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Notes

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A Unidentate Attachment of the Tetrahydroborate Group. Crystal and Molecular Structure of (Tetrahydroborato)[1,1,1-tris((diphenylphosphino)methyl)ethane-*P*,*P'*,*P''*]copper(I)

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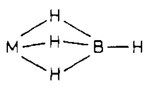
The tetrahydroborate ion BH_4^- can form transition-metal complexes in which it coordinates the metal by means of three (I), two (II), and one (III) hydrogen atoms.[1,2] While the bonding modes I and II are nearly common, the latter has been only recently ascertained in the complex $(PPh_2Me)_3Cu(BH_4)$ by X-ray and neutron diffraction analyses.[3,4]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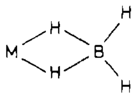
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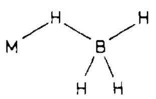
III

On the other hand, the bidentate linkage of the BH_4^- ion previously found in the $(PPh_3)_2Cu(BH_4)$ complex[5] seems to suggest that the attachment mode of the tetrahydroborate in the copper(I) complexes is essentially determined by the size and the coordinating capability of the ancillary ligands. Indeed, the replacement in the latter complex of a phenyl group by a methyl, reducing the steric requirement, can allow the linkage of three phosphines to the metal. Consequently, owing to the known trend of the copper(I) to be tetrahedrally coordinated, only one coordination site remains available for the tetrahydroborate group.

In this framework the attachment mode of the BH_4 group in the (triphos)$Cu(BH_4)$ complex,[6] where triphos is the tridentate tripod-like ligand 1,1,1-tris((diphenylphosphino)methyl)ethane, $CH_3C(CH_2PPh_2)_3$, has to be reconsidered. Therefore, we have undertaken a structural investigation of this complex, which some years ago had been hypothesized as isostructural with the isomorphous derivative (triphos)$Co(BH_4)$, where triphos and tetrahydroborate ligands act as tri- and bidentate, respectively.[6]

Experimental Section

The complex (triphos)$Cu(BH_4)$ was prepared with the method previously described.[6]

Collection and Reduction of X-ray Data. A white well-formed crystal of approximate dimensions 0.12 × 0.40 × 0.70 mm, delimited by the faces 100, $\bar{1}00$, 010, $0\bar{1}0$, 001, and $00\bar{1}$, was used for data collection. Diffraction data were collected on a Philips PW 1100 automatic diffractometer using graphite-monochromated Mo Kα radiation. Unit cell dimensions were determined from a least-squares refinement of the angular settings of 20 carefully centered reflections. The crystals, which are isomorphous with those of the (triphos)$Co(BH_4)$ compound,[6] are orthorhombic and belong to the space group $Pn2_1a$, with four molecules in a unit cell of dimensions $a = 20.482$ (8), $b = 17.275$ (7), and $c = 10.334$ (5) Å. The calculated density for four formula units per cell is 1.277 g/cm^3. Intensity data within $2\theta \leq 50°$ were collected at the scan speed of 0.07°/s by using the ω–2θ scan technique, with a scan range calculated according to the expression $A + B \tan \theta$,[7] where $A = 0.7°$ and $B = 0.69$. Stationary-background measurements were taken before and after each scan for a time equal to half the scan time. The intensity of three standard reflections measured every 120 readings showed no systematic trend. After correction for background, the intensities were assigned standard deviations calculated as described elsewhere,[8] by using the value 0.03 for the instability factor k. Of 3633 total reflections, 2016 were considered observed with $I \geq 3\sigma(I)$. Intensity data were corrected for Lorentz and polarization effects and merged to a unique set of reflections. An absorption correction (μ(Mo Kα) = 7.54 cm^{-1}) was applied, the transmission coefficients ranging from 0.91 to 0.75.

Atomic scattering factors of the appropriate neutral atoms were taken from ref 9 for non-hydrogen atoms and from ref 10 for hydrogen atoms. Both the $\Delta f'$ and $\Delta f''$ components of the anomalous dispersion were included for all non-hydrogen atoms.[11] The function $\sum w(|F_o| - |F_c|)^2$ was minimized during the least-squares refinement process; the assigned weights are given as $w = 1/\sigma^2(F_o)$. The calculations were carried out by using the SHELX76 crystallographic system[12] on a SEL 32/70 computer.

