on the carbon atoms of the ligands does not change upon coordination.

For nitrogen as the ligand atom, the binding energy increases about 2 eV indicating an increased positive character of the nitrogen donor atom as expected.[6] In the case of phosphorus there is no significant change in the binding energy upon coordination to a variety of metals. The same observation has been made previously for Ni, Pd, and Cd[7] and was interpreted in terms of π back-donation. Thus ligand π back-bonding would also appear to be important for the interesting acceptor B_9H_{13} (Table III). If so, this is support for the argument that the lack of reactivity of the phosphine compound with respect to ligand displacement is due to donation from the boron cage to the phosphorus d orbitals.[21] There is also no change in the phosphorus 2p binding energies in $(C_6H_5)_3PO$ upon coordination in agreement with the observation of others.[8] For sulfur there is a barely significant increase in the binding energy upon coordination. As in the case of nitrogen, this would indicate that there is a net transfer of electrons to tin upon coordination. For tin, in the ligand $SnCl_3^-$, there is no significant change in the binding energy upon coordination to a variety of metals. Unfortunately, this may merely reflect the insensitivity of the tin core electrons to their chemical environment. This is easy to understand (Table II) in that a coordinate interaction causing a 2-V shift for nitrogen would cause only a 0.1-V shift for tin. No conclusion can be drawn concerning the role of π back-bonding in this situation.[22]

Registry No. SnF_2, 7783-47-3; $SnCl_2$, 7772-99-8; $SnCl_2 \cdot 2H_2O$, 10025-69-1; $SnBr_2$, 10031-24-0; SnI_2, 10294-70-9; SnO, 21651-19-4; $Sn(C_4H_9)_2(NCS)_2(o\text{-}C_{12}H_8N_2)$, 38882-72-3; $SnBr_4(C_4H_8S)_2$, 38894-35-8; $SnCl_4[(C_4H_9)_3P]_2$, 38966-48-2; $SnBr_4(\alpha,\alpha'\text{-}C_{10}H_8N_2)$, 16918-62-0; $Sn(C_6H_5)_2(C_9H_6NO)_2$, 19568-50-4; $SnCl_4\{[(CH_3)_2N]_2CS\}_2$, 21470-12-2; $SnBr_4[(CH_3)_2SO]_2$, 21470-09-7; $SnBr_4[(C_6H_5)_3PO]_2$, 17927-31-0; $SnCl_4(C_5H_5N)_2$, 17100-04-8; $(NH_4)_2SnCl_6$, 16960-53-5; $SnCl_4[(CH_3)_2SO]_2$, 19979-07-8; $SnCl_4\{[(CH_3)_2N]_2CO\}_2$, 12085-24-4; $SnCl_4\{[(CH_3)_2N]_3PO\}_2$, 16971-36-1; SnO_2, 18282-10-5; SnF_4, 7783-62-2; $Pt(SnCl_3)_2\{[(CH_3)_2N]_2CS\}_2$, 38882-68-7; $Pd(SnCl_3)_2\{[CH_3)_2N]_2CS\}_2$, 38960-89-3; $PtCl(SnCl_3)[(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2]$, 38894-43-8; $PdCl(SnCl_3)[(C_6H_5)_2P(CH_2)_2P(C_6H_5)_2]$, 38882-69-8; $Pt(SnCl_3)_2[(C_5H_{10}N)_3PS]_2$, 38960-50-8; $Pd(SnCl_3)_2[(C_5H_{10}N)_3PS]_2$, 38882-70-1; $PtCl(SnCl_3)[(C_6H_5)_3Sb]_2$, 38966-51-7; $PtCl(SnCl_3)[(C_6H_5)_3As]_2$, 38894-44-9; $PtCl(SnCl_3)[(C_6H_5)_3P]_2$, 32535-75-4; $Au(SnCl_3)[(C_6H_5)_3P]_3$, 38894-46-1; $Ag(SnCl_3)[(C_6H_5)_3P]_3$, 38894-47-2; $Cu(SnCl_3)[(C_6H_5)_3P]_3$, 38882-71-2; $(CH_3)_4NSnCl_3$, 14877-00-0; $KSnF_3$, 13782-23-5; $(C_6H_5)_3P$, 603-35-0; $(C_6H_5)_3PO$, 791-28-6; $[(CH_3)_2N]_2CS$, 2782-91-4; $\alpha,\alpha'\text{-}C_{10}H_8N_2$, 366-18-7; $(C_6H_5)_3PS$, 3878-45-3.

