1879 reflections
142 parameters
$w = 9.3387/[\sigma^2(F)$
$+ 0.000176(F)^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *The unit-cell parameters,* Cs^+ *ion position and centre of the anion in* $Cs[CB_{10}H_{13}]$ *and* β- *and* γ-$Cs[C_2B_9H_{12}]$ *referred to a unit cell with* $P2_1/a$ *setting*

	γ-$Cs[C_2B_9H_{12}]^a$	$Cs[CB_{10}H_{13}]^b$	β-$Cs[C_2B_9H_{12}]^a$
Cell parameters (Å, °)			
a	11.222 (4)	12.162 (1)	12.15 (2)
b	10.696 (4)	10.647 (1)	12.55 (2)
c	8.360 (3)	7.985 (1)	6.90 (2)
β	92.62 (2)	92.441 (5)	90.0 (1)
Cs^+ position			
x	0.5106 (1)	0.51065 (3)	0.508 (8)
y	0.2591 (1)	0.24381 (3)	0.241 (6)
z	0.2428 (1)	0.15785 (4)	0.111 (3)
Geometric centre of anion			
x	0.2482	0.2427 (5)	0.28
y	0.1231	0.0916 (5)	0.061
z	0.7692	0.7063 (7)	0.65

References: (*a*) Rius, Romerosa, Teixidor, Casabó & Miravitlles (1991); (*b*) this work.

Table 2. *Average carborane-cage bond lengths and* Cs···B *contact distances* (Å)

B—$B_{average}$	1.785 (8)	B9···Cs^{iii}	3.752 (6)
B—$C_{average}$	1.66 (1)	B3···Cs^{iv}	3.767 (6)
B5···Cs^{i}	3.695 (5)	B11···Cs^{v}	4.014 (6)
B2···Cs^{ii}	3.728 (5)	B10···Cs	4.083 (6)

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, 1 - z$; (iii) $x, y, 1 + z$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, 1 + z$; (v) $x - \frac{1}{2}, \frac{1}{2} - y, z$.

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²) *for* $Cs[CB_{10}H_{13}]$

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Cs	0.51065 (3)	0.24381 (3)	0.15785 (4)	0.0414 (3)
B1	0.2495 (4)	−0.0280 (5)	0.8123 (7)	0.032 (3)
B2	0.1615 (4)	−0.0199 (5)	0.6295 (7)	0.035 (3)
B3	0.1323 (4)	0.0688 (5)	0.8111 (8)	0.034 (3)
B4	0.2555 (5)	0.1254 (5)	0.9028 (7)	0.031 (3)
B5	0.3630 (4)	0.0692 (5)	0.7798 (7)	0.032 (3)
B6	0.3043 (5)	−0.0197 (5)	0.6061 (7)	0.033 (3)
C7	0.1246 (4)	0.1338 (5)	0.6194 (7)	0.036 (3)
B8	0.1745 (5)	0.2254 (6)	0.7683 (8)	0.031 (3)
B9	0.3267 (5)	0.2286 (5)	0.7691 (8)	0.033 (3)
B10	0.3582 (5)	0.1342 (6)	0.5796 (8)	0.038 (3)
B11	0.2195 (5)	0.0899 (6)	0.4917 (7)	0.041 (3)

The H atoms were located from difference Fourier maps. The positions of the 11 non-bridging H atoms were refined and the positions of the two bridging H atoms were fixed with $U_{iso} = 1.25 \times U_{eq}(C,B_{parent})$ for all H atoms.

