perturbation expressions for the spin Hamiltonian parameters for the unpaired electron in a d_{z^2} orbital given by Maki et al.[1] are

$$g_{xx} = 2.0023 - 6b_1 \qquad g_{yy} = 2.0023 - 6b_2$$

$$g_{zz} = 2.0023 \qquad A_{xx} = P(-6b_1 - \kappa - {}^2\!/_7 - {}^3\!/_7 b_2)$$

$$A_{yy} = P(-6b_2 - \kappa - {}^2\!/_7 - {}^3\!/_7 b_1)$$

$$A_{zz} = P[-\kappa + {}^4\!/_7 + {}^3\!/_7(b_1 + b_2)]$$

The coefficients b_1 and b_2 derived from the experimental g values ($g_{xx} = 2.1123$, $g_{yy} = 2.1157$, and $g_{zz} = 2.0089$) are used in deriving the Fermi contact term κ and the dipolar term P. Assuming A_{xx} and A_{yy} to be of the same sign (negative) and taking A_{zz} to be positive, we get $\kappa = 0.0816$ and $P = 52.7 \times 10^{-4}$ cm^{-1}. Substituting these, we derive $A_{yy} = 13 \times 10^{-4}$ cm^{-1}, which is in very good agreement with the experimental value. The relative signs thus obtained, viz., A_{zz} positive, A_{xx} and A_{yy} negative, are justified by the isotropic spectrum of the ^{61}Ni-enriched complex, which is identical with that reported for the unenriched (natural) Ni(III) complex.[10] The A_{av} obtained, with the assignment of these signs, viz., 0.1×10^{-4} cm^{-1}, is lower than the line width of the isotropic spectrum of the ^{61}Ni-enriched complex. The P value obtained corresponds to 42% (±2)% of the free-ion value[12] of 125.3×10^{-4} cm^{-1} calculated recently by Morton and Preston[12] with use of the Hermann–Skillman wave function for ^{61}Ni. Though this 58 (±2)% delocalization on the ligand is slightly lower than that predicted earlier[10] through spin-density calculations, in which 68 (±2)% of the unpaired electron density was found to be on the ligands, the results are in reasonably good agreement. The deviations may be explained as due to the neglect of other possible excited states contributing to the spin Hamiltonian parameters. It is significant to note that there is a marked deviation of g_{zz} (2.0089) from the free-electron g value predicted by second-order perturbation theory. Unfortunately this cannot be further quantified due to the simple experimental optical spectrum and very complex equations involving quartet and other doublet excited states. The noninclusion of ligand spin–orbit coupling due to the second-row ligands could have also been responsible.

Acknowledgment. The authors wish to thank Dr. John Ammeter, University of Zurich, for giving us 85% isotopically pure ^{61}NiO. E.B. is grateful to UGC, New Delhi, for financial assistance and to the authorities of VOC College, Tuticorin, for permitting him to undertake this work.

Registry No. [Ni(phmp)$_2$Cl$_2$]ClO$_4$, 60536-62-1; [Co(phmp)$_2$Cl$_2$]ClO$_4$, 60489-46-5.

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Contribution from the Department of Chemistry,
University of Virginia, Charlottesville, Virginia 22901

Crystal and Molecular Structure of $(C_2H_5)_3NH^+[Co(1,2\text{-}C_2B_9H_{11})_2]^-$

Lester Borodinsky, Ekk Sinn, and Russell N. Grimes*

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Structural characterizations of a variety of transition-metal dicarbollyl sandwich complexes have been reported.[1] These species have the general formula $(R_2C_2B_9H_9)_2M^n$, where R is H or CH$_3$ and $-2 \le n \le 0$; in addition, several boron-substituted derivatives have also been crystallographically studied. All of these compounds consist of two icosahedral MC$_2$B$_9$ units fused at the metal which is common to both polyhedra, but there are significant variations in cage geometry; different relative orientations of the ligands (rotamers) have been observed, and electron-rich metals such as Ni(II), Ni(III), and Cu(II) cause a type of slip distortion in which the metal is displaced away from the centroid of the five-membered bonding face of the ligand.[1]

