Table 1. *Atomic coordinates*

$B_{eq} = \frac{8}{3}\pi^2 \text{ trace } | U_{ij} |$.

	x	y	z	B_{eq}(Å^2)
Au	0·5026 (1)	0·5067 (1)	0·2519 (1)	2·51 (2)
Br(1)	0·6395 (2)	0·3371 (2)	0·2460 (3)	3·8 (1)
Br(2)	0·3679 (2)	0·6778 (2)	0·2539 (3)	3·9 (1)
Br(3)	0·3130 (2)	0·4166 (2)	0·0922 (3)	3·8 (1)
Br(4)	0·6901 (2)	0·5954 (2)	0·4134 (3)	4·0 (1)
K	0·5181 (9)	0·9723 (6)	0·2510 (9)	6·9 (2)
O(1)	0·0613 (18)	0·6353 (12)	0·4770 (20)	4·8 (4)
O(2)	0·5512 (16)	0·8640 (12)	0·5341 (21)	4·9 (4)

Atomic coordinates are given in Table 1. Significant distances and angles are listed in Table 2.*

Related literature. Approximate but erroneous structure description by Cox & Webster (1936). The structure is different from that of K[AuCl$_4$].2H$_2$O (Théobald & Omrani, 1980) despite similar K—O distances, and O coordination by two K and several halogen atoms.

Table 2. *Interatomic distances* (Å) *and angles* (°)

Au—Br(1)	2·425 (2)	K—O(2)	2·73 (2)
Au—Br(2)	2·426 (2)	K—O(2)	2·79 (2)
Au—Br(3)	2·436 (2)	K—Br(1)	3·657 (8)
Au—Br(4)	2·422 (2)	K—Br(2)	3·819 (8)
K—O(1)	2·72 (2)	K—Br(3)	2·593 (8)
K—O(1)	2·82 (2)	K—Br(4)	3·539 (8)
Br(1)—Au—Br(2)	179·0 (1)	Br(2)—Au—Br(3)	90·3 (1)
Br(1)—Au—Br(3)	89·8 (1)	Br(2)—Au—Br(4)	89·8 (1)
Br(1)—Au—Br(4)	90·1 (1)	Br(3)—Au—Br(4)	179·3 (1)
Au—Au	4·216 (2); 4·270 (2)	K—K	4·288 (2); 4·325 (3)

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Diglyme Adduct of Dipotassium Bis(*tert*-butyl[8]annulene)ytterbate(II)

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Abstract. $[K(C_6H_{14}O_3)]_2[Yb(C_{12}H_{16})_2]$, $M_r = 840·12$, monoclinic, $P2_1/c$, $a = 10·292$ (4), $b = 20·588$ (6), $c = 20·036$ (6) Å, $\beta = 103·28$ (4)°, $V = 4131·9$ Å^3, $Z = 4$, $D_x = 1·350$ g cm^{-3}, D_m not measured, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 16·5$ cm^{-1}, $F(000) = 1728$, $T = 300$ K, $R = 0·032$ for 3212 unique reflections with $F^2 > 3\sigma(F^2)$. The Yb atom is sandwiched between two parallel [8]annulene rings (*tert*-butylcyclooctatetraene) with a mean Yb—C distance of 2·77 (4) Å. The K atoms cap the other sides of the rings at a mean K—C distance of 3·08 (5) Å; each K atom is also coordinated to four O atoms from the diglyme [bis(2-methoxyethyl) ether] ligands at distances that range from 2·86 to 3·06 Å.

Experimental. Title compound prepared by the reaction of *tert*-butylcyclooctatetraene with a solution of K and Yb metals in liquid ammonia in a manner similar to the unsubstituted compound (Kinsley, Streitwieser & Zalkin, 1985). Red, air-sensitive crystals, obtained by slow diffusion of hexane into a diglyme solution, were sealed inside quartz capillaries under Ar. Crystal 0·13 × 0·16 × 0·36 mm with six faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 32 reflections, 24° < 2θ < 42°; analytical absorption correction, range 1·19 to 1·39; max. $(\sin\theta)/\lambda = 0·54$ Å^{-1}, h −11 to 11, k 0 to 22, l −21 to 21; three standard reflections, 6% variation in intensities from average, intensities adjusted accordingly; 10 798 data, 5413 unique (including 2201, $F^2 < 3\sigma$), $R_{int} = 0·03$; structure solved by Patterson and Fourier methods; refined on F, 462 parameters; 14 cyclooctatetraene H atoms refined with isotropic thermal parameters, remaining 46 H atoms in cal-

