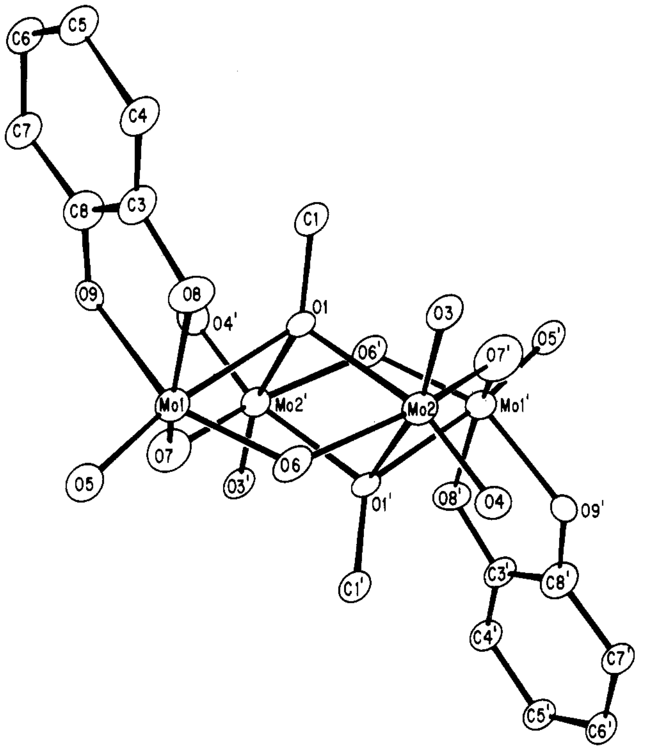

Figure 4. ORTEP view of the structure of $[Mo_4O_{10}(OCH_3)_2(OC_6H_4O)_2]^{2-}$ (**2**), showing the atom-labeling scheme. Selected bond lengths (Å): Mo1–O1, 2.416 (7); Mo1–O5, 1.699 (8); Mo1–O6, 1.817 (7); Mo1–O7, 1.824 (9); Mo1–O8, 1.981 (9); Mo1–O9, 1.982 (7); Mo2–O1, 2.270 (7); Mo2–O3, 1.682 (11); Mo2–O4, 1.677 (8); Mo2–O6, 1.983 (7); Mo2–O1′, 2.248 (8); Mo2–O7′, 1.990 (8).

chloride yields the structurally analogous **1a**.[23] Reaction of the $(Bu_4N)^+$ salt of **1** with 2 equiv of catechol in methanol[24] yields, when allowed to stand for several days, bright green crystals of $(n\text{-}Bu_4N)_2[Mo_4O_{10}(OMe)_2(OC_6H_4O)_2]$ (**2**).[25]

The key feature of structure **2** is the retention of the tetranuclear core with pseudooctahedral $[MoO_6]$ coordination geometries, as shown in Figure 4. Although a number of Mo–catecholato complexes have been previously structurally characterized,[26-30] none have displayed the tetranuclear core, which persists in reactions carried out in methanol and which appears to be a characteristic structural type under these reaction conditions.

Complexes **1** and **2** display contrasting behavior upon thermal decomposition. While thermal decomposition of **1** at 150 °C yields dimethyl ether, formaldehyde, methanol, and water as gaseous products, the decomposition of **2** produces only water and methanol as volatile products. This result is consistent with the presence of significant intramolecular C–H···O contacts in **1**, providing a mechanism for C–H bond activation,[7] and with absence of close C–H···O contacts in **2**.[31] These observations lend further support to the model proposed by McCarron et al. for C–H bond activation in the oxidation of methanol to formaldehyde.[5-7]

Complex **2** and analogous species of the general type $[Mo_4O_{10}(OCH_3)_2(XC_6H_4Y)_2]^{2-}$ react readily with organohydrazine ligands.[19] Thus, in the reactions with phenylhydrazine, complex **2a**, $[Mo_4O_{10}(OCH_3)_2(HNC_6H_4O)_2]^{2-}$, yields $[Mo_4O_6(OCH_3)_2(NNPh)_4(HNC_6H_4O)_2]^{2-}$ (**3**) or $[Mo_2(OCH_3)_2(HNC_6H_4O)_2(NNPh)_4]$ (**5**), depending upon reaction conditions. As illustrated by Figure 3, structurally analogous tetranuclear cores are observed for a range of chemical transformations.