Table I. Experimental Parameters and Crystal Data

mol wt	425.93	trans fac (max)	0.93
space group	$Pna2_1$	trans fac (min)	0.87
a, Å	29.048 (9)	2θ range, deg	1.4–50
b, Å	11.457 (4)	reflctn obsd	2076
c, Å	7.100 (2)	reflctn rfnd	1879
V, Å^3	2363	R	0.049
μ, cm^{-1}	7.539	R_w	0.054
D(calcd), g cm^{-3}	1.180	esd unit wt	2.02
A	0.60	Z	4
B	0.35		
cryst dimens (mm from centroid)	100 (0.035)	001 (0.35)	
	$\bar{1}00$ (0.035)	$00\bar{1}$ (0.35)	
	010 (0.09)		
	$0\bar{1}0$ (0.09)		

We wish to report here a structure determination of a bis(dicarbollyl) complex of cobalt(III), $Co(1,2\text{-}C_2B_9H_{11})_2^-$, which was isolated in the course of attempts to prepare mixed-ligand complexes containing $R_2C_2B_4H_4^{2-}$ and $C_2B_9H_{11}^{2-}$ ligands. Salts of $Co(1,2\text{-}C_2B_9H_{11})_2^-$ were prepared long ago by Hawthorne and co-workers,[2] and an X-ray study of the Cs$^+$ salt was reported by Zalkin, Hopkins, and Templeton;[3] however, the carbon and facial boron atoms were disordered in that study, and hence the ligand orientation could not be established. In the present investigation of the triethylammonium salt, no disorder was encountered and the framework carbon atoms were unambiguously located; this in turn allowed comparison with the known structure[4] of the isoelectronic nickel species, $(1,2\text{-}C_2B_9H_{11})_2Ni^{IV}$.

Experimental Section

Crystals of red $(C_2H_5)_3NH^+[Co(1,2\text{-}C_2B_9H_{11})_2]^-$, obtained by addition of triethylammonium chloride to a reaction mixture containing $(1,2\text{-}C_2B_9H_{11})^{2-}$, $(CH_3)_2C_2B_4H_5^-$, and CoCl$_2$ in tetrahydrofuran, were grown by slow evaporation from 10% acetone in CH$_2$Cl$_2$ at 23 °C, and a suitable crystal was mounted on a glass fiber in an arbitrary orientation. The Enraf-Nonius program SEARCH was used to obtain 25 accurately centered reflections which were used in the program INDEX to obtain an orientation matrix for data collection and to provide approximate cell dimensions. Refined cell dimensions and their estimated standard deviations were obtained from 28 accurately centered reflections. The mosaicity of the crystals was examined by the ω scan technique and judged to be satisfactory. The space group was chosen on the basis of systematic absences, later confirmed by the successful solution and refinement of the structure.

Diffraction data were collected at 295 K on an Enraf-Nonius four-circle DAD-4 diffractometer controlled by a PDP8/M computer using Mo Kα radiation from a highly oriented graphite crystal monochromator (see Table I for data collection parameters). Re-

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Table II. Positional Parameters and Their Estimated Standard Deviations for $[Et_3NH]^+[(C_2B_9H_{11})_2Co]^-$

