Table III. Bond Angles (deg) for $CH_3B(\mu\text{-}pz)_2(\mu\text{-}SS)BCH_3$ (**4**)

B-S-S*	100.8 (1)	B-C6-H6a	109.1 (18)
S-B-C6	111.3 (2)	B-C6-H6b	110.9 (18)
S-B-N1*	107.4 (2)	B-C6-H6c	113.4 (19)
S-B-N1	107.6 (2)	H6a-C6-H6b	119.9 (26)
N1-B-N1*	103.7 (2)	H6a-C6-H6c	98 (27)
B-N1-N2	118.2 (2)	H6b-C6-H6c	104.5 (26)
B*-N2-N1	118.3 (2)	C3-C4-C5	105.9 (3)
N1-B-C6	113.4 (2)	N2-C3-C4	108.7 (2)
C6-B-N1*	112.9 (2)	N1-C5-H5	122.5 (18)
B-N1-C5	133.0 (2)	N2-C3-H3	122.3 (18)
B*-N2-C3	133.1 (2)	C5-C4-H4	128.3 (22)
N2-N1-C5	108.7 (2)	C4-C5-H5	129.1 (18)
N1-N2-C3	108.4 (2)	C4-C3-H3	128.9 (18)
N1-C5-C4	108.2 (2)	C3-C4-H4	125.4 (22)

lower yield and formation of the triply bridged pyrazabole $CH_3B(\mu\text{-}pz)_2(\mu\text{-}OBCH_3eo)BCH_3$ as well as hydrogen sulfide and elemental sulfur as major byproducts.

The $CH_3B(\mu\text{-}pz)_2(\mu\text{-}SS)BCH_3$ molecule has crystallographic C_2 symmetry, but even in the solid state it is rather close to point group C_{2v}, which corresponds to the (solution) NMR data. An ORTEP plot of the species is given in Figure 1, bond distances are given in Table II, and bond angles are listed in Table III.

The bridging S_2 group imposes the boat conformation for the B_2N_4 ring of the molecule, which is common for most pyrazaboles.[5] The bond distances within the pyrazabole skeleton parallel those found for a series of other pyrazaboles, but the N1-N2 distance is one of the shortest measured in pyrazaboles. The two B-N distances as well as the C-N and C-C distances are essentially equal. The S-S bond (2.105 (1) Å) is a typical single bond but is noticeably longer than in S_8 or disulfanes; it is also longer than that found in $CH_3B(\mu\text{-}NCH_3)(\mu\text{-}SS)BCH_3$, containing trigonal boron.[6] The B-S bond distances in trigonal boron derivatives range from 1.79 to 1.84 Å,[7] but longer distances have been observed for the 1:1 molar adduct of 4,6-dibromo-1,2,3,5,4,6-tetrathiadiborolane with 1,3,2-trimethyl-1,3,2-diazaborolidine, in which all boron atoms are four-coordinate.[8] The B-S distances for the B-S-S-S-B unit of the latter adduct are 1.910 (12) and 1.930 (12) Å, respectively, comparable to that found in **4** (1.940 (3) Å).

The N-B-N angle is, however, smaller by about 2° as compared to those of other pyrazaboles; only that of $C_2H_5B(\mu\text{-}pz)_2(\mu\text{-}OBC_2H_5O)BC_2H_5$ is even smaller.[6] This is undoubtedly the consequence of the third bridge between the two boron atoms, especially since the N-B-S angle (and the N-B-O angle in $C_2H_5B(\mu\text{-}pz)_2B(\mu\text{-}OBC_2H_5O)BC_2H_5$) is also smaller than comparable data in normal pyrazaboles, i.e., those where the two B atoms are linked only by the two pz groups. It is noteworthy that the dihedral angle B-S-S-B is 0°. The folding angle θ_1 (between a BN_2 plane and the N_4 plane) is 39.9°, the largest one measured for any pyrazabole, and the roof angle θ_2 formed between the two pz rings in a butterfly arrangement is also extremely high (46.2°).[5] Thus, the additional S-S bridge between the two B atoms enhances the boat conformation of the B_2N_4 ring.

