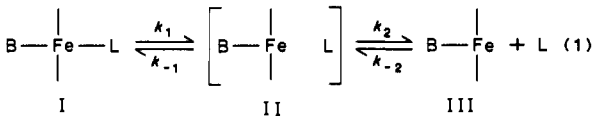
Table I. Rate Constants for Geminate Recombination and Diffusion in the Binding of Isocyanides and 1-Methylimidazole to the 1-Methylimidazole Complex of Protoheme Dimethyl Ester[c]

ligand	solvent	[L] (mM)	Γ^d	$k_{obsd} \times 10^{-10}$ (s^{-1})	k_1 (s^{-1})	$k_{-1} \times 10^{-10}$ (s^{-1})	$k_2 \times 10^{-10}$ (s^{-1})	$k_{-2} \times 10^{-8}$ (M^{-1} s^{-1})
MeNC	*a*	4.4	0.34 ± 0.03	11.3 ± 0.4	2.8	7.5	3.8	3.0
MeNC	*b*	4.0	0.45 ± 0.05	12.4 ± 0.7		6.8	5.6	
t-BuNC	*a*	1.8	0.36 ± 0.03	11.8 ± 0.4	2.3	7.6	4.2	2.2
t-BuNC	*b*	1.8	0.48 ± 0.03	12.0 ± 0.4		6.2	5.8	
TMIC	*a*	4.5	0.34 ± 0.03	10.3 ± 0.4	0.07	6.5	3.5	2.6
1-MeIm	*a*	*e*	0.20 ± 0.03	10.5 ± 0.5	7.3×10^3	8.4	2.2	2.3

[a] Solvent: toluene/1-MeIm, 80:20 by volume. [b] Solvent: DMF/1-MeIm, 80:20 by volume. [c] Data for overall equilibria and kinetics from ref 9–12 are used to calculate k_1 and k_{-2}. [d] $\Gamma = \Delta A(t = \infty)/\Delta A(t = 0) = k_2/(k_{-1} + k_2)$. [e] The solvent is 2.5 M in 1-MeIm.

and 2, respectively. In the figures, the negative absorbance change, or bleaching, at 428 and 426 nm is due to the disappearance of the ground-state six-coordinate species as the ligands are photolzyed from the iron. The positive absorbance at about 440 nm is attributed to the absorption by the five-coordinate deligated species. As the relative delay between the pump and probe pulses is increased, the absolute magnitude of these two bands will decrease as rebinding of the geminate pair occurs. The spectral changes correspond closely with the titration spectra observed in static systems involving the species I and III.



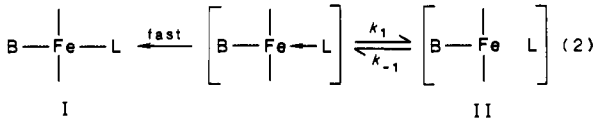
I II III

The observed relaxation rate constant, k_{obsd}, is the sum of k_{-1} (bond formation) and k_2 (diffusion). This, along with the fraction of absorption recovery at long time (yield for dissociation), affords k_{-1} and k_2. These, with published overall rate and equilibrium constants,[9-12] reveal all the rate constants in eq 1 for all of the ligands studied; see Table I. Figure 3 shows the first-order plot of the time dependence at λ_{max} (443 nm) of the five-coordinate species formed by the photolysis of bis(1-MeIm)iron(II) protoheme dimethyl ester.

In heme compounds, relaxation to the five-coordinate, high-spin "deoxy" state with the iron out-of-plane has been inferred from subpicosecond transient absorption[13] and Raman measurements[14] to occur in less than 1 ps. Therefore, we assume that the recombination rate k_{-1} characterizes a reaction between the singlet ligand and the high-spin iron(II) porphyrin. Since the bound species is diamagnetic, the spin change that accompanies recombination occurs with a rate in excess of 10^{10} s^{-1}.

The results we observe with all four ligands contrast with the behavior of CO, another diamagnetic ligand. We do not find any concentration independent recombination of carbon monoxide over the range from 10^5 to 10^{11} s^{-1}. Since we could have detected 10% geminate recombination of CO, we can use eq 1, with k_2 and k_{-2} taken to be the same as for methyl isocyanide, to estimate a maximum possible value k_{-1} of 10^9 s^{-1} for the rebinding of CO to model heme compounds. The recombination of CO is almost two orders of magnitude slower than other diamagnetic ligands.

