GF thanks NSERC Canada for Grants in Aid of Research. Part of this work was supported by a grant from the Department of Education, Ireland (F), and the loan of Rh chemical from Johnson Matthey plc, England, is gratefully acknowledged (TRS).

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A *cyclo*-Boronated Rhodatelluraborane Complex: $[2\text{-}(PPh_3)\text{-}2\text{-}(H)\text{-}2\text{-}(Ph_2PC_6H_4)\text{-}1,2\text{-}TeRhB_{10}H_9]$

By George Ferguson

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

and Faridoon and Trevor R. Spalding

Chemistry Department, University College, Cork, Ireland

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Abstract. 2,7-μ-(Triphenylphosphinato-*P*,*C*)-2-(triphenylphosphine-*P*)-1-tellura-2-rhoda-*closo*-dodecaborane(12), $C_{36}H_{39}B_{10}P_2RhTe$, $M_r = 872{\cdot}3$, monoclinic, $P2_1/a$, $a = 21{\cdot}958\,(3)$, $b = 14{\cdot}738\,(3)$, $c = 11{\cdot}599\,(2)$ Å, $\beta = 92{\cdot}80\,(2)°$, $V = 3749$ Å^3, $Z = 4$, $D_x = 1{\cdot}55$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 13{\cdot}2$ cm^{-1}, $F(000) = 1728$, $T = 298$ K, $R = 0{\cdot}030$ for 5212 observed reflections. The Rh atom is bonded to two phosphine ligands {PPh_3 [2·364 (1) Å] and $Ph_2PC_6H_4$— [2·311 (1) Å]}, an H atom (Rh—H 1·33 Å) and a TeB_4 face of the TeB_{10} cage [Rh—Te 2·5656 (4) Å]. The cage structure of the complex can be described as a derivative of *closo*-$[B_{12}H_{12}]^{2-}$. The formation of a $Ph_2PC_6H_4$ to B bond (*cyclo*-boronation) produces an Rh—P—C—C—B ring in which the Rh atom is 0·25 Å from the P—C—C—B plane. The two Rh—B distances to the B atoms which are also attached to the Te atom are distinctly longer [2·329 (4), 2·331 (4) Å] than the Rh—B distances to the two B atoms not attached to the Te atom [2·263 (5), 2·302 (5) Å]. Similarly, the two Te—B distances to B atoms also attached to the Rh atom are longer [2·379 (4), 2·439 (5) Å] than the Te—B distances to non-Rh-bonded B atoms [2·294 (5), 2·301 (5) Å].

Introduction. It is noticeable that in the chemistry of metallaboranes containing the PPh_3 ligand bonded to a Co, Rh or Ir atom, *cyclo*-boronated compounds have only been reported for Ir (Kennedy, 1986). Examples of such compounds which have been structurally characterized include $[(PPh_3)(Ph_2PC_6H_4)(H)IrB_9H_8]$ (Bould, Greenwood, Kennedy & McDonald, 1982), $[(PPh_3)(Ph_2PC_6H_4)IrB_9H_{7\,\text{or}\,9}(PPh_3)]$ (Kennedy, 1986), $[(PPh_3)(Ph_2PC_6H_4)(H)IrB_9H_{12}]$ (Bould, Crook, Greenwood & Kennedy, 1983) and $[(PPh_3)(Ph_2PC_6H_4)(H)IrB_9H_{10}(PPh_3)]$ (Kennedy, 1986). No rhodium counterparts have been reported. In metallacarbaborane chemistry fewer *cyclo*-boronated compounds are known but $[(PPh_3)(Ph_2PC_6H_4)IrB_8H_6(OMe)C(OH)]$ (Crook, Greenwood, Kennedy & McDonald, 1981) has been reported. The Rh complexes $[1,3\text{-}(\eta^2\text{-}3\text{-}CH_2{=}CHCH_2CH_2)\text{-}3\text{-}(H)\text{-}3\text{-}(PPh_3)\text{-}3,1,2\text{-}RhC_2B_9H_{10}]$ (Delaney, Knobler & Hawthorne, 1981) and $[1,3\text{-}\mu\text{-}2,3\text{-}\mu\text{-}\{1,2\text{-}\mu\text{-}(\eta^2\text{-}3,4\text{-}CH_2CH_2C(Me){=}CHCH_2CH_2CH_2)\}\text{-}3\text{-}(H)\text{-}3\text{-}(PPh_3)\text{-}3,1,2\text{-}RhC_2B_9H_9]$ (Delaney, Teller & Hawthorne, 1981) which contain carbaborane C atoms bonded to organic side chains have also been structurally characterized. The title compound was isolated in low yield from the reaction of $[2,2\text{-}(PPh_3)\text{-}2\text{-}(H)\text{-}1,2\text{-}TeRhB_{10}H_{10}]$ with diphenylacetylene in benzene at 323 K (Faridoon, Ferguson, Fontaine, Kennedy & Spalding, 1988).