The formation of $CH_3B(\mu\text{-}pz)_2(\mu\text{-}OBCH_3O)BCH_3$ as byproduct in the reaction according to eq 1 in the presence of moisture is probably due to the hydrolytic sensitivity of the starting dithiaazadiborolidine, since $CH_3B(\mu\text{-}pz)_2(\mu\text{-}SS)BCH_3$ is not moisture-sensitive. The former triply bridged pyrazabole is more readily accessible by the conventional[2] interaction of pyrazole with $(-BCH_3O-)_3$ and also by the hydrolysis of 4,8-dibromo-4,8-dimethylpyrazabole.[4]

The present work shows that additional triply bridged neutral pyrazaboles should be available by reaction of various boron heterocycles with pyrazole. It remains to be seen whether or not the third bridge can also be a single atom, but species containing a N-N unit as the third bridge between the two boron atoms of a pyrazabole skeleton are likely to exist.

Acknowledgment. We gratefully acknowledge a gift of 2-phenyl-3,5-dimethyl-1,2,4,3,5-dithiaazadiborolidine from Professor A. Meller, Universität Göttingen, West Germany. This work was supported by the Office of Naval Research (K.N.) and the Fonds der Chemischen Industrie (H.N.).

Registry No. **1** (coordination compound entry), 112840-86-5; **1** (borane entry), 112863-45-3; **3**, 63261-96-1; **4** (coordination compound entry), 112840-87-6; **4** (borane entry), 112840-89-8; Hpz, 288-13-1; $Br(CH_3)B(\mu\text{-}Pz)_2BBr(CH_3)$, 112840-88-7.

Supplementary Material Available: A listing of anisotropic thermal parameters for non-hydrogen atoms for **4** (1 page); a structure factor table (5 pages) for **4**. Ordering information is given on any current masthead page.

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Mechanism of Catalytic Isotope Exchange in Alcohols Promoted by Early-Transition-Metal Alkoxides. Comment on the Paper by Nugent and Zubyk[1]

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A recent paper by Nugent and Zubyk[1] concerning the isotopic H-D exchange in alcohols catalyzed by early-transition-metal alkoxides appears relevant to the mechanism of higher alcohol synthesis from syngas under metal oxide catalysis.[2] Nugent and Zubyk[1] reported separate α- and β-exchange processes for scrambling H-D in alcohols in the presence of various $M_x(OEt)_y$ (M = Zr, $x = 1$, $y = 4$; M = Nb, Ta, $x = 2$, $y = 10$) complexes at temperatures from 180 to 220 °C. The β-hydrogens exchanged rapidly into the hydroxyl position, while the α-hydrogens underwent a separate scrambling process; the two processes are summarized schematically as

α-exchange

$$CH_3CH_2OH + CH_3CD_2OH \leftrightarrow CH_3CHDOH$$

β-exchange

$$CH_3CH_2OD \leftrightarrow CH_{3-n}D_nCH_2OH(D)$$

Similar rates were observed for the two processes and "the same alcohol which has undergone α-exchange has also undergone (multiple) β-exchange". Racemization of 2-butanol and epimerization of *tert*-butylcyclohexanol accompanied their exchange reactions. This evidence was interpreted to indicate a common intermediate, proposed to be an organic carbonyl compound, was responsible for both exchange processes. In a previous paper,[3] amines were shown to catalyze the β-exchange process, although the authors did not elaborate on this point.

The mechanism proposed for the conversion of syngas into higher alcohols over metal oxide catalysts involves a stepwise reduction of coordinated CO to formaldehyde, which is reduced further to methanol or undergoes CO-insertion to form higher products.[2] An intermediate in the reaction path leading to higher alcohols is an η^3-enolate. Stability differences of various η^3-enolate species are thought to contribute to the branching observed in the higher alcohol products. An important competing reaction is

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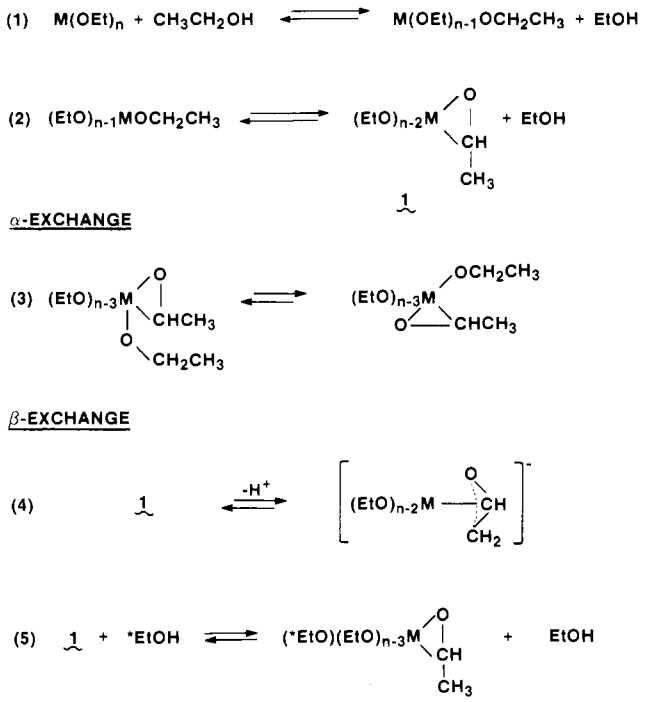
Scheme I



proposed to be alkylation of the surface η^3-enolate by an alkyl group derived from a surface alkoxide to form branched products; this reaction also provides a route for deviations from Schulz–Flory distributions.