There are several consequences of these results. First, the low quantum yields reported for transition-metal complex photodissociation should be reinvestigated for the possible occurrence of fast geminate rebinding. Second, steric effects in the binding of hemes, generally attributed to the bond making step, must be reevaluated. Finally, the differences between the rebinding of CO and the other diamagnetic ligands requires some rationalization. The alkyl isocyanides react at least 70 times faster with iron(II) porphyrin than does isoelectronic carbon monoxide. Both reactions require the conversion of high-spin iron(II) to the diamagnetic state. Since the diamagnetic isocyanides react just as rapidly as paramagnetic NO and O_2, we must look for some mechanism for spin change that is not available for CO but is accessible to the other ligands. One possibility is that the bases form transient high spin iron(II) complexes which rapidly relax to the diamagnetic complexes, eq 2. Such a reaction would be greatly accelerated by an increase in the basicity of L. This hypothesis prompted the study of the 1-MeIm reaction. The results in Table I are consistent with the mechanism.



I II

These observations probably apply to a wide variety of transition-metal ligation reactions. Both the generality and the basicity dependence of these reactions are under further study.

Acknowledgment. This work was supported by NIH Grants HL13581 and RR02353-01 and NSF Grant CHE-8409642.

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Synthesis and Properties of a Series of Ruthenium Dihydrogen Complexes

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Since the initial discovery by Kubas,[1] dihydrogen complexes of the transition metals have been the subject of considerable interest since they may serve as models for the very important process of oxidative addition of dihydrogen.[2] In this context, Kubas has recently reported a tungsten complex in which a dihydride is in reversible equilibrium with a dihydrogen complex.[3] We now report further examples of such equilibria in cationic complexes of ruthenium.

Protonation (CH_2Cl_2 solutions) of the hydrides $CpRu(CO)(PR_3)H$((PR_3 = PPh_3 (**1a**), PMe_3 (**1b**), PMe_2Ph (**1c**), PCy_3 (**1d**))[4]

(1) Kubas, G. J.; Ryan, R. R.; Swanson, B. I.; Vergamini, P. J.; Wasserman, H. J. *J. Am. Chem. Soc.* **1984**, *106*, 451.
(2) (a) Morris, R. H.; Sawyer, J. F.; Shiralian, M.; Zubkowski, J. D. *J. Am. Chem. Soc.* **1985**, *107*, 5581. (b) Crabtree, R. H.; Lavin, M. *J. Chem. Soc. Chem. Commun.* **1985**, 794.
(3) (a) Kubas, G. J.; Ryan, R. R.; Wrobleski, D. A. *J. Am. Chem. Soc.* **1986**, *108*, 1339. (b) Kubas, G. J.; Unkefer, C. J.; Swanson, B. I.; Fukushima, E. *J. Am. Chem. Soc.* **1986**, *108*, 7000.
(4) The hydride, $CpRu(CO)(PPh_3)H$, has been previously reported as the product of the reaction of $CpRu(CO)_2H$ and PPh_3.[5] Compounds **1b–d** were prepared similarly.