Data collection and cell refinement: *CAD*-4 *Express Operating Software* (Enraf–Nonius, 1992). Data reduction: *MolEN* (Fair, 1990). Structure solution: *MULTAN*11/84 (Main, Germain & Woolfson, 1984). Structure refinement: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(hexadecylpyridinium) Bis(3,6-dichloro-4,5-dihydroxy-3,5-cyclohexadiene-1,2-dionato-κ^2O^4,O^5)beryllium

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Abstract

The title compound, $2C_{21}H_{38}N[Be(C_6Cl_2O_4)_2]$, contains complex anions formed by two 3,6-dichloro-4,5-dihydroxy-3,5-cyclohexadiene-1,2-dionato residues, tetrahedrally coordinating to beryllium through deprotonated hydroxy O atoms, and hexadecylpyridinium

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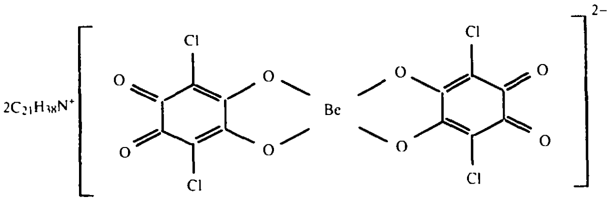
cations. The long *c* axis is mainly a result of the presence of the hexadecylpyridinium cations. The cation chains are parallel to the [31$\bar{1}$] direction and the pyridine rings are connected to the chloranilate O atoms by hydrogen bonds. The chains are regularly ordered parallel to one another.

Comment

This investigation is the second of a series studying the behaviour of different metals in anilates. All angles and distances are in good agreement with values found in the literature (Krogh Andersen, 1967; Krogh Andersen & Krogh Andersen, 1975).



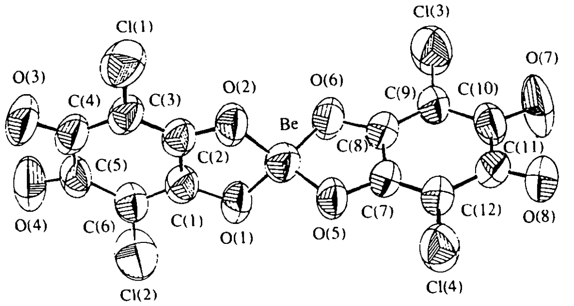


Fig. 1. View of the complex beryllium anion (displacement ellipsoids at 66.6% probability).

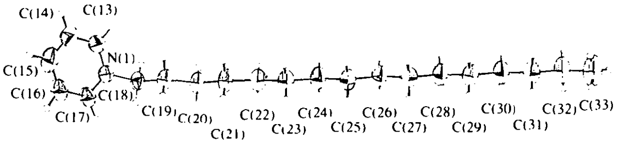


Fig. 2. View of the first hexadecylpyridinium cation (displacement ellipsoids at 50% probability).

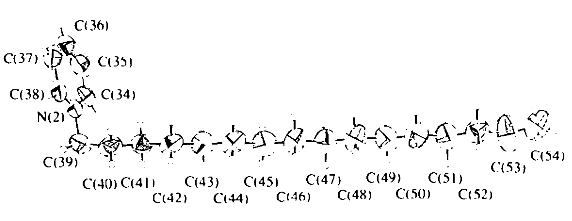


Fig. 3. View of the second hexadecylpyridinium cation (displacement ellipsoids at 50% probability).

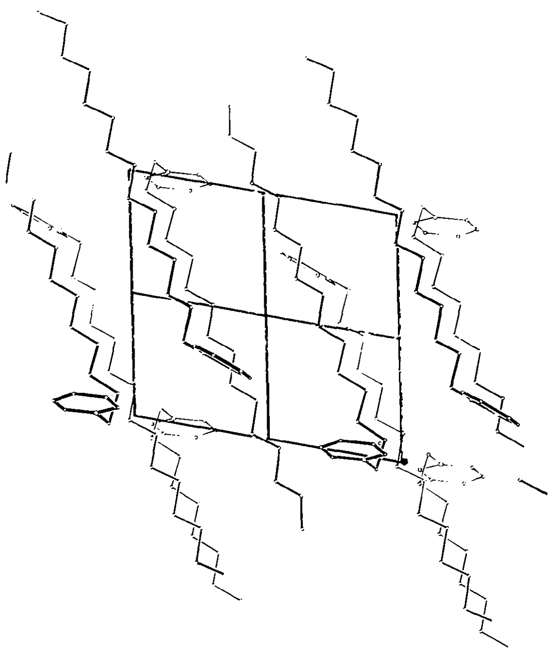


Fig. 4. View of the hexadecylpyridinium cations in the cell (projection parallel to the *c* axis, dot as origin, right-hand system).