Solution and Refinement of the Structure. On the assumption that the compound is isomorphous with (triphos)$Co(BH_4)$, the final parameters of this structure[6] were used as starting parameters for (triphos)$Cu(BH_4)$. Full-matrix least-squares refinement by assigning anisotropic temperature factors to copper and phosphorus and isotropic factors to carbon and boron atoms was undertaken. Throughout the refinement the phenyl groups were treated as rigid bodies of D_{6h} symmetry. The hydrogen atoms belonging to the triphos ligand were introduced in their calculated positions but not refined. At this stage a difference Fourier map, calculated with removal of the high-angle ($(\sin \theta)/\lambda \geq 0.3$ Å^{-1}) reflections from the calculation, revealed the tetrahydroborate hydrogen atoms among the first eight peaks of the map. Attempts to refine such hydrogen positions, using all the data as well as low-angle reflections, gave poor results with bond distances and angles within the tetrahydroborate anion quite far from the expected values. Thus a rigid-body refinement of the tetrahydroborate group was performed with constraints (B–H = 1.19 ± 0.01 Å, H–H = 1.94 ± 0.01 Å)[13] that, even imposing tetrahedral geometry to the BH_4 ion, leave that group free to move with respect to the P_3Cu fragment. An overall temperature factor was used for the tetrahydroborate group. In this way the refinement progressed and converged well, confirming the monodentate attachment of the BH_4 anion. Although the coordinates from the rigid-body refinement appeared somewhat shifted with respect to those of the individual-atom refinement, the position of the bridging hydrogen atom remained in both the refinements very close to the location from the difference Fourier map, with the three terminal hydrogen atoms at distances

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Table I. Positional and Thermal Parameters[a]

atom	x	y	z	U, Å^2
Cu	−689 (1)	−1321	748 (1)	anis
P1	406 (1)	−1298 (2)	1275 (3)	anis
P2	−622 (1)	−133 (2)	−257 (3)	anis
P3	−1020 (1)	−852 (2)	2720 (3)	anis
C1	349 (6)	928 (7)	2849 (10)	49 (3)
C2	36 (5)	224 (6)	2181 (10)	37 (2)
C3	600 (5)	−276 (6)	1650 (10)	44 (3)
C4	−406 (5)	540 (6)	1070 (10)	40 (3)
C5	−358 (5)	−207 (6)	3252 (10)	43 (3)
C6	680 (4)	−1845 (4)	2709 (8)	44 (3)
C7	1273 (4)	−1693 (4)	3326 (8)	61 (3)
C8	1478 (4)	−2161 (4)	4347 (8)	85 (4)
C9	1092 (4)	−2781 (4)	4752 (8)	87 (4)
C10	500 (4)	−2932 (4)	4136 (8)	80 (4)
C11	294 (4)	−2464 (4)	3114 (8)	59 (3)
C12	1031 (3)	−1578 (4)	89 (6)	40 (3)
C13	1649 (3)	−1239 (4)	36 (6)	47 (3)
C14	2104 (3)	−1494 (4)	−873 (6)	63 (4)
C15	1942 (3)	−2088 (4)	−1729 (6)	69 (4)
C16	1324 (3)	−2427 (4)	−1676 (6)	75 (4)
C17	869 (3)	−2172 (4)	−767 (6)	57 (3)
C18	33 (4)	−16 (4)	−1455 (7)	41 (3)
C19	354 (4)	686 (4)	−1653 (7)	70 (4)
C20	815 (4)	754 (4)	−2641 (7)	92 (5)
C21	954 (4)	120 (4)	−3431 (7)	76 (4)
C22	632 (4)	−582 (4)	−3233 (7)	64 (3)
C23	171 (4)	−650 (4)	−2245 (7)	57 (3)
C24	−1306 (3)	359 (4)	−1035 (7)	41 (3)
C25	−1356 (3)	1164 (4)	−1070 (7)	58 (3)
C26	−1873 (3)	1514 (4)	−1725 (7)	64 (4)
C27	−2341 (3)	1059 (4)	−2345 (7)	71 (4)
C28	−2291 (3)	254 (4)	−2310 (7)	71 (4)
C29	−1774 (3)	−96 (4)	−1655 (7)	62 (3)
C30	−1146 (3)	−1504 (4)	4083 (6)	36 (3)
C31	−936 (3)	−1342 (4)	5339 (6)	48 (3)
C32	−1062 (3)	−1867 (4)	6335 (6)	66 (4)
C33	−1397 (3)	−2553 (4)	6075 (6)	59 (3)
C34	−1606 (3)	−2714 (4)	4819 (6)	55 (3)
C35	−1481 (3)	−2190 (4)	3823 (6)	51 (3)
C36	−1741 (3)	−229 (4)	2849 (6)	40 (3)
C37	−1865 (3)	205 (4)	3961 (6)	54 (3)
C38	−2402 (3)	701 (4)	4004 (6)	69 (4)
C39	−2815 (3)	763 (4)	2936 (6)	71 (4)
C40	−2691 (3)	329 (4)	1825 (6)	75 (4)
C41	−2154 (3)	−167 (4)	1781 (6)	55 (3)
B	−1241 (9)	−2488 (10)	−12 (17)	77 (5)
H1	−731 (15)	−2180 (27)	162 (70)	77 (5)
H2	−1534 (34)	−2165 (38)	−844 (52)	77 (5)
H3	−1552 (30)	−2482 (41)	960 (45)	77 (5)
H4	−1146 (25)	−3143 (17)	−328 (70)	77 (5)

