Table 2. *Selected geometric parameters* (Å, °)

Ru1—C1	2.209 (5)	C2—C3	1.500 (8)
Ru1—C2	2.251 (5)	F1—B	1.368 (5)
C1—C2^i	1.407 (8)	F2—B	1.390 (12)
C1—C2	1.420 (9)		
C2^i—C1—C2	121.9 (5)	C1—C2—C3	120.5 (5)
C1^{ii}—C2—C1	118.0 (5)	F1^{ii}—B—F1	109.9 (4)
C1^{ii}—C2—C3	121.4 (6)	F1—B—F2	109.1 (5)

Symmetry codes: (i) y, z, x; (ii) z, x, y.

Preliminary microscopic examination of the crystals revealed that they did not transmit polarized light in any orientation, implying that they were cubic. This was confirmed on indexing a list of reflections generated by a random search of reciprocal space made on a Stoe Stadi-4 four-circle diffractometer. Data were collected at 150 K using an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986).

Examination of the full data set, which had been corrected for Lp and absorption effects, implied a primitive unit cell in Laue group $m\bar{3}m$ with reflections in the classes $hk0$, $h0l$ and $0kl$ systematically absent only when both indices are odd. Reflections with all indices even or all indices odd tended, in addition, to be systematically strong, a symptom of pseudo-F centring. These observations suggested that the crystal was twinned on (110) in space group $Pa\bar{3}$, with the Ru atoms occupying positions equivalent to 0,0,0 or $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, and common for both twin components. Thus, whereas reflections of the class $hk0$ are normally present for $h = 2n$ in this space group, inclusion of the twin component, which interchanges h and k, means that this absence condition only holds when both indices are odd.

The volume of the unit cell (2017.6 Å^3) implied the presence of four formula units of (1) per unit cell. Interpretation of the Patterson function was consistent with an Ru atom at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ and suggested a position for a C atom to break the pseudo-symmetry. Subsequent Fourier syntheses with coefficients ($2F_o-F_c$) and (F_o-F_c) located the BF$_4^-$ and remaining C-atom positions, respectively. During the initial stages of least-squares refinement the geometries of the mesitylene and tetrafluoroborate fragments were made subject to chemically reasonable restraints (bond lengths: C—C = 1.39 and 1.52, B—F = 1.4 Å) and the twin component was allowed to vary. The restraints were later removed and the twin component fixed at 0.5 after it had refined to 0.498 (5).

The H atom attached to the ring was placed in a calculated position (C—H = 0.95 Å), and allowed to ride on C(1). The positions of the methyl H atoms were derived from a difference synthesis. During refinement the C(3)—H bond lengths (0.98 Å) and H—C(3)—H angles (109.5°) were fixed but the H—C(3)—C(2)—C(1) torsion angles were allowed to vary. All H atoms were assigned an isotropic displacement parameter equal to $1.2U_{eq}$(C).

Data collection: *DIF4* (Stoe and Cie, 1990a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1990b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *CAMERON* (Pearce & Watkin, 1993).

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A Tungsten Nitrosyl Complex

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Abstract

Insertion of nitric oxide into one W—C bond of a bis(trimethylsilyl)tungsten complex results in the formation of a bidentate *N*-trimethylsilylmethyl-*N*-nitrosohydroxylaminato ligand. The resulting complex, (η^5-cyclopentadienyl)nitrosyl(trimethylsilylmethyl)(*N*-trimethylsilylmethyl-*N*-nitrosohydroxylaminato)tungsten, [W(NO)(C$_5$H$_5$)(C$_4$H$_{11}$N$_2$O$_2$Si)(C$_4$H$_{11}$Si)], crystallizes in space group $P\bar{1}$, $Z = 4$ (two molecules per asymmetric unit). The two independent molecules are almost identical and have a four-legged piano stool structure with a five-coordinate W atom.