Experimental

To an aqueous solution of the hydroxyberyllate complex, the stoichiometric amount of chloranilic acid–water suspension was introduced step-by-step at a constant temperature of 343 K. After 3 h at water-bath temperature, the mixture was filtered and transferred dropwise to an aqueous solution of the quaternary salt hexadecylpyridinium chloride, under very strong agitation. The initially separated mass was transformed to a flocculent red crystalline precipitate of the complex beryllium salt. Finally, the filtered and washed mass was extracted with hot ethyl acetate giving a brilliant red solution. The organic layer was dried with anhydrous Na_2SO_4 for 12 h and filtered. The solvent was then entirely expelled with flash evaporation leaving a carmine-red crystalline compound in long lustrous crystals in a yield of *ca* 95%.

Crystal data

$2C_{21}H_{38}N[Be(C_6Cl_2O_4)_2]$
$M_r = 1032.03$
Triclinic
$P\bar{1}$
$a = 10.3254\ (3)$ Å
$b = 11.2915\ (2)$ Å
$c = 25.0237\ (5)$ Å
$\alpha = 85.981\ (3)^\circ$
$\beta = 80.177\ (3)^\circ$
$\gamma = 78.517\ (3)^\circ$
$V = 2815.2\ (4)$ Å^3
$Z = 2$
$D_x = 1.217$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 32 reflections
$\theta = 7.4–10.6^\circ$
$\mu = 0.259$ mm^{-1}
$T = 293$ K
Parallelepiped
0.31 × 0.18 × 0.13 mm
Red

Data collection

Rebuilt Philips PW1100 diffractometer (Gomm, 1993)
$\theta/2\theta$ scans
Absorption correction: Gaussian by integration from crystal shape (Busing & Levy, 1957)
$T_{\min} = 0.940$, $T_{\max} = 0.971$
18 804 measured reflections
9401 independent reflections
4863 observed reflections $[F > 1.4\sigma(F)]$

$R_{\text{int}} = 0.0431$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -29 \rightarrow 29$
3 standard reflections monitored every 100 reflections
intensity decay: 2.5%