[a] Coordinates are multiplied by 10^4 and temperature factors by 10^3. The y coordinate of the Cu atom was chosen to have accordance with the y coordinate of the metal atom in the (triphos)Co(BH_4).[6]

larger than 2.68 (6) Å from the copper atom.

The absolute configuration of the structure was determined by applying the anomalous dispersion corrections. A refinement of the two configurations x, y, z and $x, \bar{y}, z$ gave R factor values of 0.055 and 0.58, respectively. An analysis of the standard deviations confirmed that the x, y, z structure was the correct one. The final value of the R_w factor is 0.058. Final positional and thermal parameters are listed in Tables I and II (Table II is supplementary material).

Results and Discussion

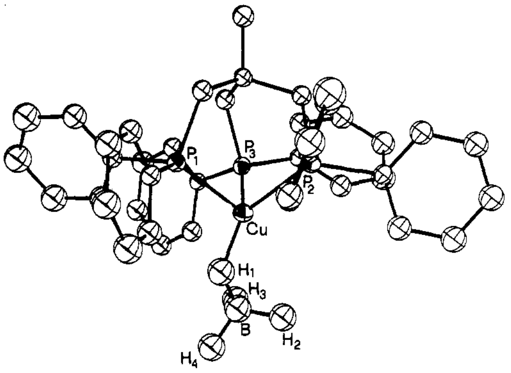

Figure 1. Perspective view of the complex molecule (triphos)Cu(BH_4) (ORTEP drawing with 30% probability ellipsoids).

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

Cu–P1	2.308 (3)	P1–C12	1.839 (7)
Cu–P2	2.304 (3)	P2–C4	1.851 (10)
Cu–P3	2.296 (3)	P2–C18	1.836 (8)
Cu–B	2.44 (2)	P2–C24	1.827 (8)
Cu–H1	1.60 (4)	P3–C5	1.839 (11)
P1–C3	1.850 (11)	P3–C30	1.822 (7)
P1–C6	1.845 (9)	P3–C36	1.832 (8)
P1–Cu–P2	91.9 (1)	P3–Cu–B	116.1 (4)
P1–Cu–P3	94.1 (1)	P1–Cu–H1	99 (1)
P2–Cu–P3	95.9 (1)	P2–Cu–H1	131 (3)
P1–Cu–B	122.7 (4)	P3–Cu–H1	130 (2)
P2–Cu–B	128.2 (4)	Cu–H1–B	121 (3)