Acknowledgment. This work was supported by a grant from the National Science Foundation to J.Z. (CHE8514634).

Supplementary Material Available: Tables of atom coordinates, bond lengths, bond angles, and thermal parameters for **1** and **2** (21 pages); tables of observed and calculated structure factors for both compounds (78 pages). Ordering information is given on any current masthead page.

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[23] (a) Anal. Calcd for $C_{36}H_{84}N_2Mo_4Cl_2O_{14}$: C, 35.3; H, 6.93; N, 2.29. Found: C, 35.4; H, 7.19; N, 2.26. (b) Crystal data: space group $P\bar{1}$, $a = 10.186$ (2) Å, $b = 11.722$ (2) Å, $c = 13.173$ (2) Å, $\alpha = 109.35$ (1)°, $\beta = 107.43$ (1)°, $\gamma = 96.99$ (1)°, $Z = 1$, $D_{calcd} = 1.45$ g cm^{-3}; 3020 reflections with $F_o \geq 6\sigma(F_o)$; $R = 0.0407$.

[24] Similar reactions have been carried out by using 2-aminophenol and 2-aminothiophenol.

[25] (a) Anal. Calcd for $C_{46}H_{86}N_2Mo_4O_{16}$: C, 42.3; H, 6.58. Found: C, 42.7; H, 6.47. The IR spectrum of **2** in KBr shows a complex pattern of absorbances in the 830–920-cm^{-1} Mo=O region and in the 2950-cm^{-1} methoxy C—H stretching region. (b) X-ray diffraction studies were carried out at ca. 20 °C with monochromatized Mo Kα radiation. Crystal data: space group $P2_1/a$, $a = 18.474$ (3) Å, $b = 16.192$ (3) Å, $c = 20.411$ (4) Å, $\beta = 107.51$ (2)°, $V = 5822.6$ (12) Å^3, $Z = 4$; crystal dimensions, 0.22 × 0.18 × 0.26 mm; the conditions of data collection and reduction were identical with those for **1**. Of 5645 reflections collected, 3832 were used in the structure refinement ($F_o \geq 6\sigma(F_o)$; Mo Kα, $\lambda = 0.71073$ Å; $R = 0.0539$). The $n\text{-}Bu_4N^+$ cations were somewhat disordered as indicated by C–C bond distances in the range 1.32 (2)–1.59 (4) Å and thermal parameters in the range 0.070–0.603 Å^2. Since only the anion was of interest and it behaved quite well, the disorder of the cations was modeled by placing the carbon atoms at the peak of highest density consistent with an atom position.

[26] Cass, M. E.; Pierpont, C. G. *Inorg. Chem.* **1986**, *25*, 123.

[27] Buchanan, R. M.; Pierpont, C. G. *Inorg. Chem.* **1979**, *18*, 1616.

[28] Pierpont, C. G.; Buchanan, R. M. *Inorg. Chem.* **1982**, *21*, 652.

[29] Pierpont, C. G.; Buchanan, R. M. *J. Am. Chem. Soc.* **1975**, *97*, 6450.

[30] Pierpont, C. G.; Downs, H. H. *J. Am. Chem. Soc.* **1975**, *97*, 2123.

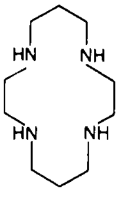
[31] Hydrogen atom positions were located and refined for **1**. Although H atoms were not located for **2**, C···O contact distances precluded significant C–H···O interactions. C–H···O contacts for **1**: (C3)H3c···O6, 2.63 Å; (C6)H6a···O4, 2.64 Å; (C3)H3c···O2, 2.75 Å; (C3)H3b···O5, 2.75 Å; (C7)H7c···O3, 2.74 Å.