Experimental. Straw-coloured small plate crystals were grown from dichloromethane. Accurate cell dimensions and crystal-orientation matrix determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $4 < \theta < 13°$. Crystal dimensions $0{\cdot}08 \times 0{\cdot}26 \times 0{\cdot}55$ mm; intensities of reflections with indices h 0 to 26, k 0 to 18, l −14 to 14, with $2 < 2\theta < 54°$ measured; ω–2θ scans; ω-scan width $(0{\cdot}60 + 0{\cdot}35\tan\theta)°$; graphite-monochromated Mo $K\alpha$ radiation; intensities of three reflections measured every 2 h showed no evidence of crystal decay. 8229 reflections measured, 7328 unique, 5212 with $I > 3\sigma(I)$ labelled observed and used in structure solution and refinement, $R_{\rm int} = 0{\cdot}016$. Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors 0·910, 0·742), Gaussian integration. Space group $P2_1/a$ uniquely from systematic absences $h0l$, $h = 2n + 1$, $0k0$, $k = 2n + 1$. The coordinates of the Rh and Te atoms were determined from analysis of the three-dimensional Patterson function and those of the remaining non-H atoms were found *via* the heavy-atom method. Refinement was by full-matrix least-squares calculations on F, initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the refinement, difference maps showed maxima in positions consistent with the expected locations of the H atoms; in the final rounds of calculations the H atoms were positioned on geometrical grounds (C—H 0·95, B—H 1·08 Å) and included (as riding atoms) in the structure-factor calculations. The position of the H atom bonded to the Rh atom was determined from a difference map and included at that position (Rh—H 1·33 Å). The final cycle of refinement included 451 variable parameters, $R = 0{\cdot}030$, $wR = 0{\cdot}042$, goodness-of-fit 1·08, $w = 1/[\sigma^2(F_o) + 0{\cdot}060(F_o)^2]$. Max. shift/e.s.d. was less than 0·01; density in final difference map $\pm 0{\cdot}60$ e Å^{-3} adjacent to the Rh and Te atoms, no chemically significant features remaining. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983). Atomic coordinates and details of molecular geometry are given in Tables 1 and 2.* Figs. 1 and 2 are views of the molecule prepared using *ORTEP*II (Johnson, 1976).