atom	*x*	*y*	*z*	atom	*x*	*y*	*z*
Co	0.16504 (2)	0.2500 (0)	−0.06126 (8)	N	0.4215 (2)	0.0428 (5)	0.0399 (7)
C(1M)	0.3865 (2)	0.1189 (8)	−0.0480 (9)	C(3E)	0.4222 (3)	0.1600 (8)	0.3345 (13)
C(2M)	0.4515 (3)	−0.0143 (7)	−0.1129 (10)	C(1)	0.2342 (2)	0.2467 (7)	−0.0098 (7)
C(3M)	0.4506 (2)	0.0997 (7)	0.1877 (10)	C(2)	0.2212 (2)	0.2872 (4)	−0.2227 (7)
C(1E)	0.4067 (3)	0.2314 (8)	−0.1341 (12)	C(1′)	0.1542 (2)	0.0781 (5)	−0.1328 (8)
C(2E)	0.4832 (3)	−0.1041 (8)	−0.0385 (13)	C(2′)	0.1450 (2)	0.1063 (5)	0.0878 (7)
B(3)	0.1815 (3)	0.3958 (6)	−0.2248 (9)	B(3′)	0.1095 (2)	0.2227 (7)	0.1179 (9)
B(4)	0.1698 (2)	0.4260 (6)	0.0233 (10)	B(4′)	0.0944 (2)	0.2641 (7)	−0.1211 (9)
B(5)	0.2045 (2)	0.3260 (6)	0.1537 (9)	B(5′)	0.1255 (2)	0.1715 (6)	−0.2740 (9)
B(6)	0.2747 (2)	0.3279 (7)	−0.1352 (12)	B(6′)	0.1215 (3)	−0.0182 (7)	−0.0073 (12)
B(7)	0.2412 (2)	0.4233 (6)	−0.2674 (10)	B(7′)	0.0909 (3)	0.0732 (7)	0.1481 (11)
B(8)	0.2098 (3)	0.5159 (6)	−0.1099 (11)	B(8′)	0.0572 (3)	0.1719 (8)	0.0150 (14)
B(9)	0.2245 (3)	0.4699 (6)	0.1226 (10)	B(9′)	0.0669 (3)	0.1410 (7)	−0.2265 (11)
B(10)	0.2639 (3)	0.3540 (7)	0.1048 (11)	B(10′)	0.1069 (3)	0.0238 (6)	−0.2391 (10)
B(11)	0.2670 (3)	0.4717 (7)	−0.0560 (11)	B(11′)	0.0636 (3)	0.0247 (7)	−0.0592 (10)
H(N)	0.411 (2)	0.004 (6)	0.123 (8)	H(1E2)	0.438 (2)	0.243 (10)	−0.084 (9)
H(1M1)	0.366 (3)	0.141 (7)	0.054 (9)	H(1E3)	0.412 (2)	0.216 (7)	−0.270 (10)
H(1M2)	0.370 (2)	0.068 (7)	−0.144 (11)	H(2E1)	0.499 (3)	−0.122 (7)	0.078 (8)
H(2M1)	0.469 (3)	0.049 (7)	−0.162 (11)	H(2E2)	0.470 (3)	−0.173 (10)	−0.069 (10)
H(2M2)	0.426 (3)	−0.044 (7)	−0.194 (11)	H(2E3)	0.508 (3)	−0.118 (9)	−0.123 (11)
H(3M1)	0.474 (3)	0.153 (8)	0.116 (10)	H(3E1)	0.399 (3)	0.203 (7)	0.284 (12)
H(3M2)	0.470 (3)	0.043 (7)	0.237 (11)	H(3E2)	0.444 (3)	0.206 (9)	0.432 (11)
H(1E1)	0.387 (2)	0.319 (7)	−0.131 (10)	H(3E3)	0.406 (3)	0.109 (9)	0.407 (10)
H(1)	0.252 (2)	0.154 (6)	−0.007 (10)	H(1′)	0.184 (3)	0.052 (6)	−0.163 (10)
H(2)	0.224 (2)	0.244 (8)	−0.314 (9)	H(2′)	0.168 (2)	0.094 (6)	0.184 (9)
H(3)	0.160 (2)	0.416 (7)	−0.339 (11)	H(3′)	0.100 (2)	0.246 (8)	0.247 (9)
H(4)	0.147 (2)	0.477 (6)	0.106 (8)	H(4′)	0.084 (2)	0.354 (6)	−0.157 (9)
H(5)	0.193 (2)	0.292 (5)	0.293 (8)	H(5′)	0.137 (2)	0.195 (6)	−0.407 (8)
H(6)	0.297 (2)	0.281 (7)	−0.223 (11)	H(6′)	0.129 (3)	−0.100 (10)	0.015 (15)
H(7)	0.252 (2)	0.447 (6)	−0.399 (8)	H(7′)	0.081 (2)	0.042 (5)	0.278 (8)
H(8)	0.193 (2)	0.602 (8)	−0.095 (9)	H(8′)	0.023 (2)	0.191 (7)	0.058 (9)
H(9)	0.223 (2)	0.526 (6)	0.250 (10)	H(9′)	0.042 (2)	0.150 (5)	−0.330 (8)
H(10)	0.287 (2)	0.336 (5)	0.230 (8)	H(10′)	0.114 (2)	−0.003 (7)	−0.370 (9)
H(11)	0.286 (3)	0.527 (7)	−0.045 (10)	H(11′)	0.030 (2)	−0.029 (7)	−0.080 (8)