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culated positions with fixed isotropic thermal parameters; anisotropic thermal parameters for non-H atoms; $R = 0{\cdot}076$ for 5413 data, $R = 0{\cdot}032$ for 3212 reflections for which $F^2 > 3\sigma$, $wR = 0{\cdot}038$, $S = 1{\cdot}34$; $w = [\sigma(F)]^{-2}$, $p = 0{\cdot}04$ in calculation of $\sigma(F^2)$; max. $(\text{shift}/\sigma) = 0{\cdot}03$; empirical extinction correction, $F_{\text{corr}} = (1 + 1{\cdot}65 \times 10^{-7}I)$; max. and min. of ΔF synthesis $0{\cdot}7$ and $-0{\cdot}8$ e Å^{-3}; atomic f for neutral Yb, K, O and C, and spherical bonded H from *International Tables for X-ray Crystallography* (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

Table 1. *Atomic parameters*

$$B_{\text{eq}} = \textstyle\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j/3.$$

	x	y	z	B_{eq}(Å^2)
Yb	0·04544 (3)	0·18694 (2)	0·24563 (2)	3·45 (1)
K(1)	0·34906 (19)	0·19628 (10)	0·44746 (8)	4·27 (5)
K(2)	−0·26406 (16)	0·18968 (10)	0·04465 (8)	4·05 (5)
O(1)	0·4529 (5)	0·3275 (3)	0·50844 (29)	5·2 (2)
O(2)	0·3451 (6)	0·2295 (3)	0·57997 (28)	5·3 (2)
O(3)	0·3212 (7)	0·0963 (4)	0·5509 (3)	6·6 (3)
O(4)	−0·3663 (5)	0·3170 (3)	−0·01679 (28)	4·9 (2)
O(5)	−0·2526 (6)	0·2208 (3)	−0·08799 (27)	4·6 (2)
O(6)	−0·2512 (7)	0·0862 (3)	−0·0605 (3)	6·0 (2)
C(1)	0·1471 (9)	0·1051 (5)	0·3523 (4)	3·9 (2)
C(2)	0·0682 (9)	0·1533 (6)	0·3786 (4)	4·2 (3)
C(3)	0·0602 (10)	0·2214 (6)	0·3804 (5)	4·5 (3)
C(4)	0·1246 (11)	0·2731 (6)	0·3546 (5)	4·4 (3)
C(5)	0·2251 (10)	0·2748 (5)	0·3177 (4)	3·9 (3)
C(6)	0·3045 (8)	0·2284 (5)	0·2949 (4)	3·5 (3)
C(7)	0·3165 (8)	0·1602 (5)	0·2945 (4)	3·6 (3)
C(8)	0·2532 (8)	0·1067 (4)	0·3175 (4)	3·2 (3)
C(9)	0·3093 (9)	0·0382 (4)	0·3015 (4)	4·6 (3)
C(10)	0·4548 (10)	0·0321 (5)	0·3429 (6)	8·2 (4)
C(11)	0·3052 (11)	0·0317 (5)	0·2245 (5)	6·9 (4)
C(12)	0·2329 (12)	−0·0209 (5)	0·3196 (6)	7·4 (4)
C(13)	−0·2241 (8)	0·1565 (5)	0·1990 (4)	3·8 (3)
C(14)	−0·2159 (8)	0·2258 (5)	0·1979 (4)	3·9 (3)
C(15)	−0·1388 (10)	0·2717 (5)	0·1732 (5)	4·3 (3)
C(16)	−0·0375 (10)	0·2681 (5)	0·1353 (5)	4·2 (3)
C(17)	0·0264 (9)	0·2162 (6)	0·1090 (4)	4·0 (3)
C(18)	0·0173 (9)	0·1479 (5)	0·1125 (4)	3·7 (3)
C(19)	−0·0568 (9)	0·1014 (5)	0·1401 (4)	3·7 (3)
C(20)	−0·1589 (9)	0·1022 (5)	0·1767 (4)	3·6 (3)
C(21)	−0·2120 (9)	0·0356 (4)	0·1947 (4)	4·5 (3)
C(22)	−0·3623 (10)	0·0285 (5)	0·1541 (5)	6·7 (4)
C(23)	−0·2045 (10)	0·0297 (5)	0·2712 (5)	6·7 (4)
C(24)	−0·1424 (11)	−0·0239 (5)	0·1726 (6)	6·7 (4)
C(25)	0·4333 (10)	0·3749 (5)	0·4539 (5)	6·5 (4)
C(26)	0·3666 (9)	0·3408 (5)	0·5541 (5)	5·3 (3)
C(27)	0·3941 (9)	0·2908 (5)	0·6097 (5)	5·4 (3)
C(28)	0·3351 (11)	0·1813 (6)	0·6293 (5)	6·8 (4)
C(29)	0·2546 (14)	0·1262 (6)	0·5956 (6)	7·7 (5)
C(30)	0·2563 (16)	0·0378 (7)	0·5240 (6)	9·9 (6)
C(31)	−0·3517 (10)	0·3642 (5)	0·0379 (5)	6·4 (4)
C(32)	−0·2846 (9)	0·3319 (4)	−0·0633 (5)	5·2 (3)
C(33)	−0·3034 (9)	0·2815 (5)	−0·1177 (5)	5·2 (3)
C(34)	−0·2393 (10)	0·1725 (5)	−0·1372 (4)	5·5 (3)
C(35)	−0·1686 (10)	0·1152 (5)	−0·1000 (5)	5·9 (3)
C(36)	−0·2009 (4)	0·0265 (6)	−0·0324 (5)	8·5 (5)
H(1)	0·126 (6)	0·060 (3)	0·368 (3)	2·1 (14)†
H(2)	0·014 (8)	0·130 (4)	0·406 (4)	5·5 (23)†
H(3)	−0·003 (9)	0·242 (4)	0·403 (4)	5·8 (26)†
H(4)	0·090 (12)	0·313 (6)	0·357 (6)	10·3 (43)†
H(5)	0·244 (8)	0·320 (4)	0·305 (4)	5·2 (23)†
H(6)	0·374 (8)	0·248 (4)	0·278 (3)	4·0 (20)†
H(7)	0·395 (8)	0·149 (4)	0·265 (4)	6·4 (22)†
H(8)	−0·301 (7)	0·144 (3)	0·214 (3)	3·3 (17)†
H(9)	−0·292 (8)	0·252 (4)	0·228 (4)	6·3 (22)†
H(10)	−0·159 (8)	0·310 (4)	0·180 (4)	4·1 (21)†
H(11)	−0·018 (7)	0·315 (4)	0·120 (4)	4·9 (19)†
H(12)	0·093 (9)	0·231 (4)	0·079 (5)	6·9 (25)†
H(13)	0·095 (9)	0·127 (4)	0·088 (4)	6·5 (24)†
H(14)	−0·027 (8)	0·052 (5)	0·136 (4)	6·6 (25)†