Acknowledgments. The authors wish to thank Professor C. Curran and Drs. D. Naik and M. Arnold both for the generous gift of many tin compounds and for helpful discussions. P. G. wishes to thank the National Science Foundation for support as an undergraduate research participant.

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[22] Note Added in Proof. See W. E. Swartz, Jr., P. H. Watts, Jr., E. R. Lippincott, J. C. Watts, and J. E. Huheey, *Inorg. Chem.*, 11, 2632 (1972), for the report of a similar study of tin compounds.

Contribution from the Departments of Chemistry,
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Hydrogenation of 3,4-Bis(dichloroboryl)-2,2,5,5-tetramethylhexane

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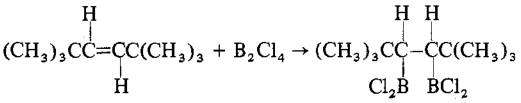
Recently Biallas[1] reported that the difunctional Lewis acid 1,2-bis(dichloroboryl)cyclohexane can react with $LiBH_4$ to give the corresponding 1,2-bis(dihydroboryl) compound. This new hydridic species was found to be relatively stable and exhibited infrared and nmr spectra consistent with those of a structure containing both terminal and bridged B-H bonds. We now wish to report the results of a study in which 3,4-bis(dichloroboryl)-2,2,5,5-tetramethylhexane (II), prepared from *trans*-2,2,5,5-tetramethyl-3-hexene and tetrachlorodiborane(4) (eq 1), is converted to the hydridic compound in a similar manner with a borohydride salt (eq 2).[2]



$$\underset{\text{I}}{(CH_3)_3C\overset{H}{C}{=}\underset{H}{C}C(CH_3)_3} + B_2Cl_4 \rightarrow \underset{\text{II}}{(CH_3)_3C\underset{Cl_2B}{\overset{H}{C}}{-}\underset{BCl_2}{\overset{H}{C}}C(CH_3)_3} \qquad (1)$$

$$\text{II} + 4LiBH_4 \xrightarrow{\text{ether}} \underset{\text{III}}{C_{10}H_{20}B_2H_4} + 4LiCl + 2B_2H_6 \qquad (2)$$

Product III is isolated at −45° by trap-to-trap fractional condensation and purified by sublimation. Compound III is a thermally stable, white, waxy solid with a molecular weight (cryoscopic) indicative of a monomeric substance. The ir spectra of II and III are summarized in the Experimental Section. Prominent features of the spectrum of III are the appearance of a sharp singlet absorption at 2495 cm^{-1} characteristic of a terminal B-H stretching vibration[3] and a strong relatively broad absorption at 1575 cm^{-1} assigned to the complex vibrational mode of the bridging B-H-B group.[4] Both of these bands in III seemingly occur at the expense of the broad BCl_2 stretching vibrations at 850-950 cm^{-1} in II.[2] The spectrum of III is essentially unchanged whether observed in solution or as a smear between KBr disks.

The boron-11 nmr spectrum of III consists of a single broad resonance at −25 ppm relative to $BF_3 \cdot Et_2O$.[5] The shape and position of the signal are independent of solvent and concentration.

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[1] M. J. Biallas, *Inorg. Chem.*, 10, 1320 (1971).