Table 1. *Positional and thermal parameters and e.s.d.'s*

	x	y	z	B^*(Å^2)
Te	0·13730 (1)	0·12008 (2)	−0·01756 (2)	3·311 (5)
Rh	0·14595 (1)	0·26179 (2)	0·11065 (2)	2·351 (5)
P1	0·18382 (5)	0·39785 (6)	0·18266 (8)	2·67 (2)
P2	0·14688 (4)	0·17882 (7)	0·28517 (8)	2·47 (2)
C11	0·1706 (2)	0·4842 (3)	0·0747 (3)	3·1 (1)
C12	0·1350 (2)	0·4577 (3)	−0·0210 (3)	3·2 (1)
C13	0·1205 (2)	0·5249 (3)	−0·1053 (4)	4·5 (1)
C14	0·1418 (2)	0·6124 (3)	−0·0912 (4)	4·8 (1)
C15	0·1783 (2)	0·6365 (3)	0·0041 (4)	4·6 (1)
C16	0·1931 (2)	0·5729 (3)	0·0883 (4)	3·8 (1)
C21	0·1475 (2)	0·4435 (3)	0·3087 (3)	3·4 (1)
C22	0·0973 (2)	0·5006 (3)	0·2921 (4)	4·4 (1)
C23	0·0676 (2)	0·5314 (4)	0·3863 (5)	6·0 (1)
C24	0·0867 (3)	0·5051 (4)	0·4962 (4)	6·6 (1)
C25	0·1349 (3)	0·4492 (4)	0·5136 (4)	5·9 (1)
C26	0·1660 (2)	0·4172 (3)	0·4196 (4)	4·4 (1)
C31	0·2665 (2)	0·4075 (3)	0·2124 (3)	3·1 (1)
C32	0·3047 (2)	0·3709 (3)	0·1334 (4)	3·9 (1)
C33	0·3667 (2)	0·3763 (3)	0·1466 (5)	4·9 (1)
C34	0·3933 (2)	0·4208 (4)	0·2390 (5)	5·6 (1)
C35	0·3567 (3)	0·4598 (4)	0·3163 (5)	6·3 (1)
C36	0·2934 (2)	0·4553 (4)	0·3052 (4)	5·0 (1)
C41	0·0785 (2)	0·1838 (3)	0·3691 (3)	2·9 (1)
C42	0·0639 (2)	0·1167 (3)	0·4459 (4)	3·8 (1)
C43	0·0137 (2)	0·1252 (4)	0·5130 (4)	4·3 (1)
C44	−0·0217 (2)	0·1991 (4)	0·5022 (4)	4·6 (1)
C45	−0·0086 (2)	0·2663 (3)	0·4264 (4)	4·6 (1)
C46	0·0414 (2)	0·2598 (3)	0·3579 (4)	3·6 (1)
C51	0·2093 (2)	0·1991 (3)	0·3935 (3)	2·9 (1)
C52	0·2677 (2)	0·2138 (3)	0·3587 (4)	3·7 (1)
C53	0·3156 (2)	0·2243 (4)	0·4377 (5)	5·1 (1)
C54	0·3061 (3)	0·2215 (5)	0·5530 (5)	6·5 (1)
C55	0·2478 (3)	0·2097 (4)	0·5920 (4)	5·3 (1)
C56	0·1997 (2)	0·1977 (3)	0·5121 (4)	4·0 (1)
C61	0·1574 (2)	0·0574 (3)	0·2561 (3)	2·9 (1)
C62	0·2154 (2)	0·0223 (3)	0·2444 (4)	4·4 (1)
C63	0·2243 (2)	−0·0664 (3)	0·2129 (5)	5·1 (1)
C64	0·1751 (3)	−0·1236 (3)	0·1943 (5)	4·9 (1)
C65	0·1174 (2)	−0·0919 (3)	0·2080 (4)	4·6 (1)
C66	0·1082 (2)	−0·0010 (3)	0·2368 (4)	3·8 (1)
B1	0·1581 (2)	0·2680 (3)	−0·0877 (4)	3·3 (1)
B2	0·1109 (2)	0·3567 (3)	−0·0309 (4)	3·2 (1)
B3	0·0499 (2)	0·3142 (3)	0·0572 (4)	3·2 (1)
B4	0·0515 (2)	0·1945 (3)	0·0689 (4)	3·0 (1)
B5	0·1022 (3)	0·1942 (4)	−0·1815 (4)	4·1 (1)
B6	0·0977 (3)	0·3109 (4)	−0·1746 (4)	3·9 (1)

* Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $\frac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23}]$.