Refinement

Refinement on F
$R = 0.068$
$wR = 0.032$
$S = 1.44$
4863 reflections
851 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.47$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl(1)	0.0036 (1)	0.3956 (1)	0.24004 (5)	0.0834 (6)
Cl(2)	0.3306 (1)	0.2613 (1)	0.00644 (5)	0.0800 (6)
Cl(3)	0.3115 (1)	0.94273 (9)	0.07040 (5)	0.0851 (6)
Cl(4)	0.7218 (1)	0.6049 (1)	0.20389 (5)	0.0773 (6)
O(1)	0.3690 (2)	0.4393 (2)	0.0862 (1)	0.057 (1)
O(2)	0.2339 (2)	0.4985 (2)	0.1746 (1)	0.058 (1)
O(3)	−0.0273 (3)	0.2057 (2)	0.1677 (1)	0.084 (2)
O(4)	0.0815 (3)	0.1788 (2)	0.0631 (1)	0.081 (2)
O(5)	0.4895 (2)	0.5450 (2)	0.1570 (1)	0.056 (1)
O(6)	0.3237 (2)	0.6782 (2)	0.1089 (1)	0.059 (1)
O(7)	0.5513 (3)	0.9994 (2)	0.1060 (1)	0.093 (2)
O(8)	0.7080 (3)	0.8667 (2)	0.1713 (1)	0.075 (1)
N(1)	0.3050 (3)	0.7347 (3)	0.8856 (1)	0.051 (2)
N(2)	−0.0145 (3)	0.1408 (4)	0.8452 (2)	0.059 (2)
C(1)	0.2729 (4)	0.3792 (3)	0.0994 (2)	0.049 (2)
C(2)	0.1921 (4)	0.4146 (3)	0.1537 (2)	0.050 (2)
C(3)	0.0896 (4)	0.3592 (4)	0.1763 (2)	0.056 (2)
C(4)	0.0562 (4)	0.2668 (4)	0.1490 (2)	0.060 (2)
C(5)	0.1276 (4)	0.2428 (3)	0.0893 (2)	0.058 (2)
C(6)	0.2407 (4)	0.2970 (3)	0.0696 (2)	0.055 (2)
C(7)	0.5145 (4)	0.6532 (4)	0.1492 (2)	0.048 (2)
C(8)	0.4143 (4)	0.7338 (4)	0.1195 (2)	0.050 (2)
C(9)	0.4241 (4)	0.8502 (3)	0.1058 (2)	0.055 (2)
C(10)	0.5307 (4)	0.8987 (4)	0.1194 (2)	0.058 (2)
C(11)	0.6261 (4)	0.8193 (4)	0.1551 (2)	0.056 (2)
C(12)	0.6134 (4)	0.6959 (4)	0.1661 (2)	0.055 (2)
C(13)	0.3281 (5)	0.7658 (4)	0.9330 (2)	0.071 (2)
C(14)	0.3033 (5)	0.6955 (5)	0.9789 (2)	0.084 (3)
C(15)	0.2578 (5)	0.5918 (5)	0.9756 (2)	0.088 (3)
C(16)	0.2363 (5)	0.5604 (4)	0.9271 (2)	0.077 (3)
C(17)	0.2606 (4)	0.6329 (4)	0.8815 (2)	0.063 (2)
C(18)	0.3372 (4)	0.8104 (4)	0.8356 (2)	0.056 (2)
C(19)	0.4841 (4)	0.7750 (4)	0.8115 (2)	0.063 (2)
C(20)	0.5178 (4)	0.8648 (4)	0.7651 (2)	0.064 (2)
C(21)	0.6641 (5)	0.8372 (4)	0.7403 (2)	0.076 (2)
C(22)	0.7025 (5)	0.9331 (4)	0.6982 (2)	0.073 (2)
C(23)	0.8443 (5)	0.9039 (4)	0.6688 (2)	0.073 (2)
C(24)	0.8825 (5)	1.0045 (4)	0.6298 (2)	0.075 (2)
C(25)	1.0229 (5)	0.9777 (4)	0.5982 (2)	0.073 (2)
C(26)	1.0599 (5)	1.0809 (4)	0.5610 (2)	0.075 (2)
C(27)	1.1997 (5)	1.0556 (4)	0.5289 (2)	0.072 (2)
C(28)	1.2339 (5)	1.1609 (4)	0.4919 (2)	0.071 (2)
C(29)	1.3749 (5)	1.1369 (4)	0.4599 (2)	0.072 (2)
C(30)	1.4068 (5)	1.2416 (4)	0.4228 (2)	0.076 (2)
C(31)	1.5469 (5)	1.2203 (4)	0.3906 (2)	0.079 (3)
C(32)	1.5747 (6)	1.3257 (5)	0.3533 (2)	0.099 (3)
C(33)	1.7127 (7)	1.3084 (6)	0.3202 (3)	0.128 (4)
C(34)	−0.0248 (4)	0.2590 (5)	0.8494 (3)	0.073 (3)
C(35)	0.0057 (6)	0.2992 (6)	0.8937 (3)	0.095 (3)
C(36)	0.0466 (6)	0.2244 (8)	0.9343 (3)	0.098 (4)
C(37)	0.0557 (5)	0.1021 (7)	0.9314 (3)	0.096 (3)
C(38)	0.0235 (5)	0.0597 (4)	0.8842 (3)	0.074 (2)
C(39)	−0.0467 (6)	0.0955 (5)	0.7944 (3)	0.091 (3)
C(40)	0.0803 (7)	0.0589 (5)	0.7539 (2)	0.094 (3)
C(41)	0.1334 (5)	0.1636 (5)	0.7228 (2)	0.080 (3)
C(42)	0.2660 (6)	0.1290 (5)	0.6860 (2)	0.090 (3)
C(43)	0.3146 (6)	0.2315 (5)	0.6524 (2)	0.092 (3)
C(44)	0.4485 (7)	0.2028 (6)	0.6169 (2)	0.098 (3)
C(45)	0.4947 (6)	0.3034 (5)	0.5815 (3)	0.097 (3)
C(46)	0.6292 (7)	0.2794 (6)	0.5485 (2)	0.101 (3)
C(47)	0.6720 (6)	0.3812 (5)	0.5138 (3)	0.100 (3)
C(48)	0.8088 (7)	0.3588 (5)	0.4819 (3)	0.106 (4)
C(49)	0.8525 (7)	0.4597 (6)	0.4470 (3)	0.106 (4)
C(50)	0.9899 (7)	0.4395 (6)	0.4156 (3)	0.106 (4)
C(51)	1.0291 (7)	0.5414 (5)	0.3803 (3)	0.107 (4)
C(52)	1.1679 (7)	0.5232 (6)	0.3509 (3)	0.103 (3)
C(53)	1.2053 (8)	0.6215 (7)	0.3142 (4)	0.154 (6)
C(54)	1.338 (1)	0.6216 (7)	0.2893 (4)	0.147 (6)
Be(1)	0.3566 (6)	0.5384 (5)	0.1325 (2)	0.058 (3)