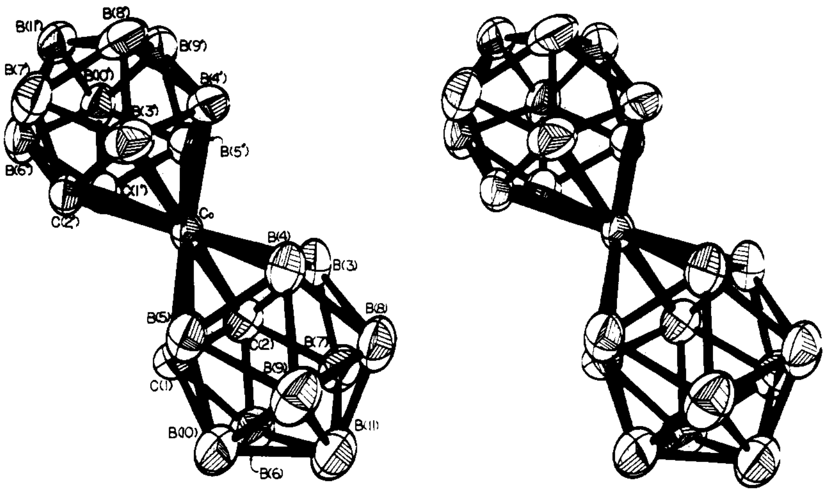

Figure 1. Stereoview of the $Co(1,2\text{-}C_2B_9H_{11})_2^-$ anion. Hydrogen atoms are omitted for clarity.

duction of the data was conducted by standard procedures described elsewhere.[5] Raw intensity data were corrected for Lorentz-polarization effects and for absorption. Those reflections for which $F_o^2 > 3\sigma(F_o^2)$, where F_o^2 was estimated from counting statistics ($p = 0.03$),[6] were used in the final refinement of the structural parameters.

Full-matrix least-squares refinement was based on F, and the function minimized was $\sum w(|F_o| - |F_c|)^2$. The weights w were taken as $2F_o/(F_o^2)$, where F_o and F_c are the observed and calculated structure factor amplitudes, respectively. The atomic scattering factors for nonhydrogen atoms were taken from Cromer and Waber[7] and those for hydrogen from Stewart et al.[8] The effects of anomalous dispersion for all nonhydrogen atoms were included in F with values of Cromer and Ibers[9] for $\Delta f'$ and $\Delta f''$.

The position of the cobalt atom was determined from a three-dimensional Patterson function calculated from all the intensity data, and most of the remaining nonhydrogen atoms were located from subsequent Fourier difference maps. The cage carbon positions were unambiguously located from the characteristically short C–C vectors and from their smaller isotropic thermal parameters compared to boron. The positions of C(2), B(3), C(1′), B(5′), and a methyl carbon atom on the cation were calculated and included in subsequent refinements. After anisotropic temperature factors were introduced for all nonhydrogen atoms, Fourier difference maps located most of the H atoms. The remainder, consisting of several methyl and

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Table III. Bond Distances (Å) and Important Angles (Deg)