Unusually Rapid Homolysis of the Cobalt-Carbon Bond in a Macrocyclic Benzylcobalt Complex

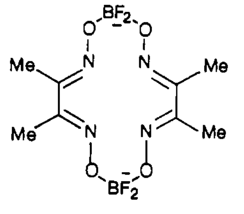
Sir:

The highest rate constant reported for homolysis of a cobalt–carbon bond in a series of macrocyclic benzylcobalt complexes is that for benzylcobalamin, $k = 2.7 \times 10^{-3}$ s^{-1} at 25.5 °C.[1] The ready homolysis of this complex as compared to other $C_6H_5CH_2Co(N_4\text{-macrocycle})$ complexes has been attributed in part to configurational motions of the corrin ligand,[1a] and to steric interactions that cause the angular distortion of the Co–C bond.[1c] We now report that a newly prepared benzylcobalt complex of a synthetic macrocycle, $C_6H_5CH_2Co([14]aneN_4)(H_2O)^{2+}$ ($[14]aneN_4$ = 1,4,8,11-tetraazacyclotetradecane), homolyzes in aqueous solutions even more rapidly than benzylcobalamin does.

The structures of the ligands $[14]aneN_4$ and $(dmgBF_2)_2$ (see later) are



[14]aneN4



$(dmgBF_2)_2$

The organocobalt complex $C_6H_5CH_2Co([14]aneN_4)(H_2O)^{2+}$ was prepared by photolysis of $C_6H_5CH_2Co(dmgH)_2H_2O$ in the presence of $Co([14]aneN_4)(H_2O)_n^{2+}$ (n = 1 or 2),[2] followed by

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[1] (a) Schrauzer, G. N.; Grate, J. H. *J. Am. Chem. Soc.* **1981**, *103*, 541. (b) Blau, R. J.; Espenson, J. H. *J. Am. Chem. Soc.* **1985**, *107*, 3530. (c) Christianson, D. W.; Lipscomb, W. N. *J. Am. Chem. Soc.* **1985**, *107*, 2682.

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Table I. Kinetic Data and Activation Parameters[a] for Unimolecular and Bimolecular Homolyses of Benzylcobalt Complexes, $C_6H_5CH_2Co(L)^{n+}$

Co(L)	k_h/s^{-1} [b–d]	$k_{Cr}/M^{-1}\ s^{-1}$ [c,d]
$Co(14\text{-ane})(H_2O)^{2+}$ [e]	0.00211 (1.5)	0.114 (1.5)
	0.00960 (9.5)	0.200 (9.5)
	0.0300 (16.4)	
	0.0266 (17.2)	0.36 (17.2)
	0.0404 (19.5)	
	0.0941 (25.0)	~0.37 (25.0)
	$\Delta H^* = 25.4 \pm 1.0$ $\Delta S^* = 22.0 \pm 3.3$	$\Delta H^* = 8 \pm 2$ $\Delta S^* = -33 \pm 6$
cobalamin[f]	$\Delta H^* = 24.6 \pm 0.6$ $\Delta S^* = 12.3 \pm 2.0$	$k_h(25\ °C) = 2.7 \times 10^{-3}\ s^{-1}$
cobinamide[f]	$\Delta H^* = 26.9 \pm 0.5$ $\Delta S^* = 9.2 \pm 1.5$	$k_h(25\ °C) = 1.2 \times 10^{-5}\ s^{-1}$
$Co(dmgBF_2)_2(H_2O)$ [g]	$\Delta H^* = 28.6 \pm 0.8$ $\Delta S^* = 14.0 \pm 2.6$	$k_h(25\ °C) = 7.6 \times 10^{-6}\ s^{-1}$
Co(saloph)py[h]	$\Delta H^* = 23.6 \pm 1.0$ $\Delta S^* = 1.3 \pm 3$	$k_h(25\ °C) = 6.0 \times 10^{-5}\ s^{-1}$
$Co[C_2(DO)(DOH)_{pn}]$ [i]	$\Delta H^* = 27.9 \pm 0.8$ $\Delta S^* = 8 \pm 2$	$k_h(25\ °C) = 1.2 \times 10^{-6}\ s^{-1}$

