960(16)
B(1)-H(1)	0.960(16)	C(37)-H(37b)	0.960(15)
N(11)-N(12)	1.391(15)	C(37)-H(37c)	0.960(15)
N(11)-C(15)	1.346(15)	C(41)-C(42)	1.385(21)
N(12)-C(13)	1.343(16)	C(41)-C(46)	1.394(21)
C(13)-C(14)	1.380(18)	C(42)-C(43)	1.408(21)
C(13)-C(16)	1.496(21)	C(42)-H(42)	0.960(15)
C(14)-C(15)	1.380(19)	C(43)-C(44)	1.420(22)

C(14)-H(14)	0.960(13)	C(43)-H(43)	0.960(16)
C(15)-C(17)	1.521(19)	C(44)-C(45)	1.349(22)
C(16)-H(16a)	0.960(14)	C(44)-H(44)	0.960(16)
C(16)-H(16b)	0.960(14)	C(45)-C(46)	1.408(21)
C(16)-H(16c)	0.960(14)	C(45)-H(45)	0.960(16)
C(17)-H(17a)	0.960(13)	C(46)-H(46)	0.960(15)
C(17)-H(17b)	0.960(13)	C(51)-H(51a)	0.960(17)
C(17)-H(17c)	0.960(14)	C(51)-H(51b)	0.960(18)
N(21)-N(22)	1.351(16)	H(51a)-H(51b)	1.54701(24)
N(21)-C(25)	1.349(18)		

Table 2.4. Complete List of Bond Angles (deg) for Tp'(CO)$_2$WCHCHC(O)Ph (15).

C(1)-W(1)-C(2)	97.8(6)	B(1)-N(22)-N(21)	120.3(11)
C(1)-W(1)-O(3)	115.6(4)	B(1)-N(22)-C(23)	129.2(12)
C(1)-W(1)-C(6)	67.7(5)	N(21)-N(22)-C(23)	110.3(10)
C(1)-W(1)-N(11)	161.2(4)	N(22)-C(23)-C(24)	105.8(12)
C(1)-W(1)-N(21)	90.2(5)	N(22)-C(23)-C(26)	125.4(12)
C(1)-W(1)-N(31)	80.3(5)	C(24)-C(23)-C(26)	128.8(13)
C(1)-W(1)-H(6)	57.6(4)	C(23)-C(24)-C(25)	107.2(12)
C(2)-W(1)-O(3)	112.0(5)	C(23)-C(24)-H(24)	126.3(15)
C(2)-W(1)-C(6)	67.3(5)	C(25)-C(24)-H(24)	126.5(14)
C(2)-W(1)-N(11)	86.4(5)	N(21)-C(25)-C(24)	109.3(12)
C(2)-W(1)-N(21)	161.6(5)	N(21)-C(25)-C(27)	122.0(12)
C(2)-W(1)-N(31)	82.0(5)	C(24)-C(25)-C(27)	128.6(13)
C(2)-W(1)-H(6)	59.5(4)	C(23)-C(26)-H(26a)	110.3(13)
O(3)-W(1)-C(6)	73.4(4)	C(23)-C(26)-H(26b)	108.9(14)
O(3)-W(1)-N(11)	79.1(3)	C(23)-C(26)-H(26c)	109.2(13)
O(3)-W(1)-N(21)	78.7(4)	H(26a)-C(26)-H(26b)	109.5(15)
O(3)-W(1)-N(31)	155.7(4)	H(26a)-C(26)-H(26c)	109.5(15)
O(3)-W(1)-H(6)	90.45(23)	H(26b)-C(26)-H(26c)	109.5(15)
C(6)-W(1)-N(11)	130.1(4)	C(25)-C(27)-H(27a)	110.0(15)
C(6)-W(1)-N(21)	131.0(4)	C(25)-C(27)-H(27b)	109.3(13)
C(6)-W(1)-N(31)	130.9(5)	C(25)-C(27)-H(27c)	109.1(14)
C(6)-W(1)-H(6)	17.1(4)	H(27a)-C(27)-H(27b)	109.5(15)
N(11)-W(1)-N(21)	81.0(4)	H(27a)-C(27)-H(27c)	109.5(14)
N(11)-W(1)-N(31)	82.2(4)	H(27b)-C(27)-H(27c)	109.5(16)
N(11)-W(1)-H(6)	137.56(24)	W(1)-N(31)-N(32)	119.5(8)
N(21)-W(1)-N(31)	83.1(4)	W(1)-N(31)-C(35)	133.3(10)
N(21)-W(1)-H(6)	137.5(3)	N(32)-N(31)-C(35)	106.9(11)
N(31)-W(1)-H(6)	113.9(3)	B(1)-N(32)-N(31)	120.6(10)
Cl(2)-Cl(1)-C(51)	33.6(6)	B(1)-N(32)-C(33)	128.4(11)
Cl(1)-Cl(2)-C(51)	37.1(6)	N(31)-N(32)-C(33)	110.9(10)
Cl(1)-Cl(2)-H(51a)	53.53(16)	N(32)-C(33)-C(34)	106.6(11)