The molecular structure of the complex consists of monomolecular (triphos)Cu(BH_4) units. A perspective view of the molecule is shown in Figure 1, and selected bond distances and angles are reported in Table III. The copper atom is linked to the three phosphorus atoms of the triphos ligand and to one bridging hydrogen atom of the tetrahydroborate group in a distorted tetrahedral environment. The closest nonbonded hydrogen atom is at a distance of 2.68 (6) Å. The monodentate attachment of the tetrahydroborate ligand is unequivocally confirmed by the 2.44 (2) Å Cu–B distance.[14] This value is only slightly shorter than the value of 2.518 (3) Å reported for $(PPh_2Me)_3Cu(BH_4)$ containing a monodentate tetrahydroborate group.[4] This difference can be probably accounted for by the slightly smaller size of the tripod ligand with respect to the three monodentate phosphines (see below). On the other hand the value of the Cu–B distance is significantly larger than the value of 2.184 (9) Å reported for $(PPh_3)_2Cu(BH_4)$, where a bidentate BH_4 attachment is present.[5] The Cu–H–B fragment is definitively bent with an angle of 121 (3)°, which matches the more reliable value of 121.7 (4)° reported in the neutron diffraction study of $(PPh_2Me)_3Cu(BH_4)$.[4] The bridging hydrogen atom, whose distance from the metal atom of 1.60 (4) Å is in good agreement with the value of 1.697 (5) Å in $(PPh_2Me)_3Cu(BH_4)$,[4] is significatively displaced from the pseudo-threefold axis defined by the CuP_3 group. As a matter of fact the P–Cu–H1 angles range from 99 (1) to 131 (3)°. On the contrary the P_3CuB fragment is less distorted with the P–Cu–B angles ranging from 116.1 (4) to 128.2 (4)°.

With regard to the P–Cu–P angles in this compound, a comparison with the $(PPh_2Me)_3Cu(BH_4)$ complex evidences some differences (91.9 (1)–95.9 (1) vs. 108.9 (1)–117.2 (1)°), which can be well justified on the basis of the steric requirement of the tripod-like ligand. On the other hand, the same angles, owing to the reduced steric requirement of the monodentate tetrahydroborate group, are somewhat larger than those reported for the complex (triphos)Co(BH_4) containing a bidentate BH_4 group (89.6 (2)–93.3 (2)°).[6]

The present study provides evidence for the fact that the monodentate attachment of the tetrahydroborate group in this complex is imposed by the tendency of the copper atom toward the tetrahedral geometry rather than steric requirements, as demonstrated by the bidentate attachment of the BH_4 group in the isomorphous (triphos)Co(BH_4) complex.[6]

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The different attachment mode of the BH_4 group in the two isomorphous copper and cobalt analogues is not surprising, since the isomorphism of these complexes is mainly due to the requirements of the triphos ligand. As a matter of fact, all the compounds of the series (triphos)M(L) are isomorphous in spite of the nature of the ancillary ligand (L = I,[15] CS_2,[16] SO_2,[6] SO_4,[17] SeO_4,[17] and BH_4[6,18]).

Registry No. (triphos)Cu(BH_4), 82678-89-5.

Supplementary Material Available: A listing of structure factor amplitudes and Table II giving anisotropic thermal parameters (13 pages). Ordering information is given on any current masthead page.

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Kinetics of the Permanganate Ion Oxidation of the Bis(μ-oxo)bis(oxomolybdate(V)) Ion

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Studies of the kinetics behavior of the permanganate ion with one-electron inorganic reductants in acidic media have shown the Mn(VII) → Mn(VI) step to be rate determining, although the MnO_4^- ion is a strong multiequivalent oxidant.[2,3] Since the reductant ions have been limited to those that undergo a one-electron change, it would be of interest to investigate the kinetic behavior of MnO_4^- with a reductant system that provides the opportunity of a two-electron change. Such a reductant might represent conditions in which the MnO_4^- ion would react via a two-electron step (Mn(VII) → Mn(V)). The reactions of Pt(II) → Pt(IV)[4] and U(IV) → U(VI)[5] with MnO_4^- have been characterized, by means of ^{18}O-exchange experiments, as occurring via oxygen atom transfer.