[a] ΔH^* in kcal/mol, ΔS^* in cal/(mol K). The solvent is water unless otherwise noted. [b] The rate constants were measured in the presence of 1.2–3.6 mM Fe^{3+} and 0.25–1.0 mM O_2. The intercepts of the plots of k_{obsd} vs $[Cr^{2+}]$ ($[Cr^{2+}]$ = 0.017–0.067 M) gave identical values. [c] The temperature (°C) is given in parentheses. [d] μ = 0.20 M ($HClO_4$ + $LiClO_4$). [e] This work. [f] Reference 1a. [g] Reference 6d. [h] In pyridine (Tsou, T.-T.; Loots, M.; Halpern, J. *J. Am. Chem. Soc.* **1982**, *104*, 623), saloph = *N,N'*-(bis(salicylidene)-*o*-phenylene)diamino. [i] In *o*-dichlorobenzene (Finke, R. G.; Smith, B. L.; Mayer, B. J.; Molinero, A. A. *Inorg. Chem.* **1983**, *25*, 3677), $C_2(DO)(DOH)_{pn}$ = 11-hydroxy-2,10-diethyl-3,9-dimethyl-1,4,8,11-tetraazaundeca-1,3,8,10-tetraen-1-olato.

ion exchange.[3] Dilute aqueous solutions of $C_6H_5CH_2Co([14]aneN_4)(H_2O)^{2+}$ decompose at room temperature to yield bibenzyl and $Co([14]aneN_4)(H_2O)_n{}^{2+}$. The rate constant for decomposition decreases with time owing to the accumulation of $Co([14]ane)(H_2O)_n{}^{2+}$ in solution.

The loss of the organocobalt is accelerated by addition of Cr^{2+}, O_2, and Fe^{3+}, i.e. reagents that react rapidly with $Co([14]aneN_4)(H_2O)_n{}^{2+}$ and/or benzyl radicals. The reaction with Cr^{2+} yields the known complex $C_6H_5CH_2Cr(H_2O)_5{}^{2+}$ under all conditions; 1 mM oxygen produces benzaldehyde and benzyl alcohol in a 6:1 ratio, and the μ-peroxo complex $Co([14]ane)O_2Co([14]ane)^{4+}$.[4] The reaction with 0.05 M Fe^{3+} yields benzyl alcohol (82%), bibenzyl (18%), and the oxidized cobalt complex.

In the presence of any of these scavengers the reaction obeys a first-order rate law, $-d\ln[C_6H_5CH_2Co([14]ane)H_2O^{2+}]/dt = k_h$. The rate constant k_h at 25 °C and 0.20 M ionic strength (0.094 s^{-1}) is independent of the nature and concentration of the scavenging reagents, Table I. All the kinetic and product analysis[5] data are consistent with the reaction being unimolecular homolysis:

$$C_6H_5CH_2Co([14]aneN_4)H_2O^{2+} \xrightarrow{k_h} C_6H_5CH_2^{\bullet} + Co([14]aneN_4)(H_2O)_n{}^{2+} \quad (1)$$

At the highest concentration of Cr^{2+} used, 0.067 M, the observed rate constant at 25 °C is some 20% higher than k_h, owing to the contribution from bimolecular homolytic displacement.[6,7] The effect becomes more pronounced at lower temperatures, such that at 1.5 °C k_h contributes only negligibly to the overall rate constant at high $[Cr^{2+}]$. The rate law is given by eq 2. The

$$-d\ln[C_6H_5CH_2Co([14]ane)H_2O^{2+}]/dt = k_h + k_{Cr}[Cr^{2+}] \quad (2)$$

increasing contribution from the S_H2 component at lower temperatures is expected owing to the dramatically different activation parameters for the two processes, Table I.

The values of the activation parameters[8] and rate constants at 25 °C for homolysis of the macrocyclic benzylcobalt complexes in aqueous solutions, along with two examples in nonaqueous solvents, are summarized in Table I. The rate constants span 5 orders of magnitude; the highest one is that for $C_6H_5CH_2Co([14]aneN_4)H_2O^{2+}$. The top four complexes were studied in aqueous solutions in the absence of added bases. The ligand trans to the alkyl group is thus H_2O[9] except for benzylcobalamin, which in the base-on form has a coordinated dimethylbenzimidazole. The difference in the homolysis rate constants between benzylcobalamin and benzylcobinamide largely reflects the effect of the coordinated dimethylbenzimidazole in the former.