Discussion. The Rh atom is bonded to PPh_3, H and $Ph_2PC_6H_4$ ligands as well as a TeB_4 face of the $TeB_{10}H_9$ cage, Fig. 1. The *closo* twelve-vertex distorted dodecahedral cage can be described in terms of Wade's rules as a variation of *closo*-$[B_{12}H_{12}]^{2-}$ with the Rh(P) (P′)H unit equivalent to BH and Te as $[BH]^{2-}$ (Wade, 1976). Cyclization to form the five-membered $\overline{\text{Rh—P1—C11—C12—B2}}$ ring has occurred at a boron position which is not adjacent to the Te atom, Fig. 2. The (P)(P′)(H)-ligand conformation about the Rh atom is different from that in the reactant complex $[2,2\text{-}(PPh_3)_2\text{-}2\text{-}(H)\text{-}1,2\text{-}TeRhB_{10}H_{10}]$ (Faridoon, Ferguson, Fontaine, Kennedy & Spalding, 1988) where the Rh—H vector is *trans* to the Te atom and lies above the mid-point of the B2—B3 bond and the Rh—P1 and Rh—P2 bonds are located above the Te—B1 and Te—B4 bonds. In the cyclized product the position of the Rh—H bond corresponds to a 46° rotation of the Rh—H moiety away from an eclipsed orientation with Rh—Te. The cyclic $\overline{\text{Rh—P—C—C—B}}$ system is not quite planar; the Rh atom is 0·25 Å from the P—C—C—B

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* Lists of structure factors, all bond angles, thermal parameters, calculated H-atom coordinates, and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44902 (64 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester, CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and selected bond angles* (°)

Te	Rh	2·5656 (4)
Te	B1	2·379 (4)
Te	B4	2·439 (5)
Te	B5	2·294 (5)
Te	B9	2·301 (5)
Rh	P1	2·311 (1)
Rh	P2	2·364 (1)
Rh	B1	2·331 (4)
Rh	B2	2·263 (5)
Rh	B3	2·302 (5)
Rh	B4	2·329 (4)
P1	C11	1·799 (4)
P1	C21	1·827 (4)
P1	C31	1·837 (4)
P2	C41	1·830 (4)
P2	C51	1·838 (4)
P2	C61	1·838 (4)
C11	C12	1·383 (5)
C11	C16	1·404 (6)
C12	C13	1·417 (6)
C12	B2	1·582 (6)
C13	C14	1·378 (7)
C14	C15	1·380 (7)
C15	C16	1·380 (6)
C21	C22	1·394 (6)
C21	C26	1·386 (6)
C22	C23	1·375 (6)
C23	C24	1·378 (9)
C24	C25	1·348 (8)
C25	C26	1·397 (6)
C31	C32	1·380 (6)
C31	C36	1·393 (6)
C32	C33	1·366 (6)
C33	C34	1·363 (8)
C34	C35	1·359 (8)
C35	C36	1·392 (7)
C41	C42	1·379 (5)
C41	C46	1·387 (6)
C42	C43	1·385 (6)
C43	C44	1·341 (7)
C44	C45	1·365 (7)
C45	C46	1·390 (6)
C51	C52	1·379 (6)
C51	C56	1·402 (5)
C52	C53	1·369 (6)
C53	C54	1·364 (7)
C54	C55	1·390 (8)
C55	C56	1·382 (6)
C61	C62	1·385 (6)
C61	C66	1·392 (5)
C62	C63	1·375 (6)
C63	C64	1·379 (7)
C64	C65	1·366 (7)
C65	C66	1·398 (6)
B1	B2	1·813 (7)
B1	B5	1·935 (7)
B1	B6	1·745 (7)
B2	B3	1·833 (7)
B2	B6	1·808 (6)
B2	B7	1·786 (7)
B3	B4	1·770 (7)
B3	B7	1·784 (6)
B3	B8	1·809 (7)
B4	B8	1·750 (6)
B4	B9	1·970 (6)
B5	B6	1·725 (8)
B5	B9	1·900 (7)
B5	B10	1·728 (9)
B6	B7	1·793 (8)
B6	B10	1·755 (8)
B7	B8	1·784 (7)
B7	B10	1·777 (8)
B8	B9	1·763 (8)
B8	B10	1·802 (8)
B9	B10	1·750 (8)
Rh	H(Rh)	1·33

