

## References

- B. A. FRENZ & ASSOCIATES, INC. (1983). *SDP-Plus*. College Station, Texas 77840, USA, and Enraf-Nonius, Delft, The Netherlands.
- BOULD, J., CROOK, J. E., GREENWOOD, N. N. & KENNEDY, J. D. (1983). *J. Chem. Soc. Chem. Commun.* pp. 949–950.
- BOULD, J., GREENWOOD, N. N., KENNEDY, J. D. & McDONALD, W. S. (1982). *J. Chem. Soc. Chem. Commun.* pp. 465–467.
- CROOK, J. E., GREENWOOD, N. N., KENNEDY, J. D. & McDONALD, W. S. (1981). *J. Chem. Soc. Chem. Commun.* pp. 933–934.
- DELANEY, M. S., KNOBLER, C. B. & HAWTHORNE, M. F. (1981). *Inorg. Chem.* **20**, 1341–1347.
- DELANEY, M. S., TELLER, R. G. & HAWTHORNE, M. F. (1981). *J. Chem. Soc. Chem. Commun.* pp. 235–236.
- FARIDOON, FERGUSON, G., FONTAINE, X. L. R., KENNEDY, J. D., NI DHUBHGHAILL, O., REED, D. & SPALDING, T. R. (1988). In preparation.
- FARIDOON, FERGUSON, G., FONTAINE, X. L. R., KENNEDY, J. D. & SPALDING, T. R. (1988). In preparation.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5315. Oak Ridge National Laboratory, Tennessee, USA.
- KENNEDY, J. D. (1986). *Prog. Inorg. Chem.* **34**, 211–434.
- WADE, K. (1976). *Adv. Inorg. Chem. Radiochem.* **18**, 1–66.

*Acta Cryst.* (1988). **C44**, 1374–1376

## Structures of Alkalides and Electrides. I. Structure of Potassium Cryptand[2.2.2]\* Electride

BY DONALD L. WARD,† RUI H. HUANG AND JAMES L. DYE

*Department of Chemistry and Center for Fundamental Materials Research, Michigan State University,  
East Lansing, Michigan 48824-1322, USA*

(Received 12 December 1987; accepted 9 March 1988)

**Abstract.** K<sup>+</sup>.C<sub>18</sub>H<sub>36</sub>N<sub>2</sub>O<sub>6</sub>.e<sup>-</sup>,  $M_r = 415.60$ , monoclinic,  $C2/c$ ,  $a = 12.129(8)$ ,  $b = 20.692(13)$ ,  $c = 21.519(16)$  Å,  $\beta = 95.23(6)^\circ$ ,  $V = 5378(6)$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.026$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 2.2$  cm<sup>-1</sup>,  $F(000) = 1800$ ,  $T = 202(2)$  K; final  $R = 0.041$  for 3614 [ $I > 3\sigma(I)$ ] of 6394 unique reflections. The structure confirmed the primary feature of electrides: that they consist of complexed cations and noise-level electron density in the space between the cations. The average K<sup>+</sup>–O distance is 2.827(28) Å.

**Introduction.** Electrides are those ionic compounds in which trapped electrons serve as the anions (Dye, 1984, 1987). The first crystal structure of an electride, Cs<sup>+</sup>(18-crown-6)<sub>2</sub>.e<sup>-</sup>, was determined in 1985 (Dawes, Ward, Huang & Dye, 1986). Cs<sup>+</sup>(18-crown-6)<sub>2</sub>.e<sup>-</sup> represents one type of electride in which trapped electrons are isolated and the interaction between the electrons is weak. The optical spectrum and powder conductivity indicate electron-trapping energies of at least 0.5 eV. K<sup>+</sup>(cryptand[2.2.2]).e<sup>-</sup>, on the other hand, is a different type of electride in which trapped electrons interact with each other strongly enough to give extensive electron pairing. In this case the optical spectrum and conductivity measurement suggest weak binding of the electron pair. The temperature depen-

dence of the magnetic susceptibility indicates a spin-pairing energy of ~0.05 eV (Huang, Faber, Moegenborg, Ward & Dye, 1988). Structure determination is essential for understanding the properties of this electride.