The bis(μ-oxo)bis(oxomolybdate(V)) ion, $Mo_2O_4^{2+}$, was chosen because of its dimeric nature,[6–8] its ability to be quantitatively oxidized to Mo(VI)[9] by MnO_4^-, and the possible detection of a mixed-valence (Mo^V/Mo^{VI}) intermediate.[10] There also exists the possibility of estimating the electrode potential for the $Mo^V_2 \rightarrow Mo^VMo^{VI} + e^-$ reaction.[11] Murmann has shown the rate of oxygen exchange between $Mo_2O_4^{2+}$ and solvent and the rate of the opening of the bridging ring to be slow.[12] Redox experiments by Sykes have led to the characterization of the aquo dimer as an outer-sphere reactant.[13]

Experimental Section

The bis(μ-oxo)bis(oxomolybdate(V)) complex was prepared by three different methods. (a) Reduction of Na_2MoO_4 (8.0 g) in 250 mL of 3 M HCl was accomplished at 80 °C by slow addition with stirring of $N_2H_4{\cdot}2HCl$ (6 g).[9,13] The brown Mo^V_2 solution was heated for 2–3 h to remove excess hydrazine and reduce the volume. This method was discontinued as it gave low stoichiometric values and erratic rate constants. The results were presumably due to the presence of $N_2H_5^+$. (b) Reduction of the Na_2MoO_4/3M HCl solution was accomplished by shaking with metallic mercury.[14] The Hg and Hg_2Cl_2 were filtered, and the solution was stored at 5 °C over fresh Hg. (c) Dimerization of monomeric Mo(V) was accomplished by dissolving weighed samples of $(NH_4)_2(MoOCl_5)$ (gift from Climax Molybdenum) in 2 M HCl, followed by aquation for several hours.[15]

Table I. Rate Constants, k_{obsd}, for the $MnO_4^-/Mo_2O_4^{2+}$ Reaction (T = 25.0 °C, [H^+] = 0.50 M, μ = 1.00 ($LiClO_4$))

$10^3 \times [Mo_2O_4^{2+}]_0$, M	$10^5 \times [MnO_4^-]_0$, M	k_{obsd}, s^{-1}	$10^3 \times [Mo_2O_4^{2+}]_0$, M	$10^5 \times [MnO_4^-]_0$, M	k_{obsd}, s^{-1}
0.90	4.68	5.35	1.47	7.72	8.00
2.29	4.68	12.9	2.29	9.37	12.0
3.37	7.72	19.2	5.38	7.72	31.0
4.40	11.3	23.2	6.60	15.1	36.6[a]
4.87	4.91	27.0	6.60	15.1	36.5[b]
5.28	2.85	29.5[c]			

[a] $[Mo(VI)]_0 = 4.2 \times 10^{-3}$ M. [b] $[Mo(VI)]_0 = 2.1 \times 10^{-3}$ M. [c] $HClO_4$/HTFMS mixture.

Chloride-free solutions were obtained by diluting an aliquot of the dimer/HCl solution with distilled water such that [H^+] < 0.1 M and collecting the dimer on a Dowex 50-X8 (50–100 mesh) or Amberlite IR-120 ion-exchange column (acid form). The column was washed free of Cl^- with deoxygenated distilled water and the $Mo_2O_4^{2+}$ ion eluted with deoxygenated $HClO_4$ or trifluoromethanesulfonic acid (HTFMS) of the desired molarity and ionic strength (μ = 1.0, $LiClO_4$). While the decomposition of the dimer is slower in HTFMS than in $HClO_4$, identical kinetic results were obtained. The [$Mo_2O_4^{2+}$] was determined spectrophotometrically at λ = 384 nm (ϵ = 103 M^{-1} cm^{-1}).[16] Total Mo analysis was accomplished by H_2O_2 oxidation.[17] $LiClO_4$ (G. F. Smith) was recrystallized twice from distilled water, and stock solutions were standardized by ion-exchange methods. The permanganate stock solution was standardized against As_2O_3. Reaction MnO_4^- solutions were prepared by diluting aliquots of the standardized stock with distilled water or $H^+/LiClO_4$ solutions of known molarity and ionic strength. Solutions of HTFMS are slowly oxidized by MnO_4^-.

Results

The reaction stoichiometry in 0.4–1.0 M $HClO_4$ (μ = 1.0) was shown by visual and spectrophotometric titrations at 384 and 524 nm ($\lambda_{max}(MnO_4^-)$) to correspond to the ratio of 2.51 ± 0.30 (mol of $Mo_2O_4^{2+}$ consumed/mol of MnO_4^- added). The overall reaction can be represented by the equation

$$2Mn^{VII} + 5Mo^V_2 \rightarrow 2Mn^{II} + 10Mo^{VI}$$

With use of pseudo-first-order conditions ($[Mo_2O_4^{2+}]_0 \geq$

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