[2] A small quantity of ethyl chloride, presumably from a side reaction of diethyl ether and I, was detected in the material trapped at −196° on fractional condensation. Similar ether cleavages have been observed with other polyfunctional BCl_2 derivatives: M. Zeldin, *J. Inorg. Nucl. Chem.*, 33, 1179 (1971).

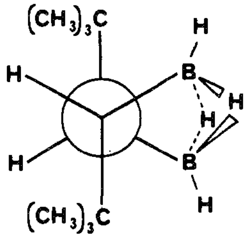
[3] It has been well documented that diborane derivatives having two terminal hydrogens have both asymmetric in-phase and symmetric out-of-phase stretches which appear as a doublet in the ir near 2500 cm^{-1}: W. J. Lehmann, C. O. Wilson, J. F. Ditter, and I. Shapiro, *Advan. Chem. Ser.*, No. 32, 139 (1961).

[4] W. J. Lehmann and I. Shapiro, *Spectrochim. Acta*, 17, 396 (1961).

[5] The boron-11 chemical shift of III is consistent with the values obtained for similar compounds: 1,2-tetramethylenediborane, −18.6 ppm; 1,2-(1′-methyltrimethylene)diborane, −18.6 ppm (H. G. Weiss, W. J. Lehmann, and I. Shapiro, *J. Amer. Chem. Soc.*, 84, 3840 (1962)); 1,2-dimethyldiborane, −20.5 ppm for cis and trans isomers (R. E. Williams, H. D. Fisher, and C. O. Wilson, *J. Phys. Chem.*, 64, 1583 (1960)).

The proton spectrum of III contains two absorptions: a relatively broad singlet at 1.2 ppm assigned to the methyl group protons and a broad multicenter envelope centered at 0.9 ppm attributed to the methine protons. An area ratio of 7:1 respectively suggests that the usually broadened hydridic protons are buried under the high-field peak.[5] Hydride exchange, boron-hydrogen coupling, and quadrupole effects are probably responsible for the lack of resolution. At low temperatures (~−5°) III precipitates before spectral changes can occur.

Since the stereochemistry of II is fixed, owing to the known stereospecific cis addition of B_2Cl_4 to olefins,[6-9] it seems reasonable to postulate a structure for III which involves intramolecularly associated dihydroboryl groups. A similar structure has been postulated by Biallas[1] for the cyclohexane derivative and is analogous to the cyclic organodiborane(6) compounds obtained by hydroboration of butadiene.[10-14]



III

Further evidence favoring a cyclic organodiborane type structure is obtained from thermolysis studies. Above 250°, III rapidly decomposes and generates diborane, hydrogen, uncharacterized white solids, and the original trans olefin, I, to the apparent exclusion of any cis isomer.[15] The yields of olefin range from 50 to 70% and identification is made by comparison of infrared spectrum, index of refraction, and glpc results with those of authentic olefins.

That the trans olefin is the only hydrocarbon isolated not only supports the diborane type structure but also suggests an unusual stereospecific degradation mechanism which may involve elimination of the elusive B_2H_4 moiety as a transient species.[16,17]

Furthermore, thermal decomposition of $C_{10}H_{20}B_2D_4$, prepared from II and $LiBD_4$, gives the trans olefin and B_2D_6. The specific distribution of deuterium in the diborane argues against dehydroboration involving hydrogen from the hydrocarbon framework.[18] It is noteworthy that II and other polyfunctional dichloroboryl compounds thermally decompose to $HBCl_2$, which presumably results from abstraction of hydrogen from the hydrocarbon framework.

The presence of other substances in the decomposition of III is a result of complex secondary reactions such as (1) disproportionation of B_2H_4 to B_2H_6 and other hydrides, (2) hydroboration of free olefin with B_2H_6 or B_2H_4,[13] and (3) reaction of B_2H_4 with B_2H_6 to form higher hydrides.[16]

Further work on other difunctional Lewis acids is currently under way and will be reported later.

