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A Pentamethylcyclopentadienyl, Chloro, Oxo, Ether Complex of Ytterbium(III)

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Abstract. 2,3,4-μ_3-Chloro-1,2;1,2;2,3;2,4;3,4;3,5;4,5-hepta-μ-chloro-2,5-bis(diethyl ether)-3,4,5-μ_3-oxo-1,1,3,4,5-pentakis(η^5-pentamethylcyclopentadienyl)-pentaytterbium(III), [Yb$_5$O(C$_4$H$_{10}$O)$_2$(C$_{10}$H$_{15}$)$_5$Cl$_8$], $M_r = 1989.23$, monoclinic, $P2_1/m$, $a = 17.007$ (3), $b = 16.485$ (3), $c = 13.592$ (3) Å, $\beta = 111.39$ (2)°, $V = 3548.2$ Å^3, $Z = 2$, $D_x = 1.86$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 68.49$ cm^{-1}, $F(000) = 1906$, $T = 296$ K, $R = 0.056$ for 4061 [$F^2 > 2\sigma(F^2)$] of 6532 total unique data. The structure consists of a single large cluster in which the five Yb atoms are linked together by Cl and O bridges. The π-bonding cyclopentadienyl rings and the O atoms of the ether ligands are bonded to the Yb atoms on the periphery of the cluster.

Experimental. The title complex (1) was isolated from the reaction of [Yb(C$_5$Me$_5$)$_2$(OEt$_2$)] with YbCl$_3$ containing some YbOCl as an impurity. A blue air-sensitive crystal, 0.15 × 0.27 × 0.33 mm, was sealed inside a quartz capillary in an argon-filled drybox. X-ray diffraction intensities (θ–2θ scans) were obtained using a modified Picker FACS-I automatic diffractometer equipped with a graphite monochromator. Cell dimensions from 27 reflections, 20 < 2θ < 26°; analytical absorption correction, range 2.0 to 3.1; max. sinθ/λ = 0.60 Å^{-1}, h −20 to 20, k 0 to 19, l −16 to 16; three standard reflections, 2.2, 2.1, 2.3% variation in standards' intensities from average, intensities adjusted isotropically; 13 177 data, 6532 unique [including 4061, $F^2 > 2\sigma(F^2)$], $R_{int} = 0.045$; structure solved by Patterson and Fourier methods; refined on F, 281 parameters; H atoms not included; distance restraints on one ether [O(2)—C(29) 1.45 (1), C(29)—C(30) 1.54 (1) Å] and two atoms of a methyl Cp ring [C(23)—C(23')

1.41 (1), C(23)—C(28) 1.54 (1) Å]; anisotropic thermal parameters on 28 atoms (4 Yb, 5 Cl, 2 O and 17 C atoms), and isotropic on 16 atoms (1 O and 15 C atoms); $R = 0.099$ (all data), $R = 0.056$ [$F^2 > 2\sigma(F^2)$ data], $wR = 0.072$, $S = 1.44$; $w = 4F^2[\sigma^2(F^2) + (0.035F^2)^2]^{-1}$; max. (shift/$\sigma$) = 0.06; extinction correction $F_{obs}(1 + 3.4 \times 10^{-8}I)$, max. correction 2.7%; max. and min. of ΔF synthesis 2.3 and -1.9 e Å^{-3}; atomic f for neutral Yb, Cl, O and C atoms from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

(1)
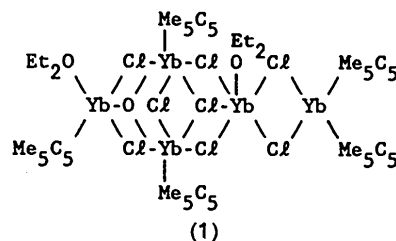
Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Fig. 1 shows a schematic diagram of the structure.

Related literature. Comparable distances for bridging Yb—Cl are 2.595 and 2.756 Å for Li[YbCl$_2$-(C$_5$Me$_5$)$_2$][Et$_2$O] and Yb(C$_5$Me$_5$)$_2$(μ-Cl)$_2$AlCl$_2$ respectively (Watson, Whitney & Harlow, 1981).