**Experimental.** Single crystals were obtained by recrystallization from dimethyl ether–diethyl ether solutions using temperature scanning. The dry crystals were transferred onto a cold copper block (at 223 K) in a nitrogen glove bag and covered with purified octane. A suitable single crystal was picked up by a glass fiber with grease on its tip and transferred under a cold nitrogen stream (213 K) onto the diffractometer. During data collection the crystal was kept in a cold (203 K) nitrogen stream.

The crystal used for data collection was plate-shaped, dark gray in color, and had approximate dimensions 0.20 × 0.60 × 0.80 mm. The diffractometer was a Nicolet P3F using graphite-monochromatized Mo  $K\alpha$  radiation and a locally modified Nicolet LT-1 low-temperature system. Unit-cell parameters were determined by least squares from the setting angles of 14 reflections in the range  $7.5 < \theta < 10^\circ$ . Intensity data were collected using  $\theta$ – $2\theta$  scans at  $4^\circ \text{ min}^{-1}$  (in  $2\theta$ ) to  $(\sin\theta)/\lambda = 0.5385 \text{ \AA}^{-1}$ ; reflection indices:  $0 \leq h \leq 15$ ,  $0 \leq k \leq 26$ , and  $-28 \leq l \leq 27$ ; 6692 total data. A linear decay correction was based on the intensities of three monitor reflections (5.7%

\* 4,7,13,16,21,24-Hexaoxa-1,10-diazabicyclo[8.8.8]hexacosane.

† To whom correspondence should be addressed.

Table 1. Positional and isotropic thermal parameters and their e.s.d.'s for potassium cryptand[2.2.2] electride

H atoms were refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:  $\frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$ .