The activation enthalpy for unimolecular homolysis of a metal–carbon bond is a reasonable approximation for the bond dissociation energy, since the reverse process has low energy requirements.[6d,10,11] Despite the relatively small differences in ΔH^* between the three benzylaquo complexes[9] (Co(L) = $Co([14]aneN_4)H_2O$, cobinamide, and $Co(dmgBF_2)_2H_2O$), it appears that $C_6H_5CH_2Co([14]aneN_4)H_2O^{2+}$ has the weakest Co–C bond. The destabilizing effect of the saturated equatorial ligands on the Co–C bond is expected[7] on the basis of the effect of the ligands on the energy of the metal d orbitals.[12] The threshold energy for photohomolysis of $CH_3Co([14]aneN_4)H_2O^{2+}$ [7] is lower than those of several methylcobalt complexes with unsaturated macrocyclic ligands of similar steric demands. This is also consistent with a lower energy of the Co–C bonds for complexes containing $[14]aneN_4$. The effect of ligand saturation alone on the Co–C bond energy in $C_6H_5CH_2Co([14]aneN_4)H_2O^{2+}$ is, however, difficult to assess owing to the significant differences in steric and electronic properties between the three benzylaquo[9] complexes in Table I. Even for the two smallest complexes the steric crowding around the Co–C bond might be substantially different owing to the planarity of the $Co(dmgBF_2)_2$ fragment,[13] as opposed to the chair configuration adopted by the $Co([14]aneN_4)^{2+}$.[2,3]

The value of ΔH^* for homolysis of $C_6H_5CH_2Co(saloph)py$, a complex with an unsaturated equatorial ligand (Table I), is comparable (possibly smaller) to those for $C_6H_5CH_2Co([14]aneN_4)H_2O^{2+}$ and benzylcobalamin. The differences in solvent (pyridine vs H_2O), axial ligands (pyridine vs H_2O vs dimethylbenzimidazole) and the nature of the donor atoms of the macrocycle (N_2O_2 vs N_4) make it difficult to identify the factor(s) responsible for the low Co–C bond energy in this complex. A

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(3) Bakac, A.; Espenson, J. H. *Inorg. Chem.*, in press. The stable alkyl complexes were characterized as perchlorate salts by elemental analysis, UV-visible and ^1H NMR spectroscopy, and X-ray crystallography for R = ethyl. The benzyl complex exhibits maxima at λ = 512 nm (ϵ = 1.0 × 10^2 $M^{-1}\ cm^{-1}$) and 310 nm (ϵ ~10^4 $M^{-1}\ cm^{-1}$).

(4) Wong, C.-L.; Switzer, J. A.; Balakrishnan, K. P.; Endicott, J. F. *J. Am. Chem. Soc.* **1980**, *102*, 5511.

(5) The reaction of benzyl radicals with O_2 should yield equimolar amounts of C_6H_5CHO and $C_6H_5CH_2OH$. The high yields of C_6H_5CHO obtained in this work, as well as in the oxygen-induced homolysis of $C_6H_5CH_2Cr(H_2O)_5{}^{2+}$ (Nohr, R. S.; Espenson, J. H. *J. Am. Chem. Soc.* **1975**, *97*, 3392) and $C_6H_5CH_2$–cobalamin at pH 7,[1a] are probably the result of the subsequent oxidation of $C_6H_5CH_2OH$ by the metal peroxo complexes.[1b]

(6) (a) Espenson, J. H.; Shveima, J. S. *J. Am. Chem. Soc.* **1973**, *95*, 4468. (b) Espenson, J. H.; Sellers, T. D., Jr. *J. Am. Chem. Soc.* **1974**, *96*, 94. (c) Johnson, M. D. *Acc. Chem. Res.* **1983**, *16*, 343. (d) Bakac, A.; Espenson, J. H. *J. Am. Chem. Soc.* **1984**, *106*, 5197.

(7) Endicott, J. F.; Balakrishnan, K. P.; Wong, C.-L. *J. Am. Chem. Soc.* **1980**, *102*, 5519.