	unprimed[a]	primed[a]		unprimed[a]	primed[a]
Co–C(1)	2.044 (3)	2.058 (3)	B(4)–B(5)	1.787 (6)	1.766 (6)
Co–C(2)	2.039 (3)	2.042 (3)	B(4)–B(8)	1.819 (6)	1.793 (7)
Co–B(3)	2.090 (4)	2.080 (4)	B(4)–B(9)	1.812 (6)	1.785 (7)
Co–B(4)	2.109 (4)	2.099 (4)	B(5)–B(9)	1.763 (6)	1.769 (6)
Co–B(5)	2.100 (4)	2.102 (4)	B(5)–B(10)	1.784 (6)	1.790 (6)
C(1)–C(2)	1.626 (5)	1.621 (5)	B(6)–B(7)	1.737 (7)	1.767 (8)
C(1)–B(5)	1.710 (6)	1.686 (5)	B(6)–B(10)	1.757 (7)	1.765 (6)
C(1)–B(6)	1.745 (6)	1.707 (6)	B(6)–B(11)	1.755 (7)	1.793 (7)
C(1)–B(10)	1.708 (6)	1.683 (6)	B(7)–B(8)	1.793 (7)	1.767 (8)
C(2)–B(3)	1.696 (5)	1.703 (6)	B(7)–B(11)	1.766 (6)	1.762 (7)
C(2)–B(6)	1.739 (6)	1.720 (6)	B(8)–B(9)	1.782 (6)	1.780 (8)
C(2)–B(7)	1.692 (5)	1.674 (6)	B(8)–B(11)	1.776 (7)	1.780 (7)
B(3)–B(4)	1.828 (6)	1.811 (6)	B(9)–B(10)	1.757 (8)	1.777 (7)
B(3)–B(7)	1.787 (6)	1.810 (6)	B(9)–B(11)	1.764 (7)	1.787 (6)
B(3)–B(8)	1.801 (6)	1.781 (7)	B(10)–B(11)	1.770 (6)	1.793 (7)
N–C(1M)	1.478 (6)	N–C(3M)	1.495 (5)	C(2M)–C(2E)	1.481 (7)
N–C(2M)	1.537 (6)	C(1M)–C(1E)	1.542 (8)	C(3M)–C(3E)	1.499 (7)
N–H(N)	0.789 (3)	⟨C(E)–H⟩	0.981	⟨B–H⟩	1.050
⟨C(M)–H⟩	0.982	⟨C(cage)–H⟩	0.972		
C(1)–Co–C(1′)	100.2 (2)	B(4)–Co–B(3′)	91.1 (2)		
C(1)–Co–C(2′)	99.9 (2)	B(4)–Co–B(4′)	92.8 (2)		
C(1)–Co–B(3′)	130.7 (2)	B(4)–Co–B(5′)	130.6 (2)		
C(1)–Co–B(4′)	176.5 (2)	B(5)–Co–C(1′)	131.3 (2)		
C(1)–Co–B(5′)	131.2 (2)	B(5)–Co–C(2′)	96.5 (1)		
C(2)–Co–C(1′)	100.6 (1)	B(5)–Co–B(3′)	92.4 (2)		
C(2)–Co–C(2′)	133.5 (2)	B(5)–Co–B(4′)	130.5 (2)		
C(2)–Co–B(3′)	175.3 (2)	B(5)–Co–B(5′)	179.0 (2)		
C(2)–Co–B(4′)	130.8 (2)	C(1M)–N–C(2M)	110.0 (3)		
C(2)–Co–B(5′)	97.1 (1)	C(1M)–N–C(3M)	115.3 (4)		
B(3)–Co–C(1′)	131.5 (2)	C(1M)–N–H(N)	134.7 (3)		
B(3)–Co–C(2′)	176.1 (2)	C(2M)–N–C(3M)	111.1 (3)		
B(3)–Co–B(3′)	129.6 (2)	C(2M)–N–H(N)	138.9 (3)		
B(3)–Co–B(4′)	93.0 (2)	C(3M)–N–H(N)	133.6 (3)		
B(3)–Co–B(5′)	93.9 (2)	N–C(1M)–C(1E)	113.5 (4)		
B(4)–Co–C(1′)	174.5 (2)	N–C(2M)–C(2E)	113.3 (4)		
B(4)–Co–C(2′)	129.9 (2)	N–C(3M)–C(3E)	112.2 (4)		

[a] Atoms in one of the two cages are designated with primes (see Figure 1).