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

O(1)—C(1)	1.295 (5)	C(20)—C(21)	1.511 (6)
O(2)—C(2)	1.291 (5)	C(21)—C(22)	1.520 (7)
O(5)—C(7)	1.294 (5)	C(22)—C(23)	1.507 (6)
O(6)—C(8)	1.296 (5)	C(23)—C(24)	1.513 (7)
N(1)—C(13)	1.334 (7)	C(24)—C(25)	1.512 (6)
N(1)—C(17)	1.336 (6)	C(25)—C(26)	1.510 (7)
N(1)—C(18)	1.491 (5)	C(26)—C(27)	1.510 (7)
N(2)—C(34)	1.328 (7)	C(27)—C(28)	1.516 (7)
N(2)—C(38)	1.343 (7)	C(28)—C(29)	1.519 (6)
N(2)—C(39)	1.513 (8)	C(29)—C(30)	1.506 (7)
C(1)—C(6)	1.354 (6)	C(30)—C(31)	1.513 (7)
C(1)—C(2)	1.504 (5)	C(31)—C(32)	1.503 (7)
C(2)—C(3)	1.357 (6)	C(32)—C(33)	1.504 (8)
C(3)—C(4)	1.414 (7)	C(34)—C(35)	1.33 (1)
C(3)—Cl(1)	1.721 (4)	C(35)—C(36)	1.34 (1)
C(4)—O(3)	1.222 (5)	C(36)—C(37)	1.37 (1)
C(4)—C(5)	1.565 (6)	C(37)—C(38)	1.42 (1)
C(5)—O(4)	1.220 (6)	C(39)—C(40)	1.519 (8)
C(5)—C(6)	1.424 (6)	C(40)—C(41)	1.513 (8)
C(6)—Cl(2)	1.721 (4)	C(41)—C(42)	1.510 (7)
C(7)—C(12)	1.352 (6)	C(42)—C(43)	1.500 (8)
C(7)—C(8)	1.498 (5)	C(43)—C(44)	1.499 (8)
C(8)—C(9)	1.354 (6)	C(44)—C(45)	1.494 (8)
C(9)—C(10)	1.422 (7)	C(45)—C(46)	1.475 (8)
C(9)—Cl(3)	1.722 (4)	C(46)—C(47)	1.484 (9)
C(10)—O(7)	1.212 (5)	C(47)—C(48)	1.482 (9)
C(10)—C(11)	1.553 (6)	C(48)—C(49)	1.480 (9)
C(11)—O(8)	1.222 (6)	C(49)—C(50)	1.483 (9)
C(11)—C(12)	1.428 (6)	C(50)—C(51)	1.480 (9)
C(12)—Cl(4)	1.723 (4)	C(51)—C(52)	1.477 (9)
C(13)—C(14)	1.366 (7)	C(52)—C(53)	1.46 (1)
C(14)—C(15)	1.358 (9)	C(53)—C(54)	1.41 (1)
C(15)—C(16)	1.354 (9)	Be(1)—O(5)	1.610 (7)
C(16)—C(17)	1.374 (7)	Be(1)—O(2)	1.620 (6)
C(18)—C(19)	1.517 (6)	Be(1)—O(6)	1.637 (6)
C(19)—C(20)	1.529 (6)	Be(1)—O(1)	1.639 (7)
Be(1)—O(1)—C(1)	108.9 (3)	C(14)—C(13)—N(1)	120.5 (5)
Be(1)—O(2)—C(2)	109.1 (3)	C(13)—C(14)—C(15)	119.5 (5)
Be(1)—O(5)—C(7)	109.5 (3)	C(14)—C(15)—C(16)	119.5 (5)
Be(1)—O(6)—C(8)	108.1 (3)	C(17)—C(16)—C(15)	120.3 (5)
C(17)—N(1)—C(13)	121.0 (4)	C(16)—C(17)—N(1)	119.3 (5)
C(18)—N(1)—C(13)	119.7 (4)	C(19)—C(18)—N(1)	110.4 (3)