† Isotropically refined thermal parameters.

Atomic parameters are listed in Table 1,* and selected distances are listed in Table 2. Fig. 1 shows the molecule and numbering scheme.

Related literature. The structure of the title compound is similar to that of the dimethoxyethane (glyme) adducts of $K_2[Yb(C_8H_8)_2]$ and $K_2[Ca(C_8H_8)_2]$ (Kinsley, Streitwieser & Zalkin, 1985). In both structures the comparable distances and geometry are equivalent, and in both structures the [8]annulene rings are eclipsed. In the diglyme complex the K atom has four near O neighbors as opposed to the three in the glyme complex; but in both cases one of the O neighbors is from an adjacent ligand to produce a network of connected molecules.

Since Ce^{III} and Yb^{II} have comparable ionic radii (Shannon, 1976) there are also similarities of the above glyme and diglyme complexes to $[K(C_6H_{14}O_3)]$-$[Ce(C_8H_8)_2]$, the diglyme complex of $KCe(C_8H_8)_2$

Table 2. *Selected distances* (Å)

Yb—C(1)	2·733 (8)	Yb—C(19)	2·767 (8)
Yb—C(2)	2·710 (8)	Yb—C(20)	2·836 (8)
Yb—C(3)	2·762 (10)	Yb—Ring 1*	2·054
Yb—C(4)	2·786 (10)	Yb—Ring 2*	2·056
Yb—C(5)	2·747 (9)	K(1)—Ring 1*	2·456
Yb—C(6)	2·758 (8)	K(2)—Ring 2*	2·468
Yb—C(7)	2·791 (8)	K(1)—O(1)	3·056 (6)
Yb—C(8)	2·821 (7)	K(1)—O(2)	2·751 (6)
Yb—C(13)	2·788 (8)	K(1)—O(3)	2·981 (6)
Yb—C(14)	2·758 (8)	K(1)—O(4)†	2·865 (6)
Yb—C(15)	2·735 (8)	K(2)—O(1)†	2·857 (6)
Yb—C(16)	2·746 (8)	K(2)—O(4)	2·984 (7)
Yb—C(17)	2·766 (8)	K(2)—O(5)	2·762 (6)
Yb—C(18)	2·737 (8)	K(2)—O(6)	3·020 (7)

* Ring 1 and Ring 2 are [8]annulene rings C(1)—C(8) and C(13)—C(20) respectively.