Experimental Section

All reactions were carried out on a conventional high-vacuum system. Product manipulations were made either *in vacuo* or in a moisture–oxygen free environment. Molecular weights were determined cryoscopically in C_6H_6. Ir spectra were obtained on a Beckman IR-5A and a Perkin-Elmer 337. Proton and boron nmr spectra were taken on Varian A-60 and HR-60 (operating at 19.3 MHz) spectrometers, respectively. B_2Cl_4 was prepared by the method described by Wartik, *et al.*[19] *cis-* and *trans*-2,2,5,5-tetramethyl-3-hexene, prepared by the method of Puterbaugh and Newman,[20] were dried over $LiAlH_4$ and distilled twice on a Teflon spinning-band column prior to use (bp(trans) 121.3° (736 mm), bp(cis) 143.0° (740 mm); lit.[20] bp(trans) 125.0–125.7°, bp(cis) 143.0–143.6°). $LiBH_4$ and $LiBD_4$ were purchased from Metal Hydrides Co. and were used without further purification. Analyses were obtained by fuming nitric acid oxidation in a sealed glass tube containing an excess of $AgNO_3$. Carbon, as CO_2, was determined manometrically and gravimetrically; boron, as $B(OH)_3$, was titrated with NaOH to a mixed-indicator end point in the presence of mannitol; chlorine, as AgCl, was determined gravimetrically. Hydridic hydrogen analysis was carried out by hydrolysis of an organoboron hydride and measurement of the noncondensable gases manometrically.

Preparation of 3,4-Bis(dichloroboryl)-2,2,5,5-tetramethylhexane (II). B_2Cl_4 (9.93 mmol) and 2,2,5,5-tetramethyl-3-hexane (I) (10.20 mmol) were condensed into a vacuum ampoule. The mixture was stirred at room temperature for 40 hr. Unreacted materials were fractionated by pumping through traps maintained at 0, −45, and −95°. The residue, II, isolated at 0° is a colorless liquid which is purified by several fractional condensations into a 0° trap. Mass balance: recovered B_2Cl_4 (−95°), 2.79 mmol; olefin (−45°), 3.94 mmol; reaction ratio (mmol of olefin/mmol of B_2Cl_4), 1.02. *Anal.* Calcd for $C_{10}H_{20}B_2Cl_4$: B, 7.13; C, 39.5; Cl, 46.7. Found: B, 7.98; C, 40.7; Cl, 45.7. Ir (KBr smear) (cm^{-1}): 2860 (vs), 2800 (sh), 1470 (s), 1395 (m), 1385 (s), 1308 (m), 1280 (vw), 1210 (sh), 1202 (m), 1160 (m), 1100 (m, br), 1085 (w, sh), 1025 (m), 1015 (sh), 910 (sh), 880 (s, vbr), 780 (vw), 745 (w), 665 (w, br).

Preparation of 3,4-Bis(dihydroboryl)-2,2,5,5-tetramethylhexane (III). In a typical experiment, II (1.02 mmol) was added to a diethyl ether slurry of $LiBH_4$ (6.45 mmol) which was held at −196° under a dry N_2 flush. The vessel was sealed and evacuated, and the mixture was warmed to 0° and stirred for several days. Volatile materials were removed under vacuum by gentle warming of the residue and pumping the gases through traps at −45 and −196°. The latter trap contained a mixture of B_2H_6, diethyl ether, and a trace of ethyl chloride. A white, waxy solid, III, was retained in the −45° trap. The solid was purified by trap-to-trap sublimation. *Anal.* Calcd for $C_{10}H_{24}B_2$: B, 13.0; C, 72.4; H (hydridic), 2.43. Found: B, 13.8; C, 71.9; H (hydridic), 2.82. Mol wt: cryoscopic (C_6H_6), 153, 159; calcd, 165.9. Ir (KBr smear) (cm^{-1}): 2890 (vs), 2800 (sh), 2495 (s), 1575 (s, br), 1465 (s), 1390 (m), 1350 (s), 1285 (vw), 1215 (w), 1205 (m), 1153 (m), 1090 (m), 925 (vw), 845 (vw), 780 (w), 710 (vw). Boron-11 nmr (C_5H_{12}, C_6H_6, $CHCl_3$; reference $BF_3 \cdot Et_2O$): δ −25 ppm (s, broad). H nmr (C_6H_6): δ 1.2 (s, broad, CH_3), 0.9 (multiplet complex, broad, CH).