Comment

The ligand geometry is similar to that found in [WMe$_4${ON(Me)NO$_2$}] (Fletcher, Shortland, Skapski & Wilkinson, 1972; Fletcher & Skapski, 1973).
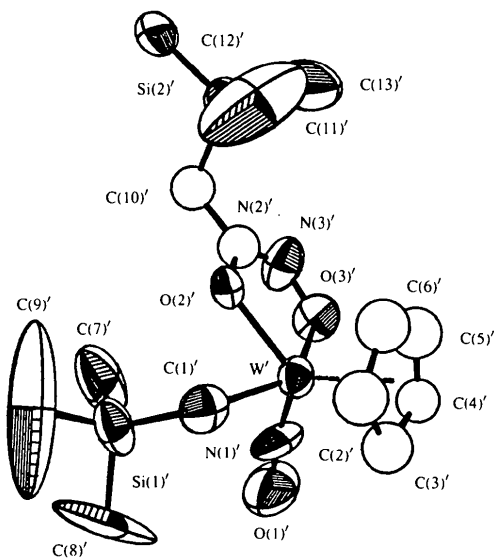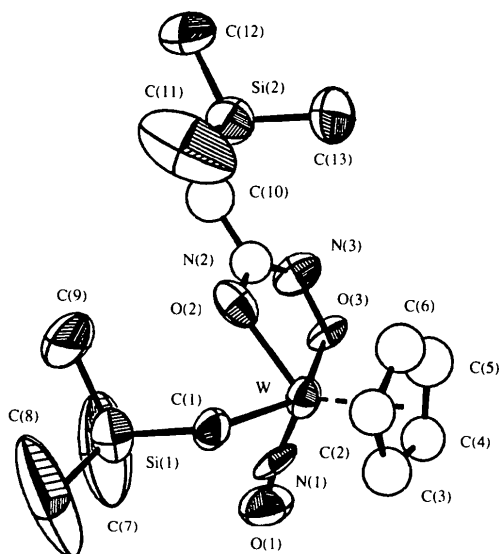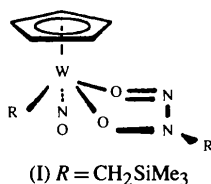
(I) $R = CH_2SiMe_3$

Fig. 1. Views of the two independent molecules (50% probability ellipsoids; unshaded atoms have isotropic displacement parameters).

Experimental

The title compound was synthesized according to the procedure described by Legzdins, Rettig & Sánchez (1988).

Crystal data

[W(NO)(C$_5$H$_5$)-(C$_4$H$_{11}$N$_2$O$_2$Si)(C$_4$H$_{11}$Si)]	Mo $K\alpha$ radiation
$M_r = 513.40$	$\lambda = 0.7107$ Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
$a = 12.610 (3)$ Å	$\theta = 10-20°$
$b = 16.038 (4)$ Å	$\mu = 5.88$ mm^{-1}
$c = 12.106 (3)$ Å	$T = 294$ K
$\alpha = 111.51 (2)°$	Plate
$\beta = 107.92 (2)°$	$0.31 \times 0.15 \times 0.05$ mm
$\gamma = 94.07 (2)°$	Yellow
$V = 2120 (1)$ Å^3	
$Z = 4$	
$D_x = 1.609$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4F diffractometer	$\theta_{max} = 26.25°$
$\omega/2\theta$ scans	$h = -15 \rightarrow 15$
Absorption correction: analytical	$k = 0 \rightarrow 17$
$T_{min} = 0.43$, $T_{max} = 0.77$	$l = -15 \rightarrow 15$
6228 measured reflections	3 standard reflections
6228 independent reflections	monitored every 150
2442 observed reflections	reflections
[$I > 3\sigma(I)$]	intensity decay: 68%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.13$
$R = 0.050$	$\Delta\rho_{max} = 1.1$ e Å^{-3}
$wR = 0.046$	$\Delta\rho_{min} = -1.9$ e Å^{-3}
$S = 1.54$	Extinction correction: none
2442 reflections	Atomic scattering factors
327 parameters	from International Tables
H-atom parameters not refined	for X-ray Crystallography (1974, Vol. IV)
$w = 1/\sigma^2(F)$	