Rh	Te	B1	56·1 (1)
Rh	Te	B4	55·4 (1)
Rh	Te	B5	96·0 (1)
Rh	Te	B9	96·0 (1)
B1	Te	B4	83·9 (2)
B1	Te	B5	48·9 (2)
B1	Te	B9	84·3 (2)
B4	Te	B5	83·8 (2)
B4	Te	B9	49·0 (2)
B5	Te	B9	48·8 (2)
Te	Rh	P1	158·57 (3)
Te	Rh	P2	94·16 (3)
Te	Rh	B1	57·9 (1)
Te	Rh	B2	94·0 (1)
Te	Rh	B3	94·4 (1)
Te	Rh	B4	59·5 (1)
P1	Rh	P2	98·71 (4)
P1	Rh	B1	105·4 (1)
P1	Rh	B2	80·2 (1)
P1	Rh	B3	96·6 (1)
P1	Rh	B4	138·2 (1)
P2	Rh	B1	150·3 (1)
P2	Rh	B2	158·3 (1)
P2	Rh	B3	111·9 (1)
P2	Rh	B4	85·9 (1)
B1	Rh	B2	46·5 (2)
B1	Rh	B3	82·4 (2)
B1	Rh	B4	87·5 (2)
B2	Rh	B3	47·3 (2)
B2	Rh	B4	81·1 (2)
B3	Rh	B4	44·9 (2)
Rh	P1	C11	108·6 (1)
Rh	P1	C21	116·4 (1)
Rh	P1	C31	118·0 (1)
C11	P1	C21	103·6 (2)
C11	P1	C31	101·6 (2)
C21	P1	C31	106·7 (2)
Rh	P2	C41	117·5 (1)
Rh	P2	C51	118·4 (1)
Rh	P2	C61	110·0 (1)
C41	P2	C51	103·5 (2)
C41	P2	C61	104·5 (2)
C51	P2	C61	100·8 (2)
P1	C11	C12	115·1 (3)
C11	C12	B2	119·9 (3)
Rh	B2	C12	115·4 (3)

plane. In the Ir–borane complexes referred to above the equivalent atoms are usually more planar (Kennedy, 1986).

The Rh—Te bond length, 2·5656 (4) Å, is shorter than the value of 2·6172 (4) Å in $[2,2\text{-}(\mathrm{PPh_3})_2\text{-}2\text{-}(\mathrm{H})\text{-}1,2\text{-}\mathrm{TeRhB_{10}H_{10}}].0{\cdot}5\mathrm{C_6H_{12}}$ (Faridoon, Ferguson, Fontaine, Kennedy, Ni Dhubhghaill, Reed & Spalding, 1988); no other Rh—Te compounds have been reported. The Rh—P bond lengths fall within the typical range but are slightly different; Rh—$\mathrm{PPh_3}$ 2·364 (1) and Rh—$\mathrm{PPh_2C_6H_4}$ 2·311 (1) Å. The bond lengths in the $\overline{\mathrm{Rh—P—C—C—B}}$ ring are not unusual [for example, C—B 1·582 (6) and Rh—B 2·263 (5) Å] when compared to other rhodaboranes or *cyclo*-boronated Ir complexes (Kennedy, 1986). However, the two Rh—B bonds to B atoms adjacent to the Te atom are longer [mean 2·330 (4) Å] than the two Rh—B bonds to B2 and B3 atoms [mean 2·283 (20) Å]. The lengths of the Te—B bonds vary between 2·294 (5) and 2·439 (5) Å with the two in the $\mathrm{TeB_4}$ ring bonded to the Rh atom, *i.e.* TeB1 and TeB4, much longer than TeB5 and TeB9. Intercage B—B bond lengths are normal.