(8) The activation parameters given[1a] for homolysis of benzylcobalamin are uncorrected for the base-on/base-off equilibrium. Assuming the thermodynamic parameters for the latter equilibrium have values ΔH = 4 kcal mol^{-1} and ΔS = 10 cal $mol^{-1}\ K^{-1}$, similar to other alkylcobalamins, the activation parameters for homolysis would change only slightly to ΔH^* = 25.2 kcal mol^{-1}, ΔS^* = 14.4 cal $mol^{-1}\ K^{-1}$.

(9) There is some controversy about the presence of the coordinated water in alkylcobinamides (Firth, R. A.; Hill, H. A. O.; Mann, B. E.; Pratt, J. M.; Thorp, R. G.; Williams, R. J. P. *J. Chem. Soc. A* **1968**, 2419. Zangrando, E.; Parker, W. O., Jr.; Bresciani-Pahor, N.; Thomas, L. B.; Marzilli, L. G.; Randaccio, L. *Gazz. Chim. Ital.* **1987**, *117*, 307).

(10) Halpern, J. *Acc. Chem. Res.* **1982**, *15*, 238.

(11) Hay, B. P.; Finke, R. G. *J. Am. Chem. Soc.* **1986**, *108*, 4820.

(12) Green, M.; Smith, J.; Tasker, P. A. *Discuss. Faraday Soc.* **1969**, *47*, 172.

(13) Bakac, A.; Brynildson, M. E.; Espenson, J. H. *Inorg. Chem.* **1986**, *25*, 4108.

systematic variation of axial bases and macrocycles would help sort out their individual contributions.[14–16]

A substantial contribution to the ease of the Co–C bond homolysis in $C_6H_5CH_2Co([14]aneN_4)(H_2O)^{2+}$ comes from the large positive value of ΔS^*. This we attribute to the influence of the overall 2+ charge on the change in solvation of the benzyl group between the ground and transition states. The ΔS^* term is much smaller for the five neutral complexes in Table I.

Acknowledgment. This work was supported by the Office of Basic Energy Sciences, Chemical Sciences Division, U.S. Department of Energy, under Contract W-7405-Eng-82.

Supplementary Material Available: A table of the kinetic data and a plot of k_{obsd} against [Cr^{2+}] at different temperatures (2 pages). Ordering information is given on any current masthead page.

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(14) The activation enthalpies for homolysis of a series of benzylcobaloximes and benzylcobalt porphyrin complexes containing bulky phosphines as axial ligands in toluene[15] and ethylene glycol[16] are all higher than the value for $C_6H_5CH_2Co(saloph)py$.

(15) Geno, M. K.; Halpern, J. *J. Am. Chem. Soc.* **1987**, *109*, 1238.

(16) Geno, M. K.; Halpern, J. *J. Chem. Soc., Chem. Commun.* **1987**, 1052.

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Received June 1, 1987

Incipient Pentagonal-Pyramidal Coordination of Tellurium in a Macrocyclic Tetra(secondary amine) Complex

Sir:

Among the seven topologically distinct six-vertex polyhedra,[1,2] the octahedron (Figure 1) is found in the vast majority of six-coordinate complexes.[3] The trigonal prism (Figure 1) is found in some tris(dithiolene)[4] and a few other six-coordinate metal complexes.[5] Among the remaining five six-vertex polyhedra the C_{2v} 6,11,7-polyhedron (Figure 1)[6] and the pentagonal pyramid (Figure 1) may both be derived from AB_6E systems based on an underlying pentagonal bipyramid with a stereochemically active lone pair in an equatorial (6,11,7-polyhedron) and in an axial (pentagonal pyramid) position, respectively. The "distorted octahedron"[8] in the tellurium(IV) anion $[Me_2NCS_2TeBr_4]^-$ may be interpreted as a C_{2v} 6,11,7-polyhedron. This communication reports the first example of a pentagonal-pyramidal six-coordinate complex based on an underlying tellurium(IV) pentagonal bipyramid with a stereochemically active lone pair in an axial position. This complex was obtained serendipitously from an attempted preparation of (cyclen)Te isoelectronic with the known[9] (cyclen)PH.