Cl(1)-Cl(2)-H(51b)	53.49(14)	N(32)-C(33)-C(36)	123.7(12)
C(51)-Cl(2)-H(51a)	24.5(6)	C(34)-C(33)-C(36)	129.6(13)
C(51)-Cl(2)-H(51b)	24.4(6)	C(33)-C(34)-C(35)	106.1(12)
H(51a)-Cl(2)-H(51b)	41.15(10)	C(33)-C(34)-H(34)	126.6(14)
W(1)-C(1)-O(1)	173.6(12)	C(35)-C(34)-H(34)	127.2(14)
W(1)-C(2)-O(2)	179.7(11)	N(31)-C(35)-C(34)	109.3(12)
W(1)-O(3)-C(4)	122.3(8)	N(31)-C(35)-C(37)	125.9(13)
O(3)-C(4)-C(5)	115.1(12)	C(34)-C(35)-C(37)	124.4(13)
O(3)-C(4)-C(41)	116.6(12)	C(33)-C(36)-H(36a)	111.1(14)
C(5)-C(4)-C(41)	128.2(13)	C(33)-C(36)-H(36b)	108.8(13)
C(4)-C(5)-C(6)	113.9(13)	C(33)-C(36)-H(36c)	108.5(13)
C(4)-C(5)-H(5)	122.4(13)	H(36a)-C(36)-H(36b)	109.5(14)
C(6)-C(5)-H(5)	123.7(13)	H(36a)-C(36)-H(36c)	109.5(14)
W(1)-C(6)-C(5)	115.1(10)	H(36b)-C(36)-H(36c)	109.5(15)
W(1)-C(6)-H(6)	122.6(10)	C(35)-C(37)-H(37a)	109.2(14)
C(5)-C(6)-H(6)	122.3(13)	C(35)-C(37)-H(37b)	110.2(14)
N(12)-B(1)-N(22)	109.5(11)	C(35)-C(37)-H(37c)	109.0(14)
N(12)-B(1)-N(32)	108.1(11)	H(37a)-C(37)-H(37b)	109.5(15)
N(12)-B(1)-H(1)	109.9(13)	H(37a)-C(37)-H(37c)	109.5(15)
N(22)-B(1)-N(32)	109.6(11)	H(37b)-C(37)-H(37c)	109.5(16)
N(22)-B(1)-H(1)	110.5(14)	C(4)-C(41)-C(42)	118.8(13)
N(32)-B(1)-H(1)	109.2(13)	C(4)-C(41)-C(46)	119.2(13)
W(1)-N(11)-N(12)	121.9(7)	C(42)-C(41)-C(46)	122.0(13)
W(1)-N(11)-C(15)	132.7(8)	C(41)-C(42)-C(43)	119.6(14)
N(12)-N(11)-C(15)	105.3(10)	C(41)-C(42)-H(42)	120.4(14)
B(1)-N(12)-N(11)	118.4(10)	C(43)-C(42)-H(42)	119.9(15)
B(1)-N(12)-C(13)	131.1(11)	C(42)-C(43)-C(44)	117.6(14)
N(11)-N(12)-C(13)	110.4(10)	C(42)-C(43)-H(43)	121.2(15)
N(12)-C(13)-C(14)	107.2(12)	C(44)-C(43)-H(43)	121.1(15)
N(12)-C(13)-C(16)	123.1(11)	C(43)-C(44)-C(45)	122.1(15)
C(14)-C(13)-C(16)	129.6(12)	C(43)-C(44)-H(44)	118.6(15)
C(13)-C(14)-C(15)	106.7(12)	C(45)-C(44)-H(44)	119.3(15)
C(13)-C(14)-H(14)	126.9(13)	C(44)-C(45)-C(46)	120.6(15)
C(15)-C(14)-H(14)	126.4(13)	C(44)-C(45)-H(45)	119.5(15)

