

2.3×10^{-4} ; max. $\Delta\rho = 4.7$, min. $\Delta\rho = -4.6 \text{ e } \text{Å}^{-3}$. Scattering factors of neutral atoms (Cromer & Mann, 1968) were used and corrected for anomalous dispersion (Cromer & Liberman, 1970).

The atomic coordinates are listed in Table 1, bond lengths in Table 2; Fig. 1 displays a perspective view of the unit-cell contents.

Related literature. The present paper is the third part of a series (part I: Krämer, 1983; part II: Krämer & Reis, 1986) on the study of lead indium bismuth chalcogenides.

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computer of the Rechenzentrum der Universität Freiburg.

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Structure of Potassium Tetrabromoaurate(III) Dihydrate

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Abstract. $\text{K}[\text{AuBr}_4] \cdot 2\text{H}_2\text{O}$, $M_r = 591.71$, monoclinic, $P2_1/n$, $a = 9.579$ (1), $b = 12.006$ (1), $c = 8.480$ (1) Å, $\beta = 94.31$ (5)°, $V = 972.5$ Å³, $Z = 4$, $D_x = 4042$ kg m⁻³, $\lambda = \text{Mo } K\alpha = 0.7107$ Å, $\mu = 31.069$ mm⁻¹, $F(000) = 1032$, $T = 291$ K, $R = 0.0406$ for 1755 unique reflections with $F > \sigma(F)$. The structure is significantly different from that of $\text{K}[\text{AuCl}_4] \cdot 2\text{H}_2\text{O}$ [Théobald & Omrani (1980). *Acta Cryst.* **B36**, 2932–2935]. The AuBr_4^- ion is almost square planar and symmetry about the Au atoms is almost centrosymmetric, despite the fact that the Au atoms do not lie in the special positions, Au(1) 000 and Au(2) $\frac{1}{2}\frac{1}{2}0$, previously reported by Cox & Webster [*J. Chem. Soc.* (1936), pp. 1635–1637].

Experimental. Platelets crystallized from aqueous solution, $0.20 \times 0.06 \times 0.03$ mm. Philips PW 1100

diffractometer. Bisecting geometry. Cell parameters from 25 reflections with $10 < \theta < 16^\circ$. Absorption correction according to *DIFABS* (Walker & Stuart, 1983), min. absolute correction 0.767, max. 1.367. $0 \leq h \leq 17$, $0 \leq k \leq 19$, $-12 \leq l \leq 12$, $3.5 < \theta < 40^\circ$. Three intensity standards: $hkl = 422$, 060, 200; $\Delta I/I < 0.03$. 1840 reflections measured, 1755 unique, $R_{\text{int}} = 0.020$. About 4500 weak reflections. Location of Au and Br by *MULTAN* (Main *et al.*, 1980); K and O located by Fourier difference mapping (*SHELX*; Sheldrick, 1976). Refinement of F . The planarity of the AuBr_4^- group helped eliminate erroneous peaks appearing in maps owing to the coordinates of Au that were almost in the special positions. H atoms not considered. Unit weights, $R = 0.0406$. Max. shift/e.s.d. ratio < 0.01 , largest peaks in final difference Fourier synthesis $0.32 \text{ e } \text{Å}^{-3}$. Scattering factors f' and f'' from *International Tables for X-ray Crystallography* (1974).

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Table 1. Atomic coordinates

	$B_{eq} = \frac{2}{3}\pi^2 \text{trace } U_{ij} $			
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
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)

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(1)	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(2)	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)

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 $\text{K}[\text{AuCl}_4]\cdot 2\text{H}_2\text{O}$ (Théobald & Omrani, 1980) despite similar K—O distances, and O coordination by two K and several halogen atoms.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42944 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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Abstract. $[\text{K}(\text{C}_6\text{H}_{14}\text{O}_3)]_2[\text{Yb}(\text{C}_{12}\text{H}_{16})_2]$, $M_r = 840.12$, monoclinic, $P2_1/c$, $a = 10.292$ (4), $b = 20.588$ (6), $c = 20.036$ (6) Å, $\beta = 103.28$ (4)°, $V = 4131.9$ Å³, $Z = 4$, $D_x = 1.350$ g cm⁻³, D_m not measured, $\lambda(\text{Mo } K\alpha) = 0.71073$ 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 $0.13 \times 0.16 \times 0.36$ mm with six faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 32 reflections, $24^\circ < 2\theta < 42^\circ$; 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, $F^2 < 3\sigma$), $R_{\text{int}} = 0.03$; 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-