C(18)—N(1)—C(17)	119.2 (4)	C(20)—C(19)—C(18)	109.0 (3)
C(38)—N(2)—C(34)	122.7 (5)	C(19)—C(20)—C(21)	111.8 (4)
C(39)—N(2)—C(34)	118.7 (4)	C(22)—C(21)—C(20)	112.4 (4)
C(39)—N(2)—C(38)	118.6 (5)	C(21)—C(22)—C(23)	114.3 (4)
C(6)—C(1)—O(1)	127.3 (4)	C(24)—C(23)—C(22)	113.1 (4)
C(2)—C(1)—O(1)	111.3 (4)	C(23)—C(24)—C(25)	114.9 (4)
C(2)—C(1)—C(6)	121.4 (4)	C(24)—C(25)—C(26)	113.9 (4)
C(3)—C(2)—O(2)	126.6 (4)	C(27)—C(26)—C(25)	114.6 (4)
C(1)—C(2)—O(2)	112.3 (3)	C(28)—C(27)—C(26)	113.4 (4)
C(1)—C(2)—C(3)	121.1 (4)	C(29)—C(28)—C(27)	113.9 (4)
C(4)—C(3)—C(2)	120.4 (4)	C(28)—C(29)—C(30)	113.3 (4)
Cl(1)—C(3)—C(2)	120.5 (3)	C(31)—C(30)—C(29)	114.3 (4)
Cl(1)—C(3)—C(4)	119.0 (3)	C(30)—C(31)—C(32)	113.0 (4)
C(3)—C(4)—O(3)	125.2 (4)	C(33)—C(32)—C(31)	114.9 (5)
C(5)—C(4)—O(3)	116.7 (4)	C(35)—C(34)—N(2)	118.9 (5)
C(5)—C(4)—C(3)	118.0 (4)	C(36)—C(35)—C(34)	122.5 (7)
C(6)—C(5)—O(4)	124.9 (4)	C(37)—C(36)—C(35)	119.8 (7)
C(4)—C(5)—O(4)	117.1 (4)	C(38)—C(37)—C(36)	117.5 (6)
C(4)—C(5)—C(6)	117.9 (4)	C(37)—C(38)—N(2)	118.6 (5)
C(5)—C(6)—C(1)	120.1 (4)	C(40)—C(39)—N(2)	110.4 (5)
Cl(2)—C(6)—C(1)	120.8 (3)	C(39)—C(40)—C(41)	114.5 (4)
Cl(2)—C(6)—C(5)	119.0 (3)	C(40)—C(41)—C(42)	114.9 (4)
C(12)—C(7)—O(5)	127.3 (4)	C(41)—C(42)—C(43)	115.0 (4)
C(8)—C(7)—O(5)	111.5 (4)	C(42)—C(43)—C(44)	117.1 (5)
C(8)—C(7)—C(12)	121.2 (4)	C(43)—C(44)—C(45)	117.3 (5)
C(9)—C(8)—O(6)	126.5 (4)	C(44)—C(45)—C(46)	118.4 (5)
C(7)—C(8)—O(6)	112.2 (3)	C(47)—C(46)—C(45)	117.1 (5)
C(7)—C(8)—C(9)	121.2 (4)	C(46)—C(47)—C(48)	117.3 (5)
C(10)—C(9)—C(8)	120.4 (4)	C(47)—C(48)—C(49)	117.8 (5)
Cl(3)—C(9)—C(8)	121.6 (3)	C(50)—C(49)—C(48)	118.5 (5)
Cl(3)—C(9)—C(10)	118.0 (3)	C(49)—C(50)—C(51)	117.0 (5)
C(9)—C(10)—O(7)	124.7 (4)	C(50)—C(51)—C(52)	117.0 (5)
C(11)—C(10)—O(7)	117.0 (4)	C(51)—C(52)—C(53)	117.2 (6)
C(11)—C(10)—C(9)	118.3 (4)	C(52)—C(53)—C(54)	122.5 (7)
C(12)—C(11)—O(8)	124.6 (4)	O(2)—Be(1)—O(5)	116.6 (4)
C(10)—C(11)—O(8)	117.4 (4)	O(6)—Be(1)—O(5)	98.6 (4)
C(10)—C(11)—C(12)	118.0 (4)	O(6)—Be(1)—O(2)	113.8 (3)
C(11)—C(12)—C(7)	120.4 (4)	O(1)—Be(1)—O(5)	117.8 (3)
Cl(4)—C(12)—C(7)	120.8 (3)	O(1)—Be(1)—O(2)	98.2 (4)
Cl(4)—C(12)—C(11)	118.7 (3)	O(1)—Be(1)—O(6)	112.8 (4)