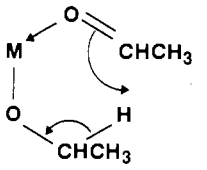
Extrapolation from the homogeneous isotope exchange to heterogeneous alcohol synthesis catalysts is justified by reports of β-exchange catalyzed by ZrO_2 and ThO_2.[4] A mechanism for the isotope exchange observed by Nugent and Zubyk can be proposed which utilizes the same concepts introduced for alcohol synthesis.[2] The mechanism is summarized in Scheme I. Alkoxide groups from the metal alkoxide complex exchange rapidly with alcohol from the solution as in eq 1.[3] The alkoxide complex can lose alcohol in what amounts to reductive elimination to form the aldehyde (or ketone) complex, **1**. Complex **1** can be considered to be the catalyst for α- and β-hydrogen exchange; its formation by eq 2 is slow compared to all other reactions.

Exchange of the α-hydrogens is proposed to occur within the catalytic complex **1** as in eq 3. The β-hydrogens are proposed to exchange with solvent hydroxyl hydrogens by loss of a proton to form an η^3-enolate complex (eq 4). Protonation regenerates **1**. One other step is needed to make the system catalytic. This step is exchange of alkoxide groups of **1** with free alcohol molecules (eq 5). The relative ordering of rates of these steps is proposed to be $4 \geq 3 > 5 \simeq 1 \gg 2$.

The α-exchange of eq 3 may proceed via a concerted step that transfers a hydrogen from the α-position of a coordinated alkoxide to the α-position of the coordinated aldehyde (or ketone). A six-member-ring transition state can be drawn for this step, which shows that the hydrogen transfer is equivalent to that involved in the Meerwein–Ponndorf–Verley reduction of organic carbonyl compounds[5] (Scheme II). This step involves only α-hydrogens (after the initial formation of **1**) and, coupled with rapid alkoxide–alcohol exchange (eq 5), provides a mechanism that can scramble all of the α-hydrogens through only a single metal complex of an organic carbonyl. Rotation of the complexed organic carbonyl about the C–O bond leads to racemization of alcohols optically active at the hydroxyl carbon.

An η^3-enolate complex can be generated from the aldehyde (or ketone) complex by a second elimination step in which an alkoxide

Scheme II



(or a base) deprotonates the aldehydic ligand (eq 4). This step exchanges β-hydrogens with hydroxyl hydrogens of the free alcohol in a process separate from the α-exchange step. No *uncomplexed* organic carbonyl needs to be invoked as an intermediate. In fact, *complete exchange of all of the α- and β-hydrogens could occur through a single metal complex of an organic carbonyl if exchange of bound alkoxide groups with the free alcohol is fast for complex* **1**. Nugent and Zubyk's observation[1] of multiply β-exchanged alcohols as initial products implies that the β-exchange is faster than the α-exchange.

There are a number of features of this mechanism that are confirmed by the observations of Nugent and Zubyk.[1] (1) The two exchange processes pass through a common organic carbonyl intermediate. (2) The α- and β-exchange processes scramble separate pools of hydrogens. (3) Multiple β-exchange can occur in the same alcohol molecule that has undergone α-exchange. (4) The base catalysis observed for the β-exchange can be explained as a result of enhanced η^3-enolate formation by deprotonation of the bound organic carbonyl intermediate. (5) Added aldehyde is not expected to greatly alter the isotope-exchange rate, since an uncomplexed organic carbonyl is not proposed to be an intermediate. (6) Racemization or epimerization of alcohols is predicted to accompany the exchange processes.

The indirect evidence for the existence of an η^3-enolate intermediate offered by the present interpretation of Nugent and Zubyk's observations is augmented by recent reports from other laboratories. Doney[6] reported the formation of an η^3-enolate upon photolytic decarbonylation of a tungsten η^1-enolate complex ($CpW(CO)_3C$-η^1-CH_2COR, R = CH_3, OEt). Their η^3-complex could be alkylated by reaction with benzaldehyde to form an aldol condensation product or returned to the η^1 coordination mode by ligand addition. The X-ray structure of one of these η^3-enolate complexes has recently been solved[7] for R = NEt_2.