0002-7863/87/1509-5865$01.50/0

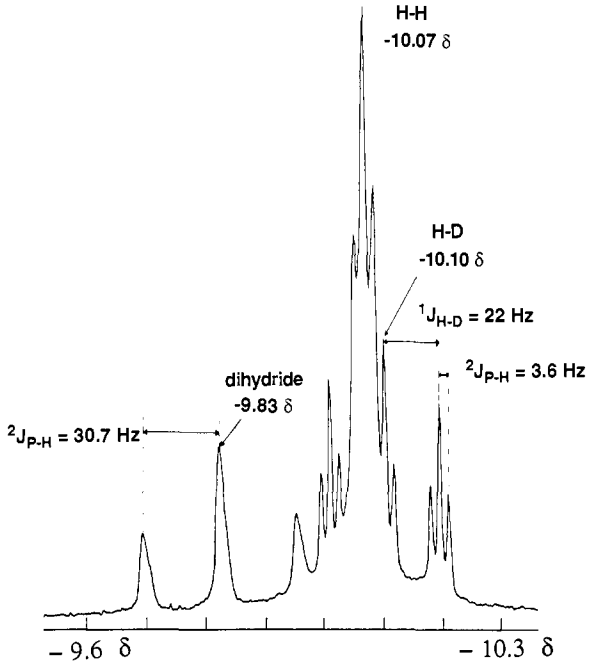

Figure 1. ^{1}H NMR spectrum (308 K, 250 MHz, CD_2Cl_2) of a mixture of H–H and H–D isotopomers of **4**. **4a** exhibits a triplet at δ −10.07 ppm for the H–H isotopomer and a triplet of triplets centered at δ −10.10 ppm due to the H–D isotopomer. Both show $^2J_{H-P}$ = 3.6 Hz. There is no resolvable H–D coupling in **4b**.

at −78 °C followed by addition of Et_2O affords the cationic dihydrogen complexes $[CpRu(CO)(PR_3)(\eta^2\text{-}H_2)]BF_4$ (**2a–d**) as colorless microcrystalline precipitates.[6] Characterization as dihydrogen adducts is based primarily on spectroscopic data. The ^{1}H NMR spectra (CD_2Cl_2) of **2a–d** all exhibit broad resonances in the hydride region.[8] A T_1 measurement (250 MHz) for **2d** gave a value of 4 ms at 205 K for the dihydrogen resonance.[9] When the preparative reaction was carried out with $DBF_4{\cdot}Et_2O$, the H–D complexes were obtained, with $^1J_{H-D}$ of 28–30 Hz, values indicative of dihydrogen complexes.[10] In no case was coupling to ^{31}P observed, even though line widths of the hydride resonances in the HD isotopomer were only 2–3 Hz.

In the case of **2d**, an additional Cp resonance and an additional hydride resonance at δ −7.42 ppm (doublet; $^2J_{H-P}$ = 20 Hz) were also observed. Integration indicated that the minor species accounts for 2–3% of the total. The ^{31}P NMR spectrum (with selective decoupling of the cyclohexyl protons) exhibited a singlet at δ 69.0 ppm and a triplet (J = 20 Hz) at δ 76.8 ppm (relative to 85% H_3PO_4). These observations are consistent with the presence of a formally seven-coordinate dihydride $[CpRu(CO)(PCy_3)H_2]BF_4$ in addition to the dihydrogen complex.[11] Spin saturation transfer experiments at 298 K showed no evidence for exchange between the dihydride species and the dihydrogen complex.

Protonation of CpRu(dmpe)H (**3**)[12] (dmpe = 1,2-bis(dimethylphosphino)ethane) under the same conditions affords a cationic complex **4** of similar physical properties. The ^{1}H NMR spectrum of **4** (308 K) reveals that a dihydrogen complex **4a** and a dihydride form **4b** are both present. (The ratio of **4a**:**4b** is 6.0:1). The hydride resonance for **4b** is a symmetric triplet ($^2J_{H-P}$ = 31 Hz) at δ −9.83 ppm while the dihydrogen complex **4a** gives a slightly broadened triplet at δ −10.07 ppm.[13] Figure 1 shows the hydride region of the ^{1}H NMR of partially deuteriated **4**, clearly showing the well-resolved H_2–P coupling in **4**. ($^2J_{H_2-P}$ = 3.6 Hz). This is the first example of resolved coupling of this type in a dihydrogen complex.[14] The H–D isotopomer of **4a** exhibits a remarkably low value for $^1J_{H-D}$ of 22 Hz. The very low $^1J_{H-D}$ value and substantial $^2J_{H_2-P}$ indicates a strong interaction between the dihydrogen ligand and the metal center, along with considerable lengthening of the H–H bond. Consistent with these observations, we find that **4** is extremely robust. Solid **4** does not appreciably lose H_2 in vacuo and is stable at room temperature in acetonitrile.[16]

