Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A potassium crown ether complex with dichloroaurate(I)

Md. Alamgir Hossain, Douglas R. Powell and Kristin Bowman-James*

Department of Chemistry, University of Kansas, Malott Hall, Room 2010, 1251 Wescoe Hall Dr., Lawrence, KS 66045-7582, USA

Correspondence e-mail: kbowman-james@ku.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.017
wR factor = 0.047
Data-to-parameter ratio = 27.2

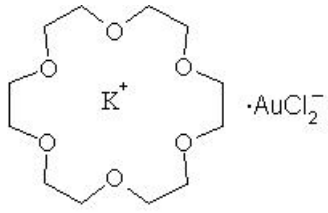
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 December 2002
Accepted 7 January 2003
Online 17 January 2003

The title compound, (1,4,7,10,13,16-hexaoxacyclooctane)-potassium dichloroaurate(I), $[K(C_{12}H_{24}O_6)][AuCl_2]$, consists of potassium ion encapsulated by the 18-membered crown ether 1,4,7,10,13,16-hexaoxacyclooctane and a linear dichloroaurate(I) monoanion. The potassium occupies a crystallographic center of symmetry with a ring coordination number of six, and two chlorides in axial sites at a distance of 3.2306 (5) Å. The linear anionic species sits on another crystallographic center of symmetry.

Comment

Although cations are the dominant species in the coordination chemistry of crown ethers, the counter-anions can also play an important role in the binding mode, cation–anion pairing, and the geometry of the structure (Bajaj & Poonia, 1988). Current interests of a number of researchers are thus also focused on the coordination chemistry of anions (Bianchi *et al.*, 1997). Our interests in this area involve exploring dual host receptors (Kavallieratos *et al.*, 2000; Qian *et al.*, 2001), extending previous work to anionic metal complexes.



(I)

The formula unit of (I) is shown in Fig. 1. The linear dichloroaurate(I) and the 18-crown-6 potassium complex form infinite alternating cation/anion layers, separated by 3.9618 (2) Å, along the *b* axis (Fig. 2).

The crown ether adopts D_{3d} symmetry, as observed in most other complexes of 18-crown-6 (Dunitz *et al.*, 1974). The potassium ion is located at the center of the crown and is coordinated to the six O atoms with an average K—O bond distance of 2.810 Å, which is consistent with previously reported results (2.801 Å; Seiler *et al.*, 1974). The linear dichloroaurate anions sit above and below the plane of the crown ether, with one of the Cl atoms closer to the potassium [3.2306 (5) Å] than is the gold [3.9618 (2) Å]. The presence of two chlorides approximately above and below the potassium ion thus completes a pseudo-hexagonal bipyramidal coordination sphere for the alkali metal ion.

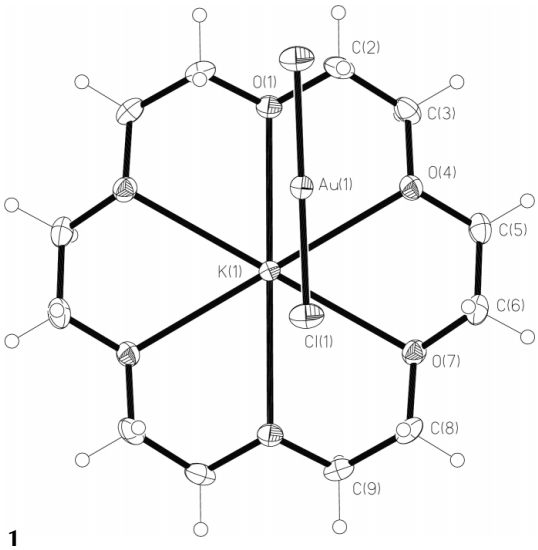


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 50% probability level.

Experimental

Equimolar amounts (0.065 mmol each) of 18-crown-6 and potassium tetrachloroaurate(III) were dissolved in methanol. Single crystals of the reduced gold complex with the potassium crown ether complex were grown by diffusion of diethyl ether into the methanolic solution of the mixture. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 3.66 (*t*, CH_2). ^{13}C NMR (125 MHz, $CDCl_3$, TMS): δ 70.4 (CH_2). MS (FAB): *m*/*z* 303 $(18C6 + K)^+$.