Further structural confirmation of the η^3-enolate mode of coordination was provided in a dimeric Ru complex by Holmgren[8] ($Ru_2(CO)_5(\mu$-η^4-$CH_2C(O)CH_2)$(dppm)). This complex was synthesized from methylene and ketene moieties and, due to the nature of the metal involved (Ru), would not be expected to compare in *reactivity* with early-transition-metal η^3-enolates. Nonetheless, it provides structural evidence for the η^3-enolate coordination mode proposed for alcohol synthesis[2] and by Prince and Raspin[9] to explain the isotope distribution in the decarbonylation of labeled aldehydes catalyzed by Ru complexes.

A bridging η^3-enolate structure has been reported also for the Reformatsky reagent,[10] [$Zn_2(\mu$-η^3-$CH_2C(O)O$-*t*-Bu-*O,C,C'*)$_2Br_2(THF)_2$]. Other zinc enolates have been shown to exhibit the reactivity expected for O-metalated as well as C-metalated enolates.[11] The η^3-enolate fragment has been characterized also as part of chelating ligands.[12]

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The common intermediacy of η^3-enolates in these seemingly disparate systems may help in unifying our understanding of their sometimes divergent reactivity patterns.

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Formation and Reaction Chemistry of Trimethylamine-Trimethylphosphine-Diborane(4)

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We reported in an earlier communication[1] to this journal that trimethylamine-trimethylphosphine-diborane(4), $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$, was formed by a ligand displacement reaction of bis(trimethylamine)-diborane(4), $B_2H_4{\cdot}2N(CH_3)_3$, with trimethylphosphine (see eq 1). The product was the first repre-

$$B_2H_4{\cdot}2N(CH_3)_3 + P(CH_3)_3 \rightarrow B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3 + N(CH_3)_3 \quad (1)$$

sentative of a previously unknown mixed-ligand adduct of diborane(4). Displacement of the second trimethylamine proceeded very slowly.[1]

The mixed-ligand adduct, $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$, can now be prepared in pure form by the reaction of $B_3H_7{\cdot}P(CH_3)_3$ with trimethylamine. Furthermore, this finding has provided a new insight into the reaction mechanism of base cleavage of triborane(7) adducts. In this paper, we describe the characterization and reaction chemistry of the mixed adduct of diborane(4).

Results

A. Cleavage of $P(CH_3)_3$ and $N(CH_3)_3$ Adducts of B_3H_7. (a) Reaction of $B_3H_7{\cdot}P(CH_3)_3$ with $N(CH_3)_3$. Formation of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$. The reaction of $B_3H_7{\cdot}P(CH_3)_3$ with 2 molar equiv of $N(CH_3)_3$ in dichloromethane at room temperature proceeds according to eq 2. The mixed-ligand adduct of B_2H_4 can

$$B_3H_7{\cdot}P(CH_3)_3 + 2N(CH_3)_3 \rightarrow B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3 + BH_3{\cdot}N(CH_3)_3 \quad (2)$$

be separated from $BH_3{\cdot}N(CH_3)_3$ as a colorless solid by fractional sublimation at room temperature. The compound is stable in the absence of air. It slowly decomposes in solution at room temperature.

NMR Spectra of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$. The $^{11}B\{^1H\}$ NMR spectrum of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$ in Figure 1 shows a broad signal at −2.7 ppm due to the amine-attached boron atom (B_N) and another at −36.4 ppm due to the phosphine-attached boron atom (B_P). The ^1H-spin-coupled ^{11}B signals are broader but do not have any fine structure. These shift values are compared with the values of $B_2H_4{\cdot}2N(CH_3)_3$ and $B_2H_4{\cdot}2P(CH_3)_3$ in Table Ia. The $^{31}P\{^1H\}$ NMR spectrum of the compound is a broad, *partially collapsed* 1:1:1:1 quartet centered at 3.6 ppm with a J_{PB} value of ca. 50 Hz. The apparent absence of a doublet feature (B-P coupling) on the B_P resonance signal (Figure 1) is attributable

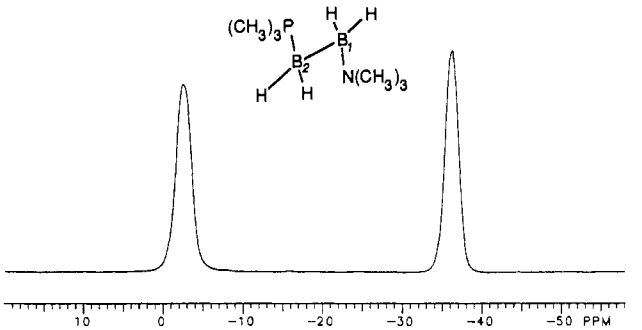

Figure 1. $^{11}B\{^1H\}$ NMR (96.2 MHz) spectrum of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$ at +20 °C in CH_2Cl_2.