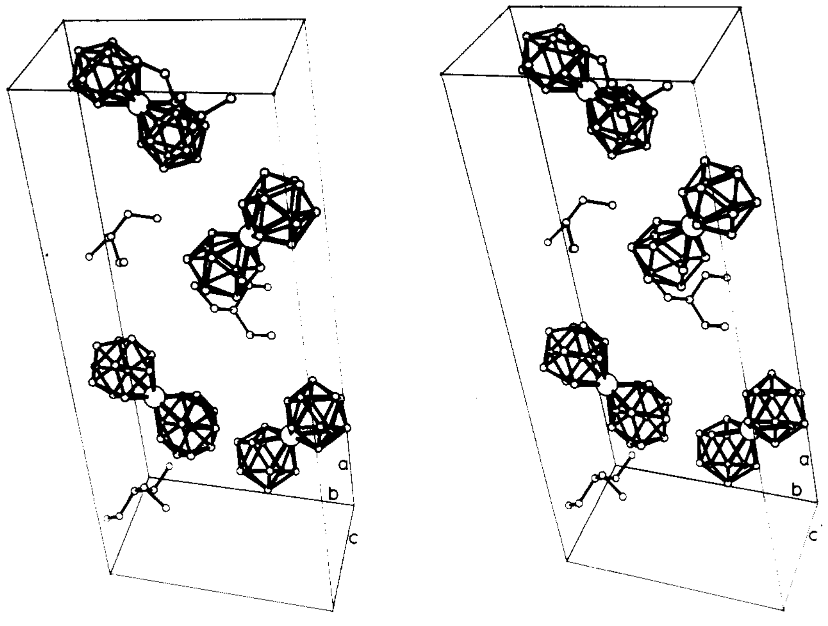

Figure 2. Unit cell packing for $(C_2H_5)_3NH^+Co(1,2\text{-}C_2B_9H_{11})_2^-$.

methylene hydrogens, were inserted in their calculated positions, refined for several cycles and thereafter held fixed.

Final positional parameters and important bond distances and angles are given in Tables II and III, respectively, while Figures 1 and 2 present stereoviews of the cobaltacarborane anion and the unit cell packing. Tables of observed and calculated structure factors and calculated mean planes are available as supplementary material (see paragraph at end of paper). The computing system and programs are described elsewhere.[10]

Results and Discussion

The $Co(1,2\text{-}C_2B_9H_{11})_2^-$ anion consists of two $C_2B_9H_{11}^{2-}$ (dicarbollide) units sandwiched around a formal Co^{3+} ion with the C_2B_3 faces on the two ligands nearly parallel (dihedral angle 3.7°). The vector distances from cobalt to the C_2B_3

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[10] Freyberg, D. P.; Mockler, G. M.; Sinn, E. *J. Chem. Soc., Dalton Trans.* **1976**, 447.

planes are virtually identical (1.466 and 1.476 Å), and the metal is approximately equidistant from the facial boron and carbon atoms although the Co–C distances are slightly shorter than the Co–B vectors [mean values 2.046 (2) and 2.097 (2) Å, respectively]. The difference of 0.05 Å correlates well with the larger covalent radius of boron vs. carbon (0.82 vs. 0.77 Å).

The two $C_2B_9H_{11}^{2-}$ ligands are mutally rotated by 37°, as measured by the dihedral angle subtended by the two planes through cobalt and bisecting the C(1)–C(2) and C(1′)–C(2′) bonds. This produces a staggered orientation of the carborane cages with the two C–C edges in close proximity, the molecule having overall approximate (not crystallographic) C_2 symmetry. The ligand orientation in this species is very similar to that previously found[4] in the isoelectronic nickel(IV) complex $Ni(1,2\text{-}C_2B_9H_{11})_2$, in which the mutual rotation of the ligands away from an eclipsed position is 36°. In contrast, the electron-rich sandwich complex $Ni^{III}(1,2\text{-}C_2B_9H_{11})_2^-$ exhibits a trans orientation with respect to the carbons in the two ligands.[11]

As noted above, the earlier X-ray study of $Cs^+[Co(1,2\text{-}C_2B_9H_{11})_2]^-$ did not locate the carbon atoms and hence the ligand orientation could not be determined. However, in the bis(8,9,12-tribromo) derivative of this ion[12] the cages adopt a trans arrangement with the facial carbon atoms on opposite sides of cobalt, undoubtedly because of steric repulsion involving bromine substituent on the cages. In both $Co(C_2B_9H_{11})_2^-$ and $Ni(C_2B_9H_{11})_2$, each 12-vertex polyhedron is a closo 26-electron ($2n + 2$) system and the metals satisfy the 18-electron rule; this is reflected in the normal metal–carbon, metal–boron, carbon–carbon, and carbon–boron bond distances and in the absence of any significant distortion from regular 12-vertex closo geometry. These systems can be contrasted with the slip-distorted sandwich complexes of 1,2- and 1,7-$C_2B_9H_{11}^{2-}$, referred to above, which usually incorporate d^n metal ions where $n > 6$. Such species have been extensively discussed elsewhere.[1,13]

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Registry No. $(C_2H_5)_3NH^+[Co(1,2\text{-}C_2B_9H_{11})_2]^-$, 80584-07-2.

Supplementary Material Available: Listings of observed and calculated structure factors, anisotropic thermal parameters, and least-squares planes (13 pages). Ordering information is given on any current masthead page.

