ORGANOMETALLICS

Oxametallacyclopentane derivatives [cyclic] (.eta.5-C5H5)2MoCH2CH(R1)C(R2)(R3)O from substituted allylic alcohols and cationic molybdenum trihydride

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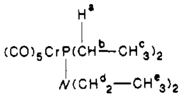
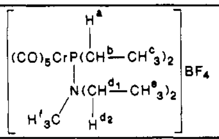
Table I. ^1H NMR Spectra of $(CO)_5CrPEt_2NEt_2$ (3) and $[(CO)_5CrP(NEt_2Me)Et_2][BF_4]$ 4 (in CH_2Cl_2)

	 $(CO)_5CrP(CH^b-CH^c_3)_2$ with H^a above and $N(CH^d_2-CH^e_3)_2$ below	 $[(CO)_5CrP(CH^b-CH^c_3)_2 / N(CH^{d_1}-CH^e_3)_2, H^f_3C, H^{d_2}]BF_4$
δ_{H_a}	2.02	2.67
δ_{H_b}	1.74	2.39
δ_{H_c}	1.11	1.37
δ_{H_d}	3.04	3.37 (d_1), 3.18 (d_2)
δ_{H_e}	1.15	1.57
δ_{H_f}		3.24
$^2J_{ab}$	14.6	15.1
$^3J_{ac}$	7.3	7.6
$^3J_{bc}$	7.3	7.7
$^3J_{de}$	6.8	7.3
$^2J_{H_aP}$	9.7	9.4
$^2J_{H_bP}$	0.2	0.5
$^3J_{H_cP}$	10.8	17.7
$^3J_{H_dP}$	9.1	*a*

[a] Because of the large quadrupole effect of the quarternary nitrogen, the resolution is not high enough to allow the evaluation of the $^3J_{H_{d1}P}$, $^3J_{H_{d2}P}$, $^3J_{H_fP}$, $^3J_{H_{d1}Me}$, $^3J_{H_{d2}H_e}$, and $^3J_{H_{d1}H_{d2}}$ coupling constants.

hydrogen halides insofar as it is not possible to isolate any intermediate complexes.

Reaction 1 is very convenient for monofluorination, since even with a large excess of $[Me_3O][BF_4]$ only one of the three NEt_2 groups is exchanged. This proved also to be the case with other compounds such as $(CO)_5CrPPh(NEt_2)_2$ and $(CO)_5CrP(-OC_2H_4O-)NEt_2$, whereby $(CO)_5CrPPh(NEt_2)F$ and $(CO)_5CrP(-OC_2H_4O-)F$ were obtained in 82% and 32% yields, respectively.

If $(CO)_5CrPPh_2NEt_2$ is used as starting material, besides $(CO)_5CrPPh_2F$ (57%), *cis*-/*trans*-$(CO)_4Cr(PPh_2F)_2$ (19%) is isolated. The latter compound stems from CO substitution. The ratio of mono- to disubstitution depends on the solvent used for the reaction; in CH_2Cl_2 it is 3:1 and in 1,2-dimethoxyethane, 1:1. In both cases the cis/trans ratio is an almost constant 3:1.

When substituents on the phosphorus were changed, we were able to synthesize 4 starting from 3. 4 is the first

$$\underset{\mathbf{3}}{(CO)_5CrPEt_2NEt_2} \xrightarrow{[Me_3O][BF_4]} \underset{\mathbf{4}}{[(CO)_5CrP(NEt_2Me)Et_2][BF_4]} \qquad (2)$$

complex with a (trialkylammonio)phosphane ligand. Alkylation of the lone electron pair of the nitrogen atom of 3 results in smaller donor/larger acceptor capacities of the phosphane ligand. Therefore, the CO-valence frequencies are shifted toward shorter wavelengths (3, 2050 (m), 1924 (vs) cm^{-1}; 4, 2078 (m), 1959 (sh), 1930 (vs) cm^{-1} in CH_2Cl_2). The large downfield shift of the ^{31}P NMR signal from 115.7 to 199.4 ppm is striking, since on protonation the signal is shifted in the opposite direction.[3,5] On the other hand, one would expect deshielding of the phosphorus when it is directly bound to a quarternary nitrogen with a full positive charge on it. The presence of the NEt_2Me group is assured by ^1H NMR spectroscopy. Conductance measurements in acetone are consistent with the formulation of a 1:1 salt.

Compound 4 corresponds to the intermediates which are to be assumed in the fluorination reactions we discussed in eq 1. At present we cannot give a definite explanation for the stability of 4. However, it is remarkable that the phosphorus in 3 has only one substituent with a lone electron pair, the NEt_2 group. So we are of the opinion that at least one nucleophilic group in the neighborhood of the reacting NEt_2 group might be necessary for the activation of the fluorinating properties of the BF_4^- ion.