Table 1. Positional and thermal parameters ($\mathring{A}^2$)

$$B_{eq} = \tfrac{1}{3}\sum B_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B or B_{eq}
Yb(1)	0·32713 (4)	0·13684 (5)	0·22881 (5)	3·28 (2)
Yb(2)	0·36221 (6)	¼	0·00730 (8)	3·11 (3)
Yb(3)	0·09337 (7)	¼	0·20326 (9)	4·66 (4)
Yb(4)	−0·05438 (6)	¼	0·38927 (8)	3·54 (3)
Cl(1)	0·2735 (3)	¼	0·3353 (4)	3·7 (2)
Cl(2)	0·4502 (3)	¼	0·2580 (5)	3·9 (2)
Cl(3)	0·3689 (3)	0·09673 (28)	0·0640 (3)	4·6 (1)
Cl(4)	0·01739 (29)	0·14491 (29)	0·3001 (4)	5·1 (2)
Cl(5)	0·16355 (27)	0·1146 (3)	0·1382 (4)	5·3 (2)
O(1)	0·2820 (9)	¼	0·1067 (11)	3·9 (5)
O(2)	0·5107 (12)	¼	0·0443 (14)	5·7 (7)
O(3)	−0·0134 (22)	¼	0·0192 (27)	12·4 (9)
C(1)	0·3224 (17)	−0·0077 (14)	0·3108 (23)	7·3 (10)
C(2)	0·3363 (18)	0·0482 (19)	0·3894 (22)	7·2 (10)
C(3)	0·4156 (21)	0·0822 (12)	0·4123 (14)	7·3 (9)
C(4)	0·4477 (12)	0·0441 (18)	0·3417 (23)	6·8 (9)
C(5)	0·3891 (22)	−0·0096 (15)	0·2835 (17)	6·8 (10)
C(6)	0·2480 (28)	−0·0638 (28)	0·267 (3)	15·4 (13)
C(7)	0·2806 (27)	0·0701 (25)	0·452 (3)	15·2 (13)
C(8)	0·4677 (25)	0·1367 (23)	0·500 (3)	13·1 (11)
C(9)	0·5312 (23)	0·0413 (25)	0·3195 (29)	13·4 (11)
C(10)	0·4089 (23)	−0·0722 (23)	0·2113 (28)	12·4 (10)
C(11)	0·2336 (11)	0·2079 (12)	−0·1549 (12)	5·6 (6)
C(12)	0·3059 (11)	0·1802 (12)	−0·1785 (12)	4·5 (6)
C(13)	0·3505 (19)	¼	−0·1913 (16)	4·9 (9)
C(14)	0·4262 (20)	¼	−0·2322 (24)	8·7 (15)
C(15)	0·3279 (19)	0·0903 (14)	−0·1964 (17)	8·9 (11)
C(16)	0·1654 (14)	0·1519 (20)	−0·1448 (19)	9·6 (11)
C(17)	−0·0233 (12)	0·2078 (13)	0·5878 (14)	6·1 (7)
C(18)	0·0500 (13)	0·1817 (14)	0·5664 (14)	5·8 (7)
C(19)	0·0941 (14)	¼	0·5509 (18)	4·1 (7)
C(20)	0·1750 (15)	¼	0·5352 (23)	6·2 (11)
C(21)	0·0831 (19)	0·0945 (14)	0·5686 (18)	9·1 (11)
C(22)	−0·0744 (18)	0·1494 (22)	0·6258 (23)	11·8 (14)
C(23)	−0·1890 (16)	0·2078 (8)	0·2259 (21)	8·6 (7)
C(24)	−0·2022 (14)	0·1778 (15)	0·3139 (19)	7·2 (5)
C(25)	−0·2169 (15)	¼	0·3686 (19)	4·3 (5)
C(26)	−0·262 (3)	¼	0·446 (4)	11·7 (13)
C(27)	−0·2084 (27)	0·0940 (28)	0·361 (3)	15·3 (13)
C(28)	−0·193 (5)	0·162 (4)	0·125 (4)	27·1 (28)
C(29)	0·5504 (24)	0·1788 (17)	0·018 (3)	14·9 (13)
C(30)	0·5924 (29)	0·1181 (27)	0·105 (3)	16·5 (15)
C(31)	−0·050 (3)	0·166 (3)	−0·029 (4)	16·6 (15)
C(32)	−0·035 (3)	0·109 (4)	−0·096 (4)	20·3 (19)