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Contribution from the Institute for Physical Chemistry,
University of Frankfurt,
D 6000 Frankfurt/M-1, Federal Republic of Germany

Pressure and Temperature Dependencies of the Mercury-Catalyzed Aquation of *mer*-$RhCl_3(OH_2)_3$ in Acidic Aqueous Solution

Walter Weber, Donald A. Palmer,[1] and Hartwig Kelm*

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The kinetics and stereochemistry of aquation and chloride anation of the series of complexes $RhCl_x(OH_2)_{6-x}^{(3-x)+}$ where $x = 0$–6, in acidic aqueous solutions have been studied in detail[2-6] and shown to be dominated by the trans effect of coordinated chloride.[6] Water exchange[7] with $Rh(OH_2)_6^{3+}$ and chloride exchange[4] with $RhCl_6^{3-}$ have been interpreted in terms of a dissociative mechanism. Consequently, the remaining reactions within the sequence may be safely assumed to conform to this assignment. In order to explain the steric course of these reactions, it was necessary to refine the mechanism further to include a tetragonal-pyramidal intermediate.[6]

A high-pressure kinetic study[8] of the reactions involving the $RhCl_6^{3-}$, $RhCl_5(OH_2)^{2-}$, and *cis*-$RhCl_4(OH)_2)_2^-$ complex ions indicated that the large positive ΔV^* values observed were also directly compatible with a D mechanism.

Chan and Harris[9] recently investigated the mercury-catalyzed aquation of the species *fac*-$RhCl_3(OH_2)_3$, *cis*-$RhCl_2(OH_2)_4^+$, and $RhCl(OH_2)_5^{2+}$. These species are virtually immune to aquation in acidic solution, presumably due to the greater trans effect of the chloride ligands with respect to the aquo ones. From the relative magnitudes of the formation constants of the respective precursor complexes $HgRhCl_x(OH_2)_{6-x}^{(5-x)+}$, they[9] proposed that an electrostatic interaction was responsible for their formation.

It was our hope that a knowledge of the volume changes associated with precursor formation and its subsequent decomposition would shed more light on the nature of the interactions involved.

Experimental Section

The *fac*-$RhCl_3(OH_2)_3$ and *mer*-$RhCl_3(OH_2)_3$ isomers were prepared in solution from $K_2[RhCl_5(OH_2)]$ and separated on a mixed-bed, ion-exchange column containing equal parts of Lewatit S1080 (H^+ form) and Lewatit M5080 (Cl^- form) by the method of Palmer and Harris.[6] These solutions were then shaken with a weak basic anion-exchange resin (Lewatit MP7080) in the ClO_4^- form to remove traces of free chloride ion.

The *fac* isomer was characterized by absorption maxima at 376 and 473 nm with the ratio of the respective extinction coefficients being 1.26, which compares very favorably with the value of 1.27 for $\epsilon(376\ \text{nm})/\epsilon(473\ \text{nm})$ recorded previously[6] but differs from a more recent[9] value of 1.11 for $\epsilon(376)/\epsilon(474)$. Absorption maxima for the *mer* species were found at 370 and 469 nm with a resulting extinction coefficient ratio of 0.89 (cf. $\epsilon(370)/\epsilon(469) = 0.84$).[6]

It should be mentioned that we were unsuccessful in our attempts to separate these two isomers using the above mentioned resins in the H^+ and ClO_4^- forms. Furthermore, the ratio of the extinction coefficients reported by Chan and Harris[9] for the *fac* isomer may indicate the presence of some *mer* isomer in their sample. Apart from an immediate spectral change, which occurred when mixing solutions of *fac*-$RhCl_3(OH_2)_3$ and $Hg(ClO_4)_2$, only a very small change took place subsequently—presumably due to the catalyzed aquation reaction—such that we were unable to study this reaction.

Solutions of mercury(II) perchlorate were prepared and analyzed as described previously.[9] Water used to make up these solutions was purified with a commercial ion-exchange column and a double-distillation assembly.

The ionic strength and acidity were kept constant at 2.0 M by the addition of $HClO_4$. The complex concentration was maintained at 2.5×10^{-4} M.

The spectra were recorded on a Zeiss PMA 10 spectrophotometer whereas the kinetics were investigated on a Zeiss PMQ II adapted to accommodate a "split-beam" differential amplifier and a high-pressure vessel.[10] The quartz cell seated inside the vessel was con-

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