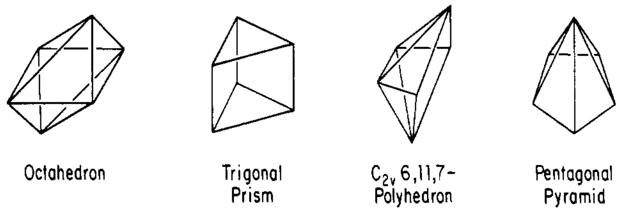

Figure 1. The four six-vertex polyhedra discussed in this paper.

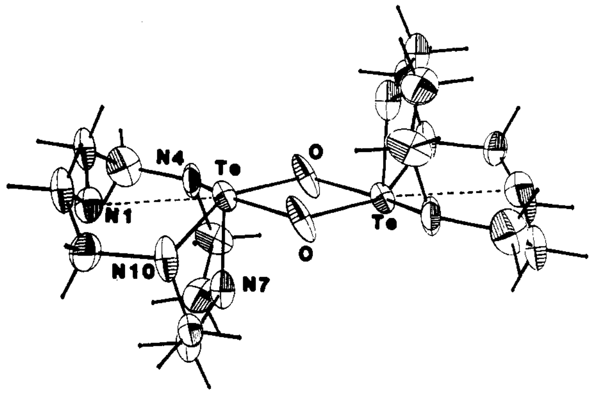

Figure 2. ORTEP diagram of the $[(C_8H_{16}N_4H_3)_2Te_2O_2]^{+2}$ cation in $[(C_8H_{16}N_4H_3)_2Te_2O_2]Cl_2 \cdot 4CHCl_3 \cdot 2CH_3OH$.

Table I. Tellurium Coordination Polyhedron in $[(cyclenH_3)_2Te_2(\mu\text{-}O)_2]Cl_2 \cdot 2MeOH \cdot 4CHCl_3$

Distances (Å)			
Apical			
Te–N7	1.996 (22)		
Basal			
Te–O1	1.998 (13)	Te–N1	2.703 (18)
Te–O1′	1.998 (13)	Te–N10	2.424 (19)
Te–N4	2.472 (18)		
Nonbonding			
Te–Te	3.107 (2)		
Angles (deg)			
Apical–Basal			
N7–Te–O1	92.3 (7)	N7–Te–N1	83.1 (7)
N7–Te–O1′	91.5 (7)	N7–Te–N10	79.0 (7)
N7–Te–N4	77.6 (7)		
Adjacent Basal–Basal			
O1–Te–O1′	77.3 (6)	N1–Te–N10	67.6 (5)
O1′–Te–N4	73.2 (6)	N10–Te–O1	73.8 (6)
N4–Te–N1	67.9 (6)		
Nonadjacent Basal–Basal			
O1–Te–N4	148.4 (6)	N1–Te–O1	141.3 (6)
O1′–Te–N1	141.0 (5)	N10–Te–O1′	149.0 (6)
N4–Te–N10	131.5 (6)		

A solution of $(CF_3CH_2O)_4Te$[10] was obtained by treatment of 2.0 g (7.4 mmol) of $TeCl_4$ in 50 mL of tetrahydrofuran and 20 mL of pentane at −72 °C with a mixture of 3.34 g (33.4 mmol) of CF_3CH_2OH and 3.4 g (33.4 mmol) of triethylamine. After the mixture was warmed slowly to room temperature over a period of 5 h, the precipitate of $[Et_3NH]Cl$ was removed by filtration of 0 °C. The filtrate was cooled to −20 °C and then treated with 1.2 g (7 mmol) of 1,4,7,10-tetraazacyclododecane ($cyclenH_4$). After the mixture was stirred for 12 h, the white precipitate was filtered. Suitable rhombohedral crystals for X-ray diffraction were obtained by crystallization from a mixture of chloroform and methanol. These white crystals, shown by the structure determination to have the stoichiometry $[(C_8H_{16}N_4H_3)_2Te_2O_2]Cl_2 \cdot 4CHCl_3 \cdot 2MeOH$, are efflorescent, losing the coordinated solvent molecules and their crystallinity upon drying. The crystals were therefore kept moist with the solvent mixture during the structure determination. Elemental analysis on the dry noncrystalline material gave values close to those expected for the solvent-free formulation $[(C_8H_{16}N_4H_3)_2Te_2O_2]Cl_2$. Anal. Calcd: C, 27.4;

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