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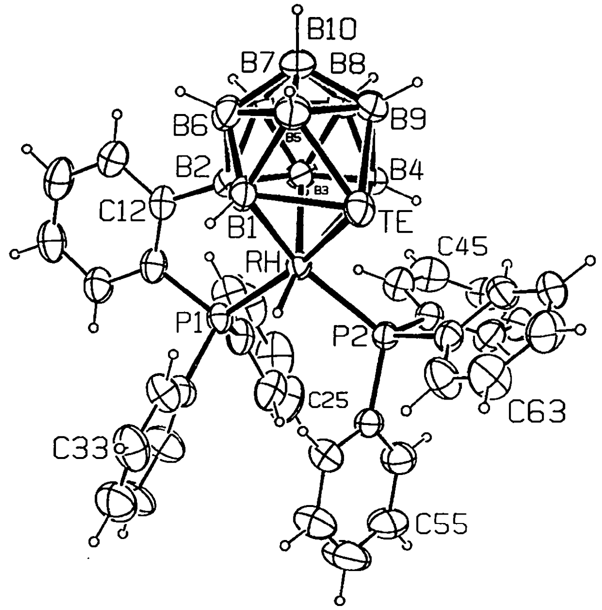

Fig. 1. A view of the complex showing the numbering scheme. Phenyl rings are numbered C*i*1–C*i*6, *i* = 1–6.

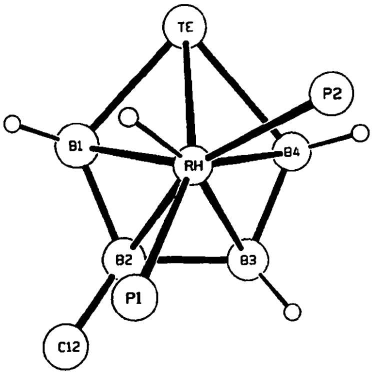

Fig. 2. A view normal to the $\mathrm{TeB_4}$ plane showing the orientation of the H atom and phosphine groups bonded to the Rh atom.

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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*

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Abstract. $K^+.C_{18}H_{36}N_2O_6.e^-$, $M_r = 415{\cdot}60$, monoclinic, $C2/c$, $a = 12{\cdot}129\ (8)$, $b = 20{\cdot}692\ (13)$, $c = 21{\cdot}519\ (16)$ Å, $\beta = 95{\cdot}23\ (6)°$, $V = 5378\ (6)$ Å^3, $Z = 8$, $D_x = 1{\cdot}026$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 2{\cdot}2$ cm^{-1}, $F(000) = 1800$, $T = 202\ (2)$ K; final $R = 0{\cdot}041$ 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 $K^+—O$ 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, $Cs^+(18\text{-crown-}6)_2.e^-$, was determined in 1985 (Dawes, Ward, Huang & Dye, 1986). $Cs^+(18\text{-crown-}6)_2.e^-$ 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^+(cryptand[2.2.2])$.e^-$, 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 dependence of the magnetic susceptibility indicates a spin-pairing energy of ~0·05 eV (Huang, Faber, Moeggenborg, 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 $7{\cdot}5 < \theta < 10°$. Intensity data were collected using θ–2θ scans at 4° min^{-1} (in 2θ) to $(\sin\theta)/\lambda = 0{\cdot}5385$ Å^{-1}; reflection indices: $0 \le h \le 15$, $0 \le k \le 26$, and $-28 \le l \le 27$; 6692 total data. A linear decay correction was based on the intensities of three monitor reflections (5·7%

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

† To whom correspondence should be addressed.

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