Experimental Section. All experiments were performed under a nitrogen atmosphere by using Schlenk glassware. The solvents were free from moisture and oxygen.

$(CO)_5CrPF(NEt_2)_2$. To a 2.20g (5.0 mmol) of 1, dissolved in 40 mL of CH_2Cl_2, was added 2.22 g (15.0 mmol) of $[Me_3O][BF_4]$ as the solid. The mixture was heated at reflux for 48 h and then concentrated to 10 mL. Addition of 20 mL of hexane allowed removal of the excess oxonium salt by filtration. After evaporation of the solvent, the product was purified twice by dissolving it in hexane and subsequent by cooling to −78 °C; 1.68 g (87% yield) of a pale yellow oil (2) was obtained. Anal. Calcd for $C_{13}H_{20}CrFN_2O_5P$ (2): C, 40.42; H, 5.22; N, 7.25. Found: C, 40.5; H, 5.2; N, 7.6. IR (hexane): ν_{CO} 2070 (m), 1958 (s), 1944 (vs) cm^{-1} δ_{MCO} 674 (m), 659 (m) cm^{-1}; ν_{PF} 770 (m) cm^{-1}; δ_{PF} 518 (w) cm^{-1}. MS (70 eV): M^+ 386 (6.2%). $^{19}F/^{31}P$ NMR (CD_2Cl_2): δ_F −50.05 (d), δ_P 198.17 (d), $^1J_{PF}$ = 1034.5 Hz.

$[(CO)_5CrP(NEt_2Me)Et_2][BF_4]$. Following the procedure given above 1.77 g (5.0 mmol) of 3 was treated with 2.22 g (15.0 mmol) of $[Me_3O][BF_4]$, but instead of hexane, 20 mL ether was added to the concentrated solution to remove excess oxonium salt. The crude product was recrystallized from 1:1 CH_2Cl_2/hexane to give 1.52 g of 4 as colorless crystals (mp 92 °C dec) in 66% yield. Anal. Calcd for $C_{14}H_{23}BCrF_4NO_5P$ (4): C, 36.94; H, 5.09; N, 3.08. Found: C, 36.7; H, 5.1; N, 3.5. IR (CH_2Cl_2): ν_{CO} 2078 (m), 1959 (sh), 1930 (vs) cm^{-1}. ^{31}P NMR (CD_2Cl_2): δ 199.4. Conductivity in acetone: $\Lambda(C\rightarrow O) = 59.6 \pm 0.5\ S\cdot val^{-1}\cdot cm^2$.

Acknowledgment. We thank the Fonds der Chemischen Industrie and Prof. Th. Kruck for supporting this work.

Registry No. 1, 105502-21-4; 2, 105502-22-5; 3, 105502-23-6; 4, 105502-25-8; $[Me_3O][BF_4]$, 420-37-1; $(CO)_3CrPPh(NEt_2)_2$, 105502-26-9; $(CO)_5CrP(-OC_2H_4O-)NEt_2$, 105502-27-0; (CO)-$CrPPh(NEt_2)F$, 105502-28-1; $(CO)_5CrP(-OC_2H_4O-)F$, 105502-29-2; $(CO)_5CrPPh_2NEt_2$, 105502-30-5; $(CO)_5CrPPh_2F$, 105502-31-6; *cis*-$(CO)_4Cr(PPh_2F)_2$, 105502-32-7; *trans*-$(CO)_4Cr(PPh_2F)_2$, 105561-82-8.

Oxametallacyclopentane Derivatives $(\eta^5\text{-}C_5H_5)_2\overline{MoCH_2CH(R^1)C(R^2)(R^3)O}$ from Substituted Allylic Alcohols and Cationic Molybdenum Trihydride

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Summary: The mechanism of formation of a cationic (γ-hydroxyalkyl)molybdenum complex by the reaction of $[(\eta\text{-}C_5H_5)_2MoH_3]^+[p\text{-}CH_3C_6H_4SO_3]^-$ with a substituted allylic alcohol is discussed in terms of the olefin insertion into the Mo–H bond. The former was deprotonated by treatment with NaOH to give the neutral oxametallacycle $(\eta\text{-}C_5H_5)_2\overline{MoCH_2CH(R^1)C(R^2)(R^3)O}$.