† Atom at $x, \frac{1}{2}-y, \frac{1}{2}+z$.

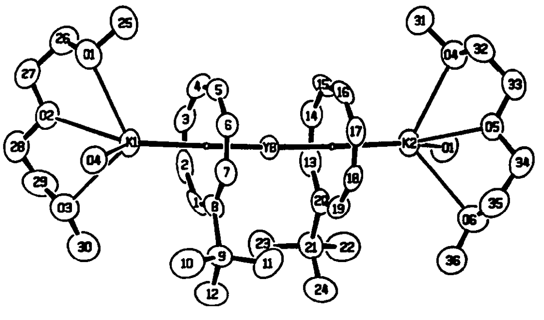

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule showing the atomic numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positions, deviations from least-squares planes, distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42883 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Hodgson & Raymond, 1972). This relationship is discussed in the paper reporting the structure of $[K(C_4H_{10}O_2)]_2[Yb(C_8H_8)_2]$ (Kinsley, Streitwieser & Zalkin, 1985).

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Structure of Dichlorobis(pyridine *N*-oxide)zinc(II)

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Abstract. $[ZnCl_2(C_5H_5NO)_2]$, $M_r = 326.5$, orthorhombic, $Fdd2$, $a = 12.318\,(4)$, $b = 28.176\,(6)$, $c = 7.268\,(2)$ Å, $V = 2522.5\,(7)$ Å^3, $Z = 8$, $D_x = 1.72$, $D_m = 1.70$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 24.1$ cm^{-1}, $F(000) = 1312$, $T = 297$ K. Final $R = 0.019$ for 599 observed reflections. The $[ZnCl_2(C_5H_5NO)_2]$ molecule has a distorted tetrahedral geometry (crystallographically required C_2) with Zn—O and Zn—Cl distances of 1·992 (2) and 2·214 (1) Å respectively. The pyridine rings are planar within 0·009 (3) Å.

Experimental. The title complex was prepared from approximately 1:1 molar ratios of anhydrous zinc dichloride and pyridine *N*-oxide in methanol/ethanol mixtures and recrystallized from the same solvents. Diffractometer used: Enraf–Nonius CAD-4 equipped with graphite monochromator. Cell dimensions: from 15 reflections in range $22 \leq 2\theta \leq 30°$. Density measured by flotation. Crystal dimensions: ~0·3 × 0·25 × 0·4 mm. Crystal faces not readily indexed, and therefore no absorption corrections made; estimate of maximum relative error in intensity due to absorption *ca* 12%. Total of 606 independent reflections (*h*, 0–14; *k*, 0–33; *l*, 0–8) measured in range $\sin\theta/\lambda \leq 0.6$ Å^{-1} using ω–2θ scan technique. Four standards monitored, no variation with time. Standard deviations assigned as $\sigma(I) = [\sigma^2_{\text{count}} + (0.04I)^2]^{1/2}$; 599 reflections with $F_o \geq 2\sigma(F_o)$ used for refinement. Structure solved by Patterson and Fourier methods: least-squares refinement (based on F) carried out using *LINEX*, a modified version of *ORFLS* (Busing, Martin & Levy, 1962); function minimized $\sum w(|F_o| - |F_c|)^2$ with weights $w = [2LpF_o/\sigma(I)]^2$. Pyridine-ring H atoms included as fixed contributions with C—H set at 0·98 Å. Initial refinement with all *hkl* indices positive gave $R = 0.035$, but with an unsatisfactory goodness of fit. Since $Fdd2$ is a polar space group, the refinement was continued with all indices reassigned with negative values, and this converged at $R = 0.019$, $wR = 0.028$ and $S = 1.13$ for the 77 variables refined. At convergence all $\Delta p_i < 0.02 \times \sigma(p_i)$ and maximum and minimum values in final difference map 0·40 and −0·55 e Å^{-3}. Atomic scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); anomalous-dispersion corrections for Zn and Cl included (Cromer & Liberman, 1970). Calculations carried out on a CDC-Cyber 175 computer. Atomic coordinates are listed in Table 1* and bond distances and angles in Table 2. An *ORTEP* view (Johnson, 1965) of the molecule is given in Fig. 1.

Related literature. Structures related to the present compound: $[CuCl_2(C_5H_5NO)_2]_2$ (Morrow, 1974), $[CuCl_2(C_5H_5NO)]_2$ (Estes & Hodgson, 1976) and $[ZnI_2(C_5H_5NO)_2]$ (Sawitzki & von Schnering, 1974).

We thank the University Computation Center for a generous allocation of computer time.

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* Lists of anisotropic thermal parameters, observed and calculated structure-factor amplitudes, H-atom coordinates and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42858 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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