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for N(2), N(2)', C(10) and C(10)' and cyclopentadienyl C atoms (indicated by values with e.s.d.'s in parentheses). For all other atoms
$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
W	0.11916 (7)	0.22523 (6)	0.02029 (8)	0.052
Si(1)	0.20054 (57)	0.08277 (54)	−0.23020 (63)	0.082
Si(2)	0.33928 (50)	0.05342 (53)	0.27335 (62)	0.078
N(1)	−0.0039 (14)	0.1872 (12)	−0.1172 (17)	0.059
N(2)	0.1436 (15)	0.0797 (13)	0.1086 (16)	0.065 (5)
N(3)	0.0628 (13)	0.1024 (14)	0.1400 (17)	0.070
O(1)	−0.0949 (14)	0.1711 (12)	−0.2047 (15)	0.100
O(2)	0.1917 (10)	0.1179 (10)	0.0539 (11)	0.068
O(3)	0.0251 (9)	0.1655 (11)	0.1032 (12)	0.059
C(1)	0.2275 (15)	0.1970 (14)	−0.0944 (18)	0.060
C(2)	0.2595 (15)	0.3629 (15)	0.1188 (18)	0.060 (6)
C(3)	0.1506 (17)	0.3742 (16)	0.0522 (20)	0.074 (7)
C(4)	0.0782 (16)	0.3660 (15)	0.1161 (19)	0.066 (6)
C(5)	0.1461 (19)	0.3463 (17)	0.2178 (21)	0.084 (7)
C(6)	0.2518 (16)	0.3485 (16)	0.2152 (20)	0.074 (7)
Cp	0.1772	0.3596	0.1440	0.07
C(7)	0.0606 (32)	0.0142 (27)	−0.3069 (36)	0.251
C(8)	0.2588 (56)	0.0938 (24)	−0.3396 (41)	0.350
C(9)	0.2641 (45)	0.0003 (27)	−0.1695 (32)	0.264

C(10)	0.1900 (17)	0.0061 (18)	0.1387 (21)	0.085 (7)
C(11)	0.4438 (21)	0.0875 (27)	0.2108 (27)	0.173
C(12)	0.3773 (21)	−0.0389 (20)	0.3246 (23)	0.127
C(13)	0.3266 (29)	0.1545 (20)	0.4002 (24)	0.152
W'	0.74095 (7)	0.36181 (7)	0.17686 (8)	0.057
Si(1)'	0.61259 (56)	0.54570 (52)	0.28445 (67)	0.088
Si(2)'	0.84664 (65)	0.32599 (50)	0.62146 (69)	0.090
N(1)'	0.7875 (19)	0.4431 (19)	0.1432 (18)	0.108
N(2)'	0.8605 (16)	0.4027 (13)	0.4512 (17)	0.069 (5)
N(3)'	0.9378 (15)	0.3891 (15)	0.4110 (19)	0.094
O(1)'	0.8313 (20)	0.4988 (15)	0.1083 (19)	0.149
O(2)'	0.7541 (11)	0.3960 (9)	0.3683 (12)	0.066
O(3)'	0.9088 (10)	0.3691 (10)	0.2900 (15)	0.085
C(1)'	0.5914 (16)	0.4205 (16)	0.1894 (18)	0.075
C(2)'	0.5881 (19)	0.2396 (18)	0.0231 (22)	0.092 (5)
C(3)'	0.6560 (19)	0.2636 (17)	−0.0387 (21)	0.086 (7)
C(4)'	0.7598 (16)	0.2412 (16)	0.0115 (20)	0.070 (6)
C(5)'	0.7570 (20)	0.2057 (18)	0.0915 (23)	0.095 (8)
C(6)'	0.6510 (20)	0.2017 (18)	0.0993 (23)	0.092 (8)
Cp'	0.6824	0.2304	0.0373	0.09
C(7)'	0.7470 (24)	0.6039 (20)	0.4182 (28)	0.156
C(8)'	0.5945 (47)	0.6044 (25)	0.1810 (34)	0.320
C(9)'	0.4977 (32)	0.5673 (26)	0.3439 (58)	0.380
C(10)'	0.8715 (16)	0.4277 (16)	0.5835 (20)	0.075 (7)
C(11)'	0.7251 (37)	0.2510 (32)	0.5168 (33)	0.272
C(12)'	0.8493 (24)	0.3697 (17)	0.7850 (22)	0.107
C(13)'	0.9744 (34)	0.2675 (31)	0.6076 (41)	0.232