Table 2. Selected distances (Å) and angles (°)

Cp(1), Cp(2), Cp(3) and Cp(4) represent the ring centers of atom groups: C(1)–C(5); C(11), C(11'), C(12), C(12'), C(13); C(17), C(17'), C(18), C(18'), C(19); and C(23), C(23'), C(24), C(24'), C(25). Primed symbols are atoms in position $x, \tfrac{1}{2}-y, z$.

Cp(1)—Yb(1)	2·324	O(3)—Yb(3)	2·50 (3)
O(1)—Yb(1)	2·428 (9)	Cl(1)—Yb(3)	2·932 (6)
C(1)—Yb(1)	2·644 (18)	Cl(4)—Yb(3)	2·76 (3)
C(2)—Yb(1)	2·584 (17)	Cl(5)—Yb(3)	2·82 (3)
C(3)—Yb(1)	2·562 (16)	Cl(4)—Yb(4)	2·653 (5)
C(4)—Yb(1)	2·569 (17)	Cp(3)—Yb(4)	2·368
C(5)—Yb(1)	2·631 (20)	Cp(4)—Yb(4)	2·348
Cl(1)—Yb(1)	2·715 (4)	C(17)—Yb(4)	2·646 (17)
Cl(2)—Yb(1)	2·723 (4)	C(18)—Yb(4)	2·664 (18)
Cl(3)—Yb(1)	2·669 (4)	C(19)—Yb(4)	2·675 (20)
Cl(5)—Yb(1)	2·625 (4)	C(23)—Yb(4)	2·635 (26)
Cp(2)—Yb(2)	2·301	C(24)—Yb(4)	2·628 (24)
O(1)—Yb(2)	2·243 (15)	C(25)—Yb(4)	2·675 (24)
O(2)—Yb(2)	2·388 (18)	O(1)—Cl(1)	3·163 (15)
Cl(2)—Yb(2)	3·183 (6)	O(1)—Cl(2)	2·850 (15)
Cl(3)—Yb(2)	2·632 (5)	O(1)—Cl(3)	3·087 (10)
C(11)—Yb(2)	2·570 (16)	O(1)—Cl(5)	3·139 (11)
C(12)—Yb(2)	2·618 (15)	C(29)—O(2)	1·463 (14)
C(13)—Yb(2)	2·634 (19)	C(31)—O(3)	1·57 (5)