Recently we reported the formation of the cationic molybdenum(IV) complexes (1), which possess a cyclic γ-hydroxyalkyl structure, by the reaction of cationic tri-

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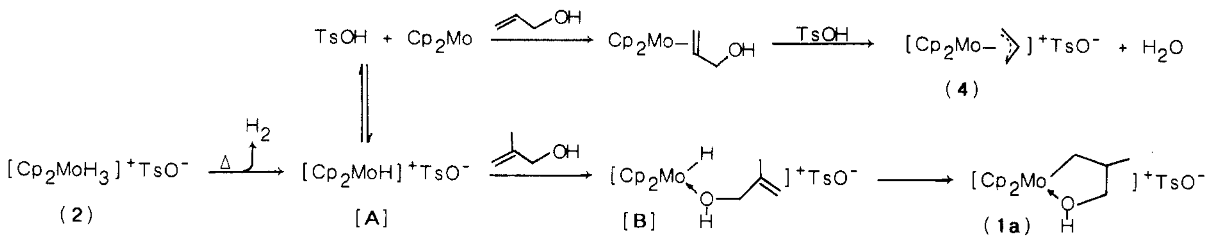
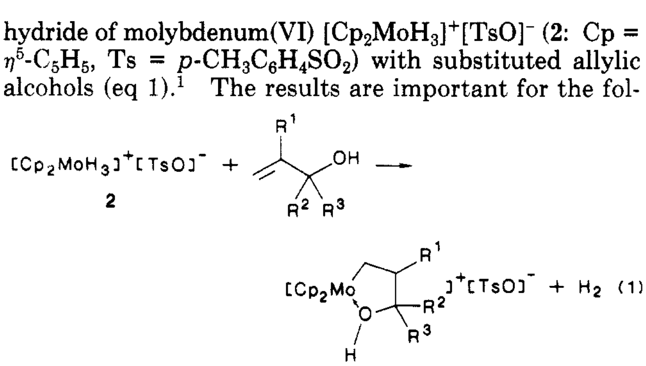
Scheme I



hydride of molybdenum(VI) $[Cp_2MoH_3]^+[TsO]^-$ (2: Cp = η^5-C_5H_5, Ts = p-$CH_3C_6H_4SO_2$) with substituted allylic alcohols (eq 1).[1] The results are important for the following two reasons: (1) Complex 1 can be regarded as a possible intermediate in the transition-metal-mediated hydroxyl-directed olefin hydrogenation, which is of current interest in terms of OH-directed stereoselective hydrogenations.[2] (2) Complex 1 is expected to be a precursor of an oxametallacyclic complex of molybdenum which may be viewed as a model for an intermediate in the transition-metal-catalyzed metathesis reaction and/or oxidation of alkenes. Here we report that deprotonation of 1 gives neutral oxametallacyclopentane derivatives 3.

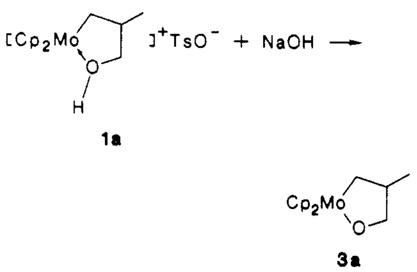


1a: $R^1 = CH_3$; $R^2 = R^3 = H$
b: $R^1 = H$; $R^2 = R^3 = CH_3$

As described previously[1] the trihydridomolybdenum cation 2 reacts with substituted allylic alcohols at 50–80 °C to give cyclic γ-hydroxyalkyl complexes **1a** and **1b** (eq 1). However, when allyl alcohol was allowed to react with 2, selective C–O bond cleavage took place to give the cationic η^3-allyl complex $[Cp_2Mo(\eta^3\text{-}C_3H_5)]^+[TsO]^-$ (4).[3] These two reactions may proceed through the common, coordinatively unsaturated monohydrido intermediate A in Scheme I.[1] Whereas allyl alcohol may interact first with the molybdenocene through the double bond (Scheme I), substituted allylic alcohols, which may be more basic at the oxygen atom as well as more hindered olefin, may add to the cationic intermediate A through its oxygen atom to give the adduct B. It should be noted that the methanol adduct corresponding to B has been isolated from the reaction of 2 with MeOH which is more basic than substituted allyl alcohols.[1]

For verification of the insertion process in the Scheme I, $[Cp_2Mo(D)(EtOD)]^+TsO^-$, which was prepared by the reaction of 2 with EtOD, was allowed to react with excess β-methallyl alcohol to give $[Cp_2MoCH_2CD(CH_3)CH_2OH]^+TsO^-$ (**1a**-d_1) as is judged from its IR and ^1H NMR spectra. The degree of deuteriation at the β-carbon in **1a**-d_1 was found to be ca. 60% by its ^1H NMR spectrum, indicating that a part of the Mo–D in $[Cp_2Mo(D)(EtOD)]^+$ has been exchanged with the alcoholic proton of β-methallyl alcohol during the reaction. The absence of the H–D exchange between solvent CD_3OD and complex **1a** was evidenced by the ^1H NMR measurement of **1a** in CD_3OD, which did not show any sign of the line broadening at room temperature except the signal assignable to the alcoholic hydrogen in **1a**.