Table I. ^{11}B NMR Shift Data (ppm)

a. $B_2H_4{\cdot}L{\cdot}L'$

	B-N	B-P
$B_2H_4{\cdot}2P(CH_3)_3$[a]		−37.5
$B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$	**−2.7**	**−36.4**
$B_2H_4{\cdot}2N(CH_3)_3$[b]	−3.5	

b. $B_3H_6{\cdot}L{\cdot}L'^+$

	B-N	B-P	BH_2
$B_3H_6{\cdot}2P(CH_3)_3^+$[c]		−39.0	−10.5
$B_3H_6{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3^+$	**−12.4**	**−41.3**	**−10.2**
$B_3H_6{\cdot}2N(CH_3)_3^+$[b]	−15.8		−9.7

[a] References 14 and 15. [b] Reference 1. [c] Reference 3.

to this small J_{BP} value and to the broadness of the signal. The $^1H\{^{11}B\}$ NMR spectrum shows a singlet at 2.52 ppm (intensity 9, amine CH_3 protons), a quintetlike signal at 1.75 ppm (intensity 2, protons on B_N), a sharp doublet at 1.14 ppm (^2J = 9.0 Hz, intensity 9, phosphine CH_3 protons), and a doublet of triplets at 0.03 ppm ($^2J_{HBP}$ = 21 Hz, $^3J_{HBBH}$ = 5.0 Hz, intensity 2, protons on B_P). The quintetlike signal of the protons attached to B_N is thought to be due to spin-spin couplings to both the B_P protons and the phosphorus. On the basis of this assumption, a value of 9.7 Hz is estimated for $^3J_{HBBP}$.

(b) Reactions of $B_3H_7{\cdot}N(CH_3)_3$ and $B_3H_7{\cdot}P(CH_3)_3$ with $P(CH_3)_3$. The trimethylamine adduct of B_3H_7 slowly reacts with $P(CH_3)_3$ at room temperature in dichloromethane. The major products are $BH_3{\cdot}N(CH_3)_3$ and $B_2H_4{\cdot}2P(CH_3)_3$, and $BH_3{\cdot}P(CH_3)_3$ and $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$ are detected in minute quantities. Thus, the appropriate equation for the reaction is

$$B_3H_7{\cdot}N(CH_3)_3 + 2P(CH_3)_3 \rightarrow B_2H_4{\cdot}2P(CH_3)_3 + BH_3{\cdot}N(CH_3)_3 \quad (3)$$

The reaction of $B_3H_7{\cdot}P(CH_3)_3$ with $P(CH_3)_3$ proceeds similarly:[2]

$$B_3H_7{\cdot}P(CH_3)_3 + 2P(CH_3)_3 \rightarrow B_2H_4{\cdot}2P(CH_3)_3 + BH_3{\cdot}P(CH_3)_3 \quad (4)$$

B. Reactions of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$. (a) With Hydrogen Chloride. The mixed-base adduct, $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$, reacts with anhydrous HCl at −80 °C in a CH_2Cl_2 solution according to eq 5. Apparently, an alternative mode of cleavage is unfa-

$$B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3 + HCl \rightarrow BH_3{\cdot}N(CH_3)_3 + BH_2Cl{\cdot}P(CH_3)_3 \quad (5)$$

vorable; $BH_3{\cdot}P(CH_3)_3$ and $BH_2Cl{\cdot}N(CH_3)_3$ are produced in trace quantities.

(b) With Tetraborane(10). Treatment of $B_2H_4{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3$ with B_4H_{10} results in the formation of a new triboron complex cation, (trimethylamine)(trimethylphosphine)hexahydrotriboron(1+) [$B_3H_6{\cdot}N(CH_3)_3{\cdot}P(CH_3)_3^+$] (see eq 6). This reaction is analogous to that of $B_2H_4{\cdot}2P(CH_3)_3$ (eq 7) or $B_2H_4{\cdot}2N(CH_3)_3$ with B_4H_{10}.[1,3]

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