	x	y	z	$B_{eq}/B_{iso}(\text{\AA}^2)$
K1	0.75630 (4)	0.08333 (3)	0.04166 (3)	3.227 (9)
O4	0.8032 (2)	-0.03005 (9)	-0.0224 (1)	4.42 (4)
O7	0.8697 (2)	-0.0209 (1)	0.1070 (1)	4.99 (5)
O13	0.5596 (1)	0.0827 (1)	0.10540 (8)	4.09 (4)
O16	0.5516 (1)	0.11103 (9)	-0.02329 (8)	3.34 (4)
O21	0.8879 (1)	0.16757 (8)	-0.02146 (9)	3.61 (4)
O24	0.8695 (2)	0.1868 (1)	0.10733 (9)	4.57 (5)
N1	0.7364 (2)	0.0833 (1)	-0.0974 (1)	3.56 (4)
N10	0.7759 (2)	0.0831 (2)	0.1797 (1)	5.26 (6)
C2	0.7343 (2)	0.0165 (2)	-0.1199 (1)	4.53 (7)
C3	0.8205 (2)	-0.0266 (1)	-0.0863 (2)	5.07 (7)
C5	0.8767 (2)	-0.0752 (2)	0.0101 (2)	5.71 (8)
C6	0.8512 (2)	-0.0805 (2)	0.0757 (2)	6.02 (8)
C8	0.8543 (3)	-0.0265 (2)	0.1722 (2)	6.89 (9)
C9	0.8624 (3)	0.0376 (2)	0.2025 (2)	6.31 (9)
C11	0.6691 (3)	0.0626 (2)	0.2019 (2)	6.9 (1)
C12	0.5712 (3)	0.0980 (2)	0.1699 (2)	6.36 (9)
C14	0.4735 (2)	0.1199 (1)	0.0736 (1)	3.77 (6)
C15	0.4546 (2)	0.0989 (1)	0.0079 (1)	3.71 (6)
C17	0.5338 (2)	0.0960 (1)	-0.0880 (1)	3.90 (6)
C18	0.6332 (2)	0.1168 (2)	-0.1203 (1)	4.04 (6)
C19	0.8318 (2)	0.1180 (2)	-0.1197 (1)	4.13 (6)
C20	0.8600 (2)	0.1800 (1)	-0.0858 (1)	4.15 (6)
C22	0.9254 (2)	0.2245 (1)	0.0109 (2)	4.42 (7)
C23	0.9610 (2)	0.2094 (1)	0.0771 (2)	4.61 (7)
C25	0.8990 (3)	0.1795 (2)	0.1724 (2)	6.42 (9)
C26	0.8048 (3)	0.1487 (2)	0.2025 (2)	6.6 (1)
H2a	0.745 (2)	0.014 (1)	-0.164 (1)	2.1 (6)
H2b	0.665 (2)	-0.003 (1)	-0.114 (1)	1.8 (6)
H3a	0.812 (2)	-0.068 (1)	-0.103 (1)	2.2 (6)
H3b	0.894 (2)	-0.012 (1)	-0.090 (1)	2.7 (7)
H5a	0.951 (2)	-0.060 (1)	0.005 (1)	2.5 (7)
H5b	0.864 (2)	-0.118 (1)	-0.010 (1)	3.0 (7)
H6a	0.778 (2)	-0.095 (1)	0.081 (1)	3.2 (7)
H6b	0.893 (2)	-0.111 (1)	0.100 (1)	2.8 (7)
H8a	0.905 (3)	-0.056 (2)	0.192 (2)	5.1 (9)
H8b	0.785 (3)	-0.049 (2)	0.173 (2)	5 (1)
H9a	0.861 (3)	0.031 (2)	0.251 (1)	3.6 (8)
H9b	0.936 (2)	0.057 (1)	0.195 (1)	2.6 (7)
H11a	0.667 (2)	0.071 (2)	0.248 (1)	3.9 (8)
H11b	0.658 (3)	0.013 (2)	0.197 (2)	7 (1)
H12a	0.501 (3)	0.084 (2)	0.191 (1)	4.3 (8)
H12b	0.573 (3)	0.143 (2)	0.176 (1)	4.1 (8)
H14a	0.493 (2)	0.167 (1)	0.076 (1)	1.5 (6)
H14b	0.406 (2)	0.116 (1)	0.096 (1)	1.3 (5)
H15a	0.436 (2)	0.053 (1)	0.003 (1)	1.0 (5)
H15b	0.394 (2)	0.127 (1)	-0.014 (1)	1.3 (5)
H17a	0.472 (2)	0.119 (1)	-0.105 (1)	0.7 (5)
H17b	0.514 (2)	0.049 (1)	-0.092 (1)	1.0 (5)
H18a	0.617 (2)	0.109 (1)	-0.167 (1)	2.7 (7)
H18b	0.644 (2)	0.165 (1)	-0.114 (1)	1.6 (6)
H19a	0.819 (2)	0.127 (1)	-0.166 (1)	1.4 (6)
H19b	0.894 (2)	0.089 (1)	-0.114 (1)	1.7 (6)
H20a	0.921 (2)	0.202 (1)	-0.104 (1)	1.7 (6)
H20b	0.801 (2)	0.212 (1)	-0.092 (1)	1.2 (5)
H22a	0.867 (2)	0.255 (1)	0.007 (1)	2.1 (6)
H22b	0.984 (2)	0.242 (1)	-0.009 (1)	2.4 (6)
H23a	1.017 (2)	0.175 (1)	0.080 (1)	2.1 (6)
H23b	0.990 (2)	0.249 (1)	0.099 (1)	2.4 (6)
H25a	0.923 (3)	0.223 (2)	0.192 (2)	5.0 (9)
H25b	0.961 (2)	0.151 (2)	0.177 (1)	3.5 (8)
H26a	0.824 (3)	0.149 (2)	0.251 (2)	4.7 (9)
H26b	0.738 (3)	0.179 (2)	0.193 (2)	4.4 (8)

decrease in average intensity). An empirical absorption correction was applied with a factor range of 0.919 to 0.999. Reflection averaging led to  $R_{int} = 1.7\%$  and 6394 unique data. Taking a data cut-off of  $I > 3\sigma(I)$ , there are 3614 observed and 2780 unobserved data. The structure was solved by direct methods. Full-matrix least-squares refinement was on  $F$  with non-H atoms

Table 2. Bond distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) for potassium cryptand[2.2.2] electride

Numbers in parentheses are e.s.d.'s in the least-significant digits.