Although **4a** and **4b** give distinct resonances in the ^{1}H NMR at room temperature, equilibration of **4a** and **4b** was detected by spin saturation transfer at 298 K (CD_3CN). Saturation of the cyclopentadienyl resonance due to **4a** led to an intensity decrease in the resonance due to **4b**. The rate constant for exchange was calculated to be 9.0×10^{-3} s^{-1}.[18] This corresponds to a free energy of activation of 20.4 kcal/mol. This result is in sharp contrast to the observations of Kubas on $W(CO)_3(P(i\text{-}Pr)_3)_2(\eta\text{-}H_2)$ and $W(CO)_3(P(i\text{-}Pr)_3)_2H_2$ where the dihydrogen and the dihydride form exchange much more rapidly.[20] Although the low value for $^1J_{H-D}$ in **4a** presumably indicates a substantial weakening and lengthening of the H–H bond, the rate of oxidative addition to give the dihydride form **4b** is surprisingly low. This low rate of **4a**/**4b** interconversion was further verified by preparation of a sample of **4** in cold CD_3CN. Immediate observation of the ^{1}H NMR spectrum at −30 °C showed *only* **4a** to be present. At −10 °C, formation of the equilibrium mixture of **4a** and **4b** was observed with $t_{1/2}$ of ca. 10 min. This observation reinforces the conclusion of Kubas[3b] that solid state and solution structures may be quite different.

The dihydrogen ligand in **4a** is highly activated toward heterolytic cleavage. Deprotonation is effected by mild bases such as Et_3N. Quantitative measurements indicate that the pK_a of **4a** in CH_3CN is 17.6.[21] Rapid proton exchange was observed be-

(5) Humphries, A. P.; Knox, S. A. R. *J. Chem. Soc., Dalton Trans.* **1975**, 1710.

(6) Satisfactory elemental analyses (C, H) were obtained for **2a–d**. A similar complex, $[CpRu(PPh_3)(t\text{-}BuNC)(H_2)]^+$, has been reported.[7]

(7) Conroy-Lewis, F. M.; Simpson, S. J. *J. Chem. Soc., Chem. Commun.* **1986**, 506.

(8) **2d**: ^{1}H NMR (CD_2Cl_2, 250 MHz) δ 5.62 (s, Cp), 2.15–1.46 (m), −7.90 (br s, η^2-H_2, $\nu_{1/2}$ = 14 Hz).

(9) The T_1 criterion for identifying dihydrogen complexes has been outlined by Crabtree and co-workers: (a) Crabtree, R. H.; Lavin, M.; Bonneviot, L. *J. Am. Chem. Soc.* **1986**, *108*, 4032. (b) Crabtree, R. H.; Hamilton, D. G. *J. Am. Chem. Soc.* **1986**, *108*, 3124.

(10) **2d**: $^1J_{HD}$ = 28.5 Hz (CD_2Cl_2). Previously reported HD complexes have $^1J_{HD}$ values of 28.6–34 Hz. (cf. ref 2, 3b, and 7).

(11) **2d** dihydride: ^{1}H NMR (CD_2Cl_2, 250 MHz) δ 5.74 (s, Cp), −7.07 (d, $^2J_{PH}$ = 20 Hz). The observation of a triplet due to the minor species in the ^{31}P NMR spectrum excludes the possibility of a cyclometallated species.

(12) Synthesized by a modification of the ref 5 procedure: 1.5 equiv of dmpe was added to a yellow solution of $CpRu(CO)_2H$ and refluxed for 30 min. A yellowish precipitate formed. The supernatant was removed by cannula filtration, and the solvent was removed on a rotary evaporator. The yellow material was sublimed onto a water cooled probe (0.05 Torr, 60–70 °C). Compound **3** was collected as air-sensitive, pale yellow needles (30% yield).

(13) Additional data: **4a**, ^{1}H NMR (CD_3CN, 250 MHz) 5.17 (s, Cp), 1.72–1.66 (m), −10.15 (η^2-H_2, $^2J_{H2\text{-}P}$ = 3.8 Hz); **4b**, 5.34 (s, Cp), −9.88 (t, $^2J_{H-P}$ = 30.5 Hz); **4a**, ^{31}P (CD_2Cl_2, 202 MHz) 56.3 ppm (poorly resolved triplet, $\nu_{1/2}$ = 10 Hz); **4b**, 46.5 ppm (t, $^2J_{PH}$ = 30 Hz).