Preparation of 3,4-Bis(dideuterioboryl)-2,2,5,5-tetramethylhexane (III-*d*). The reaction of II with $LiBD_4$ was carried out in the same manner as described above. *Anal.* Calcd for $C_{10}H_{20}B_2D_4$: B, 12.7; C, 70.7; D, 4.74. Found: B, 12.3; C, 70.9; D, 4.25. Mol wt 161 (169.9).

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Controlled Thermal Decomposition of III (and III-*d*). In a typical experiment III (0.073 mmol) was slowly volatilized by warming the solid to 80°. The vapor was pumped through a hot zone (9-in. heavy-wall Pyrex tube, 25-mm o.d.) maintained at 250-300°. Condensable materials were trapped at −80 and −196° while noncondensable products were continuously removed by pumping. Unreacted III, which was retained in the −80° trap along with I, was extracted by fractionation and recycled through the hot zone. Only B_2H_6 (0.008 mmol) was obtained in the −196° trap and only I (0.037 mmol; n^{20}D 1.4112; lit.[20] n^{20}D(cis) 1.4266, n^{20}D(trans) 1.4116) was isolated at −80°. Very slightly volatile and nonvolatile white solids coated the walls of the hot zone and the regions immediately outside the hot zone.

The decomposition of III-*d* (0.403 mmol) gave I (0.203 mmol; n^{25}D 1.4119) and B_2D_6 (0.084 mmol; 94% D_2 on hydrolysis with D_2O).

Registry No. I, 692-48-8; II, 38781-17-8; III, 38832-81-4; III-*d*, 38781-19-0; B_2Cl_4, 13701-67-2; $LiBH_4$, 16949-15-8; $LiBD_4$, 15246-28-3.

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Fluoropentacarbonylrhenium(I). Preparation and Oxidation to Trifluorotricarbonylrhenium(III)

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In this work, we report preparation of fluoropentacarbonylrhenium(I) by a nonoxidative halogen-exchange reaction starting with the corresponding carbonyl chloride and also by oxidation reactions starting with the metal carbonyl. We have previously reported an oxidation of decacarbonyldirhenium(0) with rhenium hexafluoride to form trifluorotricarbonylrhenium(III),[1] and we now find that the fluoropentacarbonyl was formed during that reaction. We also report a new oxidation of the carbonyl using xenon difluoride, which gives both of the rhenium carbonyl fluorides, successively.

Experimental Section

Reactions were carried out using vacuum-line techniques and Kel-F apparatus.[2] Rhenium and tungsten hexafluorides, xenon difluoride, and chloropentacarbonylrhenium(I) were prepared by standard methods.[3-6] Hydrogen fluoride was purified by fractional distillation.[7] Decacarbonyldirhenium(0) (Alfa Inorganics) was stored in the dark and under dry argon. Six reactions were studied.

(a) Hydrogen Fluoride with Chloropentacarbonylrhenium(I). Prior to reaction, 0.15 mmol of the solid carbonyl chloride was wrapped in PTFE filter paper secured with PTFE tape, as though in a "tea bag," in order to keep the solid separate from soluble products. Reaction with about 5 ml of liquid anhydrous hydrogen fluoride at room temperature slowly formed a brown solution. After every 12 hr for 3 days the vapors above the solution were removed, and then all volatile compounds were evaporated leaving a brittle noncrystalline brown solid.