|			
Cl(1)—Yb(1)—Cp(1)	105·79	Cl(4)—Yb(3)—Cl(4)	77·61 (19)
Cl(2)—Yb(1)—Cp(1)	108·51	Cl(4)—Yb(3)—Cl(5)	88·74 (14)
Cl(3)—Yb(1)—Cp(1)	103·57	Cl(4)—Yb(3)—Cl(5)	165·84 (14)
Cl(5)—Yb(1)—Cp(1)	108·13	Cl(4)—Yb(3)—O(3)	100·5 (6)
O(1)—Yb(1)—Cp(1)	175·06	Cl(5)—Yb(3)—Cl(5)	104·60 (20)
Cl(1)—Yb(1)—Cl(2)	80·88 (14)	Cl(5)—Yb(3)—O(3)	85·5 (5)
Cl(1)—Yb(1)—Cl(3)	149·53 (13)	Cl(4)—Yb(4)—Cl(4)	81·53 (21)
Cl(1)—Yb(1)—Cl(5)	80·88 (16)	Cl(4)—Yb(4)—Cp(3)	106·08
Cl(1)—Yb(1)—O(1)	75·7 (3)	Cl(4)—Yb(4)—Cp(4)	107·47
Cl(2)—Yb(1)—Cl(3)	82·31 (16)	Cp(3)—Yb(4)—Cp(4)	135·20
Cl(2)—Yb(1)—Cl(5)	142·27 (15)	Yb(1)—Cl(1)—Yb(1)	86·81 (15)
Cl(2)—Yb(1)—O(1)	66·9 (3)	Yb(1)—Cl(1)—Yb(3)	98·79 (15)
Cl(3)—Yb(1)—Cl(5)	97·62 (15)	Yb(1)—Cl(2)—Yb(1)	86·49 (15)
Cl(3)—Yb(1)—O(1)	74·4 (3)	Yb(1)—Cl(2)—O(1)	51·60 (17)
Cl(5)—Yb(1)—O(1)	76·7 (4)	Yb(2)—Cl(2)—O(1)	43·2 (3)
Cl(2)—Yb(2)—Cp(2)	174·31	Yb(1)—Cl(3)—Yb(2)	90·66 (13)
Cl(3)—Yb(2)—Cp(2)	104·88	Yb(3)—Cl(4)—Yb(4)	100·43 (16)
O(1)—Yb(2)—Cp(2)	113·85	Yb(1)—Cl(5)—Yb(3)	103·87 (16)
O(2)—Yb(2)—Cp(2)	111·56	Yb(1)—O(1)—Yb(1)	100·4 (5)
Cl(3)—Yb(2)—Cl(3)	147·51 (18)	Yb(1)—O(1)—Yb(2)	107·6 (4)
Cl(3)—Yb(2)—O(1)	78·14 (11)	Yb(1)—O(1)—Cl(2)	61·5 (3)
Cl(3)—Yb(2)—O(2)	90·52 (16)	Yb(2)—O(1)—Cl(2)	76·3 (4)
O(1)—Yb(2)—O(2)	134·6 (6)	Yb(2)—O(2)—C(29)	119·7 (19)
Cl(1)—Yb(3)—Cl(4)	105·91 (13)	C(29)—O(2)—C(29)	106 (3)
Cl(1)—Yb(3)—Cl(5)	74·02 (11)	Yb(3)—O(3)—C(31)	116·9 (22)
Cl(1)—Yb(3)—O(3)	145·9 (8)	C(31)—O(3)—C(31)	125 (4)
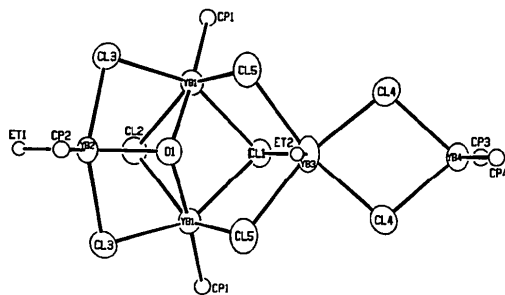
Fig. 1. *ORTEP* schematic drawing of the ytterbium complex. Pentamethylcyclopentadienes are represented by CP: CP1 [C(1)–C(10)], CP2 [C(11)–C(16)], CP3 [C(17)–C(22)] and CP4 [C(23)–C(28)]; ethyl ethers are represented by ET: ET1 [O(2), C(29), C(30)] and ET2 [O(3), C(31), C(32)].

Bridging Yb—O distances of 2·241 and 2·21 Å have been reported for $YbFe_2O_4$ (Kato, Kamada, Kimizuka & Katsura, 1975) and YbOCl (Brandt & Diehl, 1974) respectively. A comparable distance for Yb—Cp is 2·347 (2) Å in $(C_5Me_5)_2Yb(SC_6H_5)(NH_3)$ (Zalkin, Henly & Andersen, 1987).

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