(14) Morris has recently reported a dihydrogen to phosphorus coupling constant, $^2J(H_2\text{–}P)$, of 5 and 5.8 Hz in $[M(\eta^2\text{-}H_2)(H)(depe)_2]^+$ (M = Fe, Os) by using NMR simulations to match experimental spectra.[15]

(15) Bautista, M.; Earl, K. A.; Morris, R. H.; Sella, A. *J. Am. Chem. Soc.* **1987**, *109*, 3780.

(16) After several hours reflux in acetonitrile, **4** is quantitatively converted to $CpRu(dmpe)(CH_3CN)^+$, identified by comparison to its dppe analogue.[17]

(17) Bruce, M. I., et al. *Aust. J. Chem.* **1979**, *32*, 1003.

(18) T_1's for the cyclopentadienyl protons of **4a** and **4b** (CD_3CN, 250 MHz) were determined by using a 180°–τ–90° pulse sequence. These were found to be 8.6 and 10.0 s for **4a** and **4b**, respectively. Saturation of the cyclopentadienyl resonance of **4b** at 5.34 ppm caused a substantial decrease in the intensity of the cyclopentadienyl resonance of **4a** at 5.17 ppm. In another experiment saturation of the **4a** resonance led to a concurrent decrease in the intensity of the **4b** signal at 5.34 ppm. Rates were calculated as previously outlined for a two-site exchange process.[19]

(19) Faller, J. W. In *Determination of Organic Structures by Physical Methods*; Nachod, F. C., Zuckerman, J. J., Eds.; Academic Press: New York, 1973; Vol. 5, Chapter 2.

(20) We calculate an activation energy of ca. 15 kcal for dihydride/dihydrogen complex interconversion in $W(CO)_3(P(i\text{-}Pr)_3)_2H_2$ (based on spectra from ref 3b).

tween **4a** and the neutral hydride **3**, but no exchange could be detected between **3** and **4b**, establishing that **4a** is more acidic than **4b**. These observations may explain the fact that dihydrogen complexes apparently catalyze the equilibration of H_2/D_2 mixtures to give HD.[3b,7] The exchange of complexed H_2 for complexed D_2 is often facile,[3b] and activation of the coordinated hydrogen (deuterium) toward deprotonation would allow a *catalytic* amount of a proton acceptor (such as water) to facilitate H^+/D^+ exchange. Alternative mechanisms which invoke tetrahydride intermediates formed by ligand dissociation are less satisfactory.[7] The H/D exchange reactions observed by Kubas in the solid state remain inexplicable.[3b]

We are continuing to investigate systematically the effects of changing ligand environments on the coordination of dihydrogen to related ruthenium complexes.

Acknowledgment. We are grateful to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for their support of this research.

(21) For the equilibrium

$$L_nM(\eta^2\text{-}H_2)^+ + B \rightleftharpoons L_nMH + BH^+$$

then

$$K_{eq} = [L_nMH][BH^+]/[L_nM(\eta^2\text{-}H_2)^+][B]$$

in dilute solution. It follows that

$$pK_{eq} = pK_a - pK_{BH^+}$$

or

$$pK_a = pK_{eq} + pK_{BH^+}$$

where pK_{BH^+} for $HNEt_3^+$ in CH_3CN is known.[22] Since $[L_nMH] = [BH^+]$, then pK_{eq} can be calculated if $[L_nMH]$, $[L_nM(\eta^2\text{-}H_2)^+]$, and [B] are known. The *relative* concentration, $[L_nMH]/[L_nM(\eta^2\text{-}H_2)^+]$, can be found by ^1H NMR integration, and $1/[B]$ can be calculated from the known starting quantity of B and $[L_nMH]/[L_nM(\eta^2\text{-}H_2)^+]$ at equilibrium.

(22) Coetzee, J. F. *Prog. Phys. Org. Chem.* **1967**, *4*, 45.