K1	O4	2.805 (2)	O24	C23	1.417 (4)		
K1	O7	2.857 (2)	O24	C25	1.421 (4)		
K1	O13	2.858 (2)	N1	C2	1.464 (4)		
K1	O16	2.795 (2)	N1	C18	1.476 (3)		
K1	O21	2.798 (2)	N1	C19	1.479 (4)		
K1	O24	2.847 (2)	N10	C9	1.462 (5)		
K1	N1	2.981 (2)	N10	C11	1.483 (4)		
K1	N10	2.958 (2)	N10	C26	1.474 (5)		
O4	C3	1.412 (4)	C2	C3	1.508 (4)		
O4	C5	1.429 (4)	C5	C6	1.477 (6)		
O7	C6	1.414 (4)	C8	C9	1.478 (6)		
O7	C8	1.437 (4)	C11	C12	1.507 (5)		
O13	C12	1.418 (4)	C14	C15	1.478 (4)		
O13	C14	1.422 (3)	C17	C18	1.508 (4)		
O16	C15	1.428 (3)	C19	C20	1.499 (4)		
O16	C17	1.424 (3)	C22	C23	1.483 (5)		
O21	C20	1.418 (3)	C25	C26	1.506 (5)		
O21	C22	1.421 (3)					
C3	O4	C5	111.6 (2)	O7	C6	C5	110.6 (3)
C6	O7	C8	111.4 (3)	O7	C8	C9	110.4 (3)
C12	O13	C14	110.7 (2)	N10	C9	C8	114.4 (3)
C15	O16	C17	111.3 (2)	N10	C11	C12	112.8 (3)
C20	O21	C22	111.4 (2)	O13	C12	C11	110.0 (3)
C23	O24	C25	110.5 (2)	O13	C14	C15	110.1 (2)
C2	N1	C18	110.1 (2)	O16	C15	C14	109.6 (2)
C2	N1	C19	110.0 (2)	O16	C17	C18	109.4 (2)
C18	N1	C19	109.2 (2)	N1	C18	C17	113.7 (2)
C9	N10	C11	109.2 (3)	N1	C19	C20	114.0 (2)
C9	N10	C26	109.8 (3)	O21	C20	C19	110.2 (2)
C11	N10	C26	110.2 (3)	O21	C22	C23	110.4 (2)
N1	C2	C3	114.2 (2)	O24	C23	C22	109.8 (2)
O4	C3	C2	109.9 (3)	O24	C25	C26	109.6 (3)
O4	C5	C6	109.9 (3)	N10	C26	C25	114.4 (3)

refined anisotropically, H atoms isotropically, 388 total parameters,  $w = 1.0$ , and the function minimized was  $\sum w(|F_o| - |F_c|)^2$ . After the final cycle of refinement, the maximum  $\Delta/\sigma = 0.03$ ,  $R = 0.041$ ,  $wR = 0.040$ , and  $S = 0.96$ . The final difference-map peak heights ranged from  $-0.11$  (1) to  $0.10$  (1)  $e\text{\AA}^{-3}$ . Scattering factors were from Cromer & Waber (1974) and  $f'$  and  $f''$  from Cromer (1974). All computations were carried out on a VAX 11/750 computer using the *SDP* (Frenz, 1978) software system.

**Discussion.** The refined atomic coordinates and the isotropic temperature factors are presented in Table 1; bond distances and bond angles are given in Table 2.\* The molecular structure and the numbering of the atoms are shown in Fig. 1 and the stereographic packing diagram is shown in Fig. 2.

The structure determination confirmed the primary feature of electrides: that they consist of complexed cations and noise-level electron density in the space between them. The complexed cations are very similar to those in  $K^+(\text{cryptand}[2.2.2])I^-$  (Moras, Metz &

\* Lists of structure factors and tables of anisotropic thermal parameters, least-squares planes, torsion angles, r.m.s. amplitudes of thermal vibration, and bond distances and angles involving H atoms, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44845 (83 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

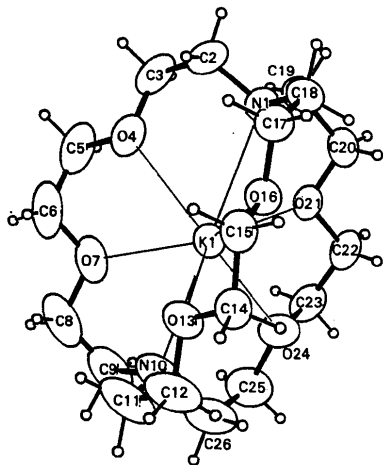


Fig. 1. The molecular structure and the numbering of the atoms [ORTEP (Johnson, 1965), 50% probability ellipsoids].