The collected vapors were condensed onto excess sodium fluoride to absorb hydrogen fluoride, and 0.14 ± 0.02 mmol of gas remained (measured tensiometrically).

(b) Rhenium Hexafluoride with Decacarbonyldirhenium(0). About 0.15 mmol of decacarbonyldirhenium(0) with about 5 ml of liquid anhydrous hydrogen fluoride was allowed to react with about 0.05 mmol of rhenium hexafluoride at room temperature. A brown solution formed rapidly, and after 10 min, the solution was poured through a PTFE filter into another Kel-F tube. The hydrogen fluoride was condensed back onto the unreacted carbonyl, and the cycle of operations was repeated as long as solid remained in the first tube. The brown product in the second tube was washed with tungsten hexafluoride to remove rhenium pentafluoride which is also formed.

(c) Xenon Difluoride and Decacarbonyldirhenium(0). An accurately weighed, 0.15-mmol sample of decacarbonyldirhenium(0) was placed in an evacuated reaction tube connected to Kel-F tubing (~1-mm bore, 12.5-cm length ≡ 1.00 ml of solution). Then 0.15 mmol of xenon difluoride dissolved in anhydrous hydrogen fluoride (0.60 ml of 0.25 *M* solution) was added. After 2-3 hr, all the solid had dissolved in the hydrogen fluoride to form a brown solution, which was evaporated to leave a brown solid.

(d) Xenon Difluoride with the Soluble Brown Product. It was assumed that the soluble brown products from the "tea bag" reaction of (a) and the oxidation reaction of (c) were fluoropentacarbonylrhenium(I), and accurately weighed 0.3-mmol samples were oxidized with 0.3-mmol amounts of xenon difluoride, as above. The solid dissolved immediately forming a brown solution, gas was evolved, rapidly at first and then slowly for 20-30 min, and a brown solid slowly precipitated leaving a pale brown solution.

(e) Same Reaction as in (d) except That More Xenon Difluoride Was Added. The brown solid was rapidly consumed and a green solution remained.

(f) Boron Trichloride with the Soluble Brown Product. A sample of the brown product of the reaction in (a) was allowed to react with liquid boron trichloride at room temperature for about 30 min. The mixture was evaporated and then heated to 50° to drive off all the boron trichloride. The residue was a white solid.

Results and Discussion

The results of the above six reactions, which are discussed below, are shown in Table I.

The brown product of the "tea bag" reaction in (a), between hydrogen fluoride and chloropentacarbonylrhenium(I), was analyzed. It was readily oxidized by aqueous alkaline peroxide to form perrhenate and fluoride which can be both determined.[1] *Anal.* Calcd for $Re(CO)_5F$: Re, 53.9; F, 5.5. Found: Re, 56; F, 5.8. Confirmation of this formulation was obtained as follows.

The ir spectra in Figure 1 and the X-ray powder patterns in Tables II and III are shown for both the white reactant, chloropentacarbonylrhenium(I), and the brown product, fluoropentacarbonylrhenium(I).

The similar patterns in the carbonyl regions of the ir spectra are consistent with similar symmetries for the five carbonyls in both compounds. The bands at 350 cm^{-1} are due to Re-C modes, and the band at 294 cm^{-1} is due to Re-Cl modes.[8] The new band at 475 cm^{-1} is therefore due to Re-F modes. The reaction in (f), between boron trichloride and fluoropentacarbonylrhenium(I), was used because boron trichloride has been shown to be an effective chlorinating reagent with fluorides.[4] The result of this reaction confirms the assignment of the 475-cm^{-1} band because the white product so formed had an ir spectrum identical with the original carbonyl chloride starting material. The poorly defined X-ray powder pattern (Table II) is consistent with the noncrystalline appearance of the solid. These ir spectra and X-ray powder patterns were found to be particularly useful in checking the complete-

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