Transition-Metal-Promoted Reactions of Boron Hydrides. 10.[1] Rhodium-Catalyzed Syntheses of *B*-Alkenylborazines

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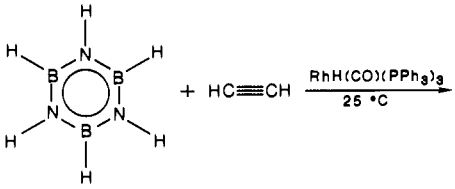
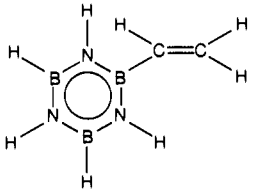
We have previously shown[1-9] that transition-metal reagents can be used to activate the B–H bonds in a variety of polyhedral boranes and carboranes and that this activation can induce numerous important synthetic transitions. We have now initiated studies of the application of transition-metal catalysts to promote the reactions of boron nitrogen ring compounds and report here the development of a metal-catalyzed, high yield route to *B*-alkenylborazines.

We have previously shown that various metal catalysts will promote the reactions of pentaborane(9) with alkynes[1-4] to give alkenyl-substituted pentaboranes. We have now found that borazine will undergo analogous reactions in the presence of rhodium catalysts.[10]





(1)

B-vinyl-$B_3N_3H_5$

In a typical reaction, 2.218 g (27.5 mmol) of borazine (Callery) and 30 mmol of acetylene were reacted in the presence of 0.0090 g (9.8×10^{-3} mmol) of $RhH(CO)(PPh_3)_3$[11] for 4 h at room temperature. Fractionation of the resulting reaction mixture gave 0.306 g (2.87 mmol) of *B*-vinyl-$B_3N_3H_5$[12] stopping in a −70 °C trap and 1.890 g (23.5 mmol) of unreacted borazine. This corresponds to a 71.8% yield based on consumed borazine and 293 catalyst turnovers during the 4-h period.

In an analogous reaction, borazine (2.472 g, 30.7 mmol) and 30 mmol of propyne were reacted in the presence of 0.0066 g (7.2×10^{-3} mmol) of $RhH(CO)(PPh_3)_3$ for 10 h at 55 °C. Fractionation of the resulting reaction mixture gave 0.364 g (3.02 mmol) of *B*-propenyl-$B_3N_3H_5$ stopping in a −65 °C trap and 2.187 g (27.16 mmol) of unreacted borazine. This corresponds to a 85.2% yield based on consumed borazine and 419 catalyst turnovers during the 10-h period. GLC analysis[13] of the product indicated that it was composed of a 80:20 mixture of *B*-*trans*-1-propenyl-$B_3N_3H_5$[14] and *B*-2-propenyl-$B_3N_3H_5$.[15] Pure samples

(1) For part 9, see: Mirabelli, M. G. L.; Sneddon, L. G. *J. Am. Chem. Soc.*, accepted for publication.
(2) Wilczynski, R.; Sneddon, L. G. *J. Am. Chem. Soc.* **1980**, *102*, 2857–2858.
(3) Wilczynski, R.; Sneddon, L. G. *Inorg. Chem.* **1981**, *20*, 3955–3962.
(4) Wilczynski, R.; Sneddon, L. G. *Inorg. Chem.* **1982**, *21*, 506–514.
(5) Davan, T.; Corcoran, E. W., Jr.; Sneddon, L. G. *Organometallics* **1983**, *2*, 1693–1694.
(6) Corcoran, E. W., Jr.; Sneddon, L. G. *Inorg. Chem.* **1983**, *22*, 182.
(7) Corcoran, E. W., Jr.; Sneddon, L. G. *J. Am. Chem. Soc.* **1984**, *106*, 7793–800.
(8) Corcoran, E. W., Jr.; Sneddon, L. G. *J. Am. Chem. Soc.* **1985**, *107*, 7446–450.
(9) Mirabelli, M. G. L.; Sneddon, L. G. *Organometallics* **1986**, *5*, 1510–1511.