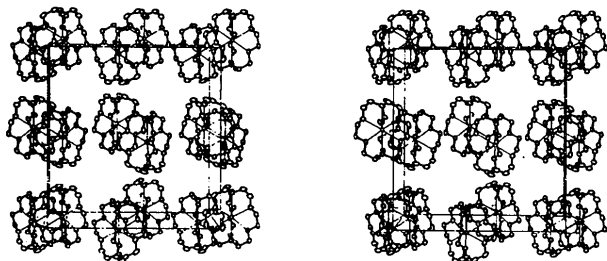


Fig. 2. Stereographic packing diagram [ORTEP (Johnson, 1965), 20% probability ellipsoids]. The *c* axis is vertical, the *b* axis is horizontal and the *a\** axis is normal to the page.

Weiss, 1973). The average  $K^+—O$  distance is 2.827(28) Å (2.785 Å in  $K^+$ (cryptand[2.2.2]).I<sup>-</sup>).

In order to study the correlation between the structure and the properties of electriles we must

investigate the shape and the distribution of the 'empty' space between the complexed cations. This has been done by using an Evans and Sutherland PS-300 display system in which the van der Waals radii of atoms were used to illustrate the molecular packing. It has been shown (Huang *et al.*, 1988) that the 'empty' space in this electrile forms two-dimensional channels almost parallel to the *a* axis and at 29° to the *c* axis. These two kinds of channels intersect to form dumbbell-shaped cavities and thus allow the trapped electrons to interact strongly with each other.

The authors gratefully acknowledge the support of this work by National Science Foundation grant DMR 84-14154. The X-ray diffractometer system was provided by NSF grant CHE-8403823.

#### References

- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- DAWES, S. B., WARD, D. L., HUANG, R. H. & DYE, J. L. (1986). *J. Am. Chem. Soc.* **108**, 3534–3535.
- DYE, J. L. (1984). *Prog. Inorg. Chem.* **32**, 327–441.
- DYE, J. L. (1987). *Sci. Am.* **257**, 66–75.
- FRENZ, B. A. (1978). *The Enraf-Nonius CAD-4 SDP-A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution*. In *Computing in Crystallography*, edited by H. SCHENK, R. OLTJOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.
- HUANG, R. H., FABER, M. K., MOEGGENBORG, K. J., WARD, D. L. & DYE, J. L. (1988). *Nature (London)*, **331**, 599–601.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- MORAS, P. D., METZ, B. & WEISS, R. (1973). *Acta Cryst.* **B29**, 383–403.

*Acta Cryst.* (1988). **C44**, 1376–1378

## Tétrachloroplatinate(II) de *trans*-Dichlorobis(propanediamine-1,3)platine(IV)

PAR J.-M. DELAFONTAINE, P. TOFFOLI, P. KHODADAD ET N. RODIER

Laboratoire de Chimie Minérale, Faculté des Sciences Pharmaceutiques et Biologiques, 5 rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

(Reçu le 15 février 1988, accepté le 7 avril 1988)

**Abstract.**  $[PtCl_2(C_3H_{10}N_2)_2][PtCl_4]$ ,  $M_r = 751.15$ , monoclinic,  $P2_1/c$ ,  $a = 7.761(2)$ ,  $b = 7.846(1)$ ,  $c = 14.892(3)$  Å,  $\beta = 107.73(2)^\circ$ ,  $V = 863.8(6)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.888$  Mg m<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.7107$  Å,  $\mu = 17.3$  mm<sup>-1</sup>,  $F(000) = 684$ ,  $T = 294(1)$  K,  $R =$

0.015 for 895 independent reflections [ $I > 2\sigma(I)$ ]. In each ion, the Pt atom is at the centre of symmetry. It displays an almost square-planar coordination in the  $[PtCl_4]^{2-}$  anion and a slightly distorted octahedral coordination in the  $[PtCl_2(C_3H_{10}N_2)_2]^{2+}$  cation which has

0108-2701/88/081376-03\$03.00

© 1988 International Union of Crystallography