The single crystals of this compound tend to be twinned, therefore, only a very small sample could be measured, resulting in a very high number of unobserved reflections. This is the reason for the difference between unweighted and weighted *R* values. The ratio could be improved by using only $F > 3\sigma(F)$ data, but then the reflections/parameter ratio would deteriorate.

One common isotropic displacement parameter for the H atoms was used. All H-atom positions could be recognized in difference Fourier maps. The refinement was carried out by the newly-developed Levenberg–Marquardt least-squares algorithm *LQFLEV* (Spengler & Zimmermann, 1994) using the second derivatives of the structure factors.

Data collection: local software (Gomm, 1993). Cell refinement: local software (Gomm, 1993). Data reduction: local software (Gomm, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *CRYSTAN* (Burzlaff & Rothammel, 1988). Software used to prepare material for publication: *CRYSTAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(η^5-Cyclopentadienyl)[2-(1,1-diphenylethyl-C^2)phenyl-C^1]oxo(trimethylsilylmethylthio)tungsten, [WO($C_4H_{11}SSi$)(C_5H_5)($C_{20}H_{16}$)]

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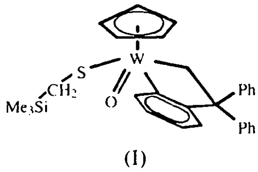
Abstract

Treatment of [CpW(NO)(CH_2SiMe_3)(CH_2CPh_3)] with sulfur produces [CpW(O)(SCH_2SiMe_3)(CH_2CPh_2-C_6H_4)], in which S is inserted in one W—C bond, but in addition, replacement of NO by O and orthometallation have occurred.

Comment

In a study of the reactions of the mixed alkyl complex CpW(NO)(CH_2SiMe_3)(CH_2CPh_3) (Brunet, Debad, Legzdins, Trotter, Veltheer & Yee, 1993), treatment with sulfur was found to give only one isolable product in very low yield. The present analysis has established the structure of this product, (I), as [CpW(O)(SCH_2SiMe_3)($CH_2CPh_2C_6H_4$)]. Sulfur has been inserted preferentially into one of the W—C(alkyl) bonds, but in addition, the nitrosyl group has been replaced by an oxo ligand and orthometallation has occurred.



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