(10) Nöth has also recently shown that olefin/alkyne hydroborations by organoboranes, such as catecholborane, can be catalyzed by rhodium complexes, see: Mannig, D.; Nöth, H. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 878–9.
(11) Ahmad, N.; Levison, J. J.; Robinson, S. D.; Uttley, M. F. *Inorg. Synth.* **1974**, *15*, 59–60.
(12) ^{11}B NMR (160.5 MHz, ppm, C_6D_6) 31.5 (d, $B_{4,6}$, J_{BH} = 129 Hz), 31.2 (s, B_2); ^1H NMR (200.13 MHz, ppm, C_6D_6) ~4.5 (q, J_{BH} = 128 Hz), 5.03 (t, J_{NH} = 49 Hz), 5.72 (m, ABC pattern, *J*'s calcd from PANIC simulation: $J_{H_A\text{-}H_B}$ = 2.76 Hz, $J_{H_A\text{-}H_C}$ = 19.73 Hz, $J_{H_B\text{-}H_C}$ = 13.62 Hz); exact mass calcd for $^{11}B_3{}^{12}C_2{}^{14}N_3{}^1H_8$ 107.0997, found 107.1000; IR spectrum (gas phase, NaCl windows, 10 cm cell) 3480 s, 3080 sh, m, 3070 m, 2980 m, 2970 m, 2600 m, 2520 vs, 2440 m, 1910 w, br, 1620 s, 1540 m, 1475 vs, br, 1425 s, sh, 1380 vs, 1350 s, sh, 1290 w, br, 1140 w, sh, 1125 m, 1120 m, 1015 m, 955 s, 930 vs, 920 vs, 735 s, 720 vs, 690 m, 680 m.
(13) Tricresyl phosphate (TCP) 6% on 60–80 mesh Chromsorb P, 120 °C; $R_v(B_3N_3H_6)$ = 1.0; R_v(B-2-propenyl-$B_3N_3H_5$) = 4.88; R_v(*trans*-1-propenyl-$B_3N_3H_5$) = 6.38.
(14) ^{11}B NMR (160.5 MHz, ppm, C_6D_6) 31.6 (d, $B_{4,6}$, J_{BH} = 136 Hz), 31.3 (s, B_2); ^1H NMR (250.15 MHz, ppm, C_6D_6) 1.65 (d of d, CH_3, $J_{CH_3\text{-}H_A}$ = 6.4 Hz, $J_{CH_3\text{-}H_B}$ = 1.5 Hz), 4.52 (q, BH, J_{BH} = 119 Hz), 5.04 (t, NH, J_{NH} = 44.9 Hz), 5.44 (d of d, CH, $J_{H_B\text{-}H_A}$ = 17.7, $J_{H_B\text{-}CH_3}$ = 1.2), 5.95 (d of q, CH, $J_{H_A\text{-}H_B}$ = 18.0 Hz, $J_{H_A\text{-}CH_3}$ = 6.1); exact mass calcd for $^{11}B_3{}^{12}C_3{}^{14}N_3{}^1H_{10}$ 121.115, found 121.113; IR spectrum (film, NaCl windows) 3440 vs, 3000 m, 2960 m, 2930 m, 2910 m, 2880 m, sh, 2850 m, 2600 sh, w, 2580 m, 2500 vs, 2440 m, sh, 2420 m, 1640 vs, 1460 vs, br, 1390 s, 1370 vs, 1340 s, 1310 m, 1280 w, 1270 w, 1145 w, 1125 m, 1075 w, 1055 w, 1035 w, 980 s, 920 s, 900 vs, 785 m, 715 s, 620 m.
(15) ^{11}B NMR (160.5 MHz, ppm, C_6D_6) 31.6 (d, $B_{4,6}$, J_{BH} = 132 Hz), 32.0 (s, B_2); ^1H NMR (250.15 MHz, ppm, C_6D_6) 1.63 (s, CH_3), 4.76 (q, BH, J_{BH} = 127 Hz), 5.19 (t, NH, J_{NH} = 50.0 Hz), 5.27 (s, CH, br), 5.43 (s, CH, br); exact mass calcd for $^{11}B_3{}^{12}C_3{}^{14}N_3{}^1H_{10}$ 121.115, found 121.116; IR spectrum (film, NaCl windows) 3440 s, 3050 m, 2950 m, 2930 m, 2910 m, sh, 2850 w, 2580 w, sh, 2500 s, 2420 w, 1625 m, 1615 m, 1505 s, sh, 1460 vs, br, 1410 s, 1395 s, 1380 s, 1345 m, sh, 1330 m, 1260 w, 1080 w, 1040 w, 980 w, 920 s, 900 vs, 740 m, 715 s, 625 m.

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