Table 2. *Selected geometric parameters* (Å)

W—N(1)	1.76 (2)	W'—N(1)'	1.62 (3)
W—O(2)	2.12 (2)	W'—O(2)'	2.13 (1)
W—O(3)	2.15 (1)	W'—O(3)'	2.11 (1)
W—C(1)	2.19 (2)	W'—C(1)'	2.19 (2)
W—C(2)	2.38 (2)	W'—C(2)'	2.37 (2)
W—C(3)	2.26 (2)	W'—C(3)'	2.34 (2)
W—C(4)	2.31 (2)	W'—C(4)'	2.31 (2)
W—C(5)	2.37 (2)	W'—C(5)'	2.38 (2)
W—C(6)	2.44 (2)	W'—C(6)'	2.43 (2)
W—Cp	2.04	W'—Cp'	2.06
Si(1)—C(1)	1.88 (2)	Si(1)'—C(1)'	1.86 (2)
Si(2)—C(10)	1.94 (2)	Si(2)'—C(10)'	1.88 (2)
N(1)—O(1)	1.23 (2)	N(1)'—O(1)'	1.28 (2)
N(2)—N(3)	1.23 (2)	N(2)'—N(3)'	1.22 (2)
N(2)—O(2)	1.29 (2)	N(2)'—O(2)'	1.38 (2)
N(2)—C(10)	1.47 (3)	N(2)'—C(10)'	1.46 (2)
N(3)—O(3)	1.31 (2)	N(3)'—O(3)'	1.30 (2)

Three standard reflections decayed to an average 32% of the initial intensities and the data were scaled accordingly; a similar decay was reported for the related compound [WMe$_4${ON(Me)NO$_2$}] (Fletcher & Skapski, 1973). The structure was determined by Patterson methods. Some large displacement parameters for the peripheral atoms indicate possible disorder. Anisotropic displacement parameters for N(2), N(2)', C(10) and C(10)', although not large, were physically unrealistic, and these atoms were treated isotropically in the final least-squares cycles, as were the cyclopentadienyl C atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: local program. Program(s) used to refine structure: *ORFLS* (Busing, Martin & Levy, 1962). Molecular graphics: *ORTEPII* (Johnson, 1976).

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Diiodobis(isopropyldiphenylphosphine oxide-*O*)cobalt(II)

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Abstract

The title compound, [CoI$_2${(C$_6$H$_5$)$_2$(C$_3$H$_7$)OP}$_2$], contains a four-coordinate Co atom in a distorted tetrahedral coordination environment. Bond lengths are: Co—I 2.558 (1) and 2.593 (2) Å, Co—O 1.958 (6) and 1.946 (6) Å. Angles at the Co atom are: I—Co—I 117.11 (5), O—Co—O 102.7 (3)°.

Comment

Relatively few structures of phosphine oxide complexes of cobalt(II) have been reported. In (Ph$_3$PO)$_2$CoCl$_2$ the metal has distorted tetrahedral coordination with Cl—Co—Cl 114.0 (1) and O—Co—O 96.4 (3)° (Mangion, Smith & Shore, 1976). Spectroscopic studies suggest similar structures for the bromo and iodo analogues (Pierrard, Rimbault & Hugel, 1976, 1982). Complexes of cobalt(II) nitrate, (R_3PO)$_2$Co(NO$_3$)$_2$, contain bidentate nitrate groups and *cis*-phosphine oxides, with pseudo-octahedral coordination at the Co atom for R = Ph (Dias Rodrigues, Francisco & Lechat, 1982) or R = Et (Alnaji, Dartigueanave, Dartigueanave, Simard & Beauchamp, 1991).

As part of an investigation of phosphine complexes of cobalt halides, we studied the interaction of CoI$_2$ with isopropyldiphenylphosphine. A structural investigation of the product showed that air oxidation had occurred to give the 2:1 complex, (I), of the phosphine oxide.