Crystal data

$[K(C_{12}H_{24}O_6)][AuCl_2]$	D_x = 2.034 Mg m^{-3}
M_r = 571.28	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 4929 reflections
a = 8.7583 (4) Å	θ = 3.0–30.5°
b = 7.9237 (4) Å	μ = 8.42 mm^{-1}
c = 13.8393 (6) Å	T = 100 (2) K
β = 103.785 (2)°	Prism, colorless
V = 932.76 (8) Å^3	0.31 × 0.16 × 0.11 mm
Z = 2	

Data collection

Bruker APEX diffractometer	2800 independent reflections
ω scans	2470 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996; Blessing, 1995)	R_{int} = 0.019
	θ_{max} = 30.5°
T_{min} = 0.180, T_{max} = 0.458	$h = -11 \rightarrow 12$
7528 measured reflections	$k = -10 \rightarrow 11$
	$l = -19 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2 + 0.1888P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.017	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.047	$\Delta\rho_{max}$ = 1.66 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −1.15 e Å^{-3}
2800 reflections	
103 parameters	
H-atom parameters constrained	

Crystal decay was determined by remeasuring the first 50 frames of data at the end of data collection and comparing the intensities from the first and last runs. All H atoms were constrained with a riding model. Residual peaks > 0.5 e Å^{-3} and troughs < 0.5 e Å^{-3} were near the Au and K atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

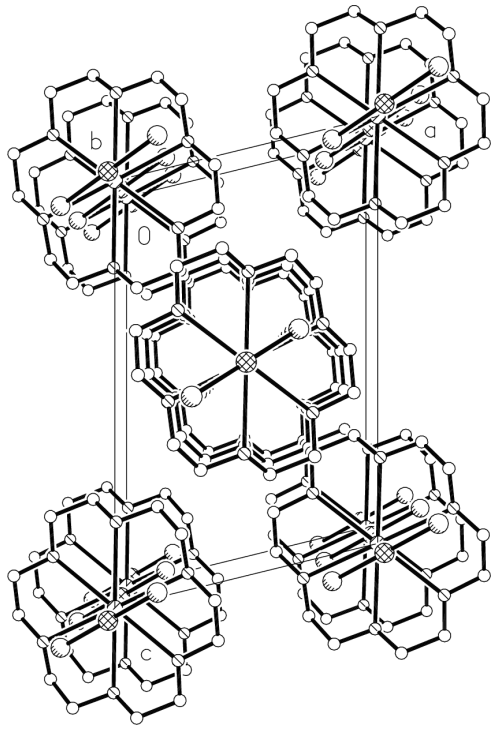


Figure 2
A packing diagram of (I). H atoms have been omitted for clarity.

This research was sponsored by the Environmental Management Science Program, Offices of Science and Environment Management, US Department of Energy, under grant DE-FG-96ER62307. The authors thank the National Science Foundation (CHE-0079282) and the University of Kansas for funds to acquire the diffractometer and computers used in this study.

References

Bajaj, A. V. & Poonia, N. S. (1988). *Coord. Chem. Rev.* **87**, 55–213.
Bianchi, A., Garcia-Espana, E. & Bowman-James, K. (1997). Editors. *Supramolecular Chemistry of Anions*. New York: Wiley.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dunitz, J. D., Dobler, M., Seiler, P. & Phizackerley, R. P. (1974). *Acta Cryst.* B**30**, 2733–2738.
Kavallieratos, K., Danby, A., VanBerkel, G. J., Kelly, M. A., Sachleben, R. A., Moyer, B. A. & Bowman-James, K. (2000). *Anal. Chem.* **72**, 5258–5264.
Qian, Q., Wilson, G. S., Bowman-James, K. & Girault, H. H. (2001). *Anal. Chem.* **73**, 497–503.
Seiler, P., Dobler, M. & Dunitz, J. D. (1974). *Acta Cryst.* B**30**, 2744–2745.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.