Treatment of the cyclic γ-hydroxyalkyl complex **1a** with an equimolar amount of NaOH in ethanol gave the orange neutral oxamolybdenacyclopentane derivative $Cp_2MoCH_2CH(CH_3)CH_2O$ (**3a**)[5] in 70% yield together with NaOTs. A similar reaction of **1b** with NaOH afforded the corresponding metallacycle derivative **3b**.[6] Both complexes **3a** and **3b** are moderately sensitive to oxygen and highly moisture sensitive. Absence of any peaks in the region of m/e 320–800 in the mass spectra of **3a** and **3b** taken at 20 eV suggests that these complexes are mononuclear complexes as shown in eq 2. The related oxametallacyclopentane derivatives have been reported recently for Ti,[7,8] Zr,[9] and W.[10] However, the method of preparation shown here has not, to our knowledge, previously been described.



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[3] Ito, T.; Igarashi, T.; Suzuki, F., *J. Organomet. Chem.*, in press. The analogous complex $[Cp_2Mo(\eta^3\text{-}C_3H_5)]^+[PF_6]^-$ has been prepared via rather complicated routes starting from Cp_2MoH_2: see ref 4. Spectral (^1H and ^{13}C NMR and IR) and analytical (C and H) results for 4 were all consistent with the formulation.

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[5] 2,2-Bis(η^5-cyclopentadienyl)-4-methyl-1-oxa-2-molybdenacyclopentane (**3a**): mp (under vacuum) 140–142 °C dec; ^1H NMR (90 MHz, C_6D_6) δ 4.60 (5 H, s, Cp), 4.51 (5 H, s, Cp), 4.4 (1 H, m, OCH_2), 3.07 (1 H, dd, $^2J_{HH} = 10.3$ Hz, $^3J_{HH} = 9.0$ Hz, OCH_2), 2.14 (1 H, dd, $^2J_{HH} = 2.3$ Hz, $^3J_{HH} = 5.0$ Hz, $MoCH_2$), 1.8 (1 H, m, CMe*H*), 1.70 (1 H, dd, $^2J_{HH} = 2.3$ Hz, $^3J_{HH} = 7.2$ Hz, $MoCH_2$), 1.27 (3 H, d, $^3J_{HH} = 5.4$ Hz, C*Me*H); ^{13}C NMR (22.5 MHz, C_6D_6) δ 93.94 (d, Cp), 84.51 (dd, C_5), 42.47 (apparent d, C_4), 26.65 (dd, C_3), 19.39 (quart, CH_3); mass spectrum (70 eV), m/e (relative intensity) 300 (15, M^+), 228 (100, Cp_2Mo). Anal. Calcd for $C_{14}H_{18}MoO$ (**3a**): C, 56.38; H, 6.08. Found: C, 56.67; H, 6.20.

[6] 2,2-Bis(η^5-cyclopentadienyl)-5,5-dimethyl-1-oxa-2-molybdenacyclopentane (**3b**): mp (under vacuum) 163–165 °C dec; ^1H NMR (90 MHz, C_6D_6) δ 4.53 (10 H, s, Cp_2), 2.10 (2 H, apparent t, $^3J_{HH} = 5.8$ Hz, $MoCH_2$), 1.77 (2 H, apparent t, $^3J_{HH} = 6.1$ Hz, $MoCH_2CH_2$), 1.19 (6 H, s, CMe_2); ^{13}C NMR (22.5 MHz, C_6D_6) δ 93.87 (d, Cp_2), 82.93 (s, C_5), 47.61 (t, C_4), 30.81 (quart, Me_2), 11.53 (t, C_3); mass spectrum (70 eV), m/e (relative intensity) 314 (18, M^+), 228 (100, Cp_2Mo). Anal. Calcd for $C_{15}H_{20}MoO$ (**3b**): C, 57.70; H, 6.45. Found: C, 57.47; H, 6.43.

[7] Cohen, S. A.; Bercaw, J. E. *Organometallics* **1985**, *4*, 1006-1014.

[8] Mashima, K.; Jyodoi, K.; Ohyoshi, A.; Takaya, H. *J. Chem. Soc., Chem. Commun.* **1986**, 1145–1146.