N(11)-C(15)-C(14)	110.3(12)	C(46)-C(45)-H(45)	119.8(16)
N(11)-C(15)-C(17)	121.7(12)	C(41)-C(46)-C(45)	118.1(14)
C(14)-C(15)-C(17)	128.0(12)	C(41)-C(46)-H(46)	120.3(14)
C(13)-C(16)-H(16a)	109.7(13)	C(45)-C(46)-H(46)	121.6(15)
C(13)-C(16)-H(16b)	109.1(12)	Cl(1)-C(51)-Cl(2)	109.4(10)
C(13)-C(16)-H(16c)	109.6(13)	Cl(1)-C(51)-H(51a)	111.6(13)
H(16a)-C(16)-H(16b)	109.5(15)	Cl(1)-C(51)-H(51b)	111.6(14)
H(16a)-C(16)-H(16c)	109.5(13)	Cl(2)-C(51)-H(51a)	108.3(14)
H(16b)-C(16)-H(16c)	109.5(14)	Cl(2)-C(51)-H(51b)	108.5(13)
C(15)-C(17)-H(17a)	109.8(12)	H(51a)-C(51)-H(51b)	107.4(17)
C(15)-C(17)-H(17b)	109.1(12)	W(1)-H(6)-C(6)	40.3(8)
C(15)-C(17)-H(17c)	109.5(12)	Cl(2)-H(51a)-C(51)	47.2(11)
H(17a)-C(17)-H(17b)	109.5(13)	Cl(2)-H(51a)-H(51b)	69.50(16)
H(17a)-C(17)-H(17c)	109.5(13)	C(51)-H(51a)-H(51b)	36.3(11)
H(17b)-C(17)-H(17c)	109.5(13)	Cl(2)-H(51b)-C(51)	47.1(10)
W(1)-N(21)-N(22)	120.9(8)	Cl(2)-H(51b)-H(51a)	69.36(18)
W(1)-N(21)-C(25)	131.8(9)	C(51)-H(51b)-H(51a)	36.3(10)
N(22)-N(21)-C(25)	107.3(10)		

Table 2.5. Atomic Parameters x, y, z, and Biso for

Tp'(CO)$_2$WCHCHC(O)Ph (15).

Name	x	y	z
W(1)	0.99150(0)	0.81195(2)	0.99684(0)
Cl(1)	0.53073(46)	0.91522(25)	0.20832(34)
Cl(2)	0.70580(57)	0.97086(28)	0.37116(38)
C(1)	0.83315(110)	0.85825(74)	0.99262(90)
O(1)	0.73674(86)	0.87864(61)	0.98932(70)
C(2)	1.09143(115)	0.88139(78)	1.07885(94)
O(2)	1.15326(83)	0.92379(56)	1.12935(68)
O(3)	1.03717(76)	0.80292(45)	0.87047(62)
C(4)	1.04604(120)	0.86028(80)	0.81712(96)
C(5)	1.01399(111)	0.92314(76)	0.84878(91)
C(6)	0.97946(112)	0.91760(77)	0.93786(91)
B(1)	1.01341(137)	0.64456(89)	1.08630(111)
N(11)	1.14691(77)	0.73714(52)	1.03431(64)
N(12)	1.13690(91)	0.66839(60)	1.07139(75)
C(13)	1.24153(108)	0.63204(73)	1.08260(87)
C(14)	1.32152(118)	0.67714(72)	1.05214(98)
C(15)	1.26148(114)	0.74160(76)	1.02512(93)
C(16)	1.25992(119)	0.55691(86)	1.12271(98)
C(17)	1.30969(120)	0.80997(71)	0.98814(98)
N(21)	0.89822(96)	0.70811(60)	0.94129(80)
N(22)	0.92033(90)	0.64592(61)	0.99177(73)
C(23)	0.85702(117)	0.58967(74)	0.94226(95)
C(24)	0.79041(119)	0.61983(81)	0.85808(97)
C(25)	0.81749(120)	0.69242(71)	0.85998(98)
C(26)	0.86249(131)	0.51280(87)	0.97408(105)
C(27)	0.77358(126)	0.74845(91)	0.78601(109)
N(31)	0.95978(93)	0.77218(63)	1.13401(76)
N(32)	0.97506(92)	0.69956(53)	1.15653(76)

C(33)	0.95648(119)	0.68650(72)	1.24270(99)
C(34)	0.93123(112)	0.75355(75)	1.27893(94)
C(35)	0.93673(127)	0.80547(75)	1.20881(104)
C(36)	0.96682(127)	0.61351(87)	1.28706(103)
C(37)	0.90691(131)	0.88345(91)	1.21462(108)
C(41)	1.08835(116)	0.84610(82)	0.72820(93)
C(42)	1.09972(122)	0.77485(82)	0.70082(99)
C(43)	1.13470(128)	0.76049(88)	0.61598(106)
C(44)	1.15732(135)	0.82091(82)	0.56197(112)
C(45)	1.14537(133)	0.88999(90)	0.58934(110)
C(46)	1.11073(123)	0.90479(83)	0.67418(100)
C(51)	0.60933(149)	0.90067(99)	0.33379(122)