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In the ^{13}C NMR of complexes 3,[5,6] the C_3 carbon which is bonded to molybdenum was found to resonate at considerably higher field as compared with the reported value for the same carbon in $(\eta^5\text{-}C_5Me_5)_2\overline{TiCH_2CH_2CH(Me)O}$ (55.8 ppm).[7] This implies that the higher electron density at the metal center in the molybdenum(IV) complexes than in titanium(IV) in spite of the opposite order of the propensity for the electron-donating ability between cyclopentadienyl and pentamethylcyclopentadienyl ligands.

The neutral oxametallacycle derivatives **3a** and **3b** revert to the parent cyclic (γ-hydroxyalkyl)molybdenum cations **1a** and **1b**, respectively, on treatment with an equimolar amount of TsOH in Et_2O. Similarly, the reaction of **3a** with anhydrous hydrogen chloride in Et_2O yields the chloride analogue of **1a** $[Cp_2\overline{MoCH_2CH(CH_3)CH_2O}H]^+Cl^-$.

Acknowledgment. We thank Dr. F. Ozawa, Tokyo Institute of Technology, for the measurement of mass spectroscopy.

Registry No. **1a**, 103822-69-1; **1a**-d_1, 105517-88-2; **1b**, 103822-61-3; **2**, 103822-61-3; **3a**, 105472-71-7; **3b**, 105472-72-8; **4**, 105472-70-6; $[Cp_2Mo(D)(EtOD)]^+TsO^-$, 105472-69-3; $[Cp_2\overline{MoCH_2CH(CH_3)CH_2O}H]Cl$, 105472-73-9; allyl alcohol, 107-18-6; β-methallyl alcohol, 513-42-8.

Synthesis and Molecular Structure of a Novel Aluminacarborane, *nido*-[μ-6,9-AlEt(OEt$_2$)-6,9-C$_2$B$_8$H$_{10}$]

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Summary: Synthesis and characterization of *nido*-[μ-6,9-AlEt(OEt$_2$)-6,9-C$_2$B$_8$H$_{10}$] (**2**) is outlined, and the results of a single-crystal X-ray structure determination for **2** are presented. Ligand exchange of OEt$_2$ in **2** for THF to produce [μ-6,9-AlEt(THF)-6,9-C$_2$B$_8$H$_{10}$] (**3**) is reported, and direct synthesis of **3** is described. Spectroscopic data for **2** and **3** are reported, and bonding in these species is discussed.

Interest in hetero- and metallacarboranes derived from the main-group elements stems from the unusual structures and bonding modes exhibited by these compounds. Characterizations of 12-vertex aluminacarboranes of composition $C_2B_9H_{11}AlR$ (R = Me, **1a**; R = Et, **1b**) in which an aluminum atom is bound to the five-membered face of the dicarbollide ligand have been described previously.[1] Synthesis of **1a** and **1b** was accomplished by direct reaction of $C_2B_9H_{13}$ with the appropriate trialkylaluminum reagent. Preparation of the bis(tetrahydrofuran) (THF) adduct of **1b** by reaction of $Na_2C_2B_9H_{11}$ with $EtAlCl_2$ in THF has been reported.[2] Here we wish to describe the synthesis by a related reaction of an aluminacarborane derived from the formal $[C_2B_8H_{10}]^{2-}$ ligand and its structural characterization.

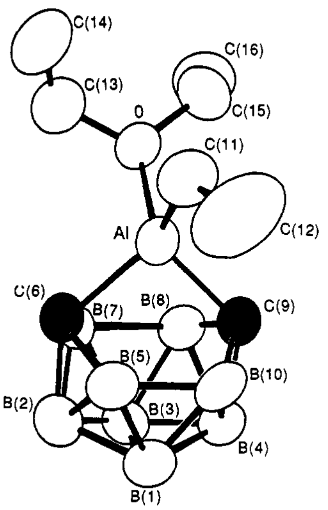

Figure 1. Molecular structure of **2**, with hydrogen atoms omitted for clarity and thermal ellipsoids drawn at 50% probability level. Interatomic distances (Å) and angles (deg): Al–C(6) = 2.030 (3), Al–C(9) = 2.025 (3), Al–B(5) = 2.500 (4), Al–B(7) = 2.509 (4), Al–B(8) = 2.499 (3), Al–B(10) = 2.514 (4), Al–C(1) = 1.966 (3), Al–O(1) = 1.909 (2); C(6)–Al–C(9) = 99.44 (12), C(1)–Al–O(1) = 99.87 (13), C(13)–O(1)–C(15) = 115.88 (25).

Reaction of diethylaluminum chloride–diethyl etherate with an equimolar amount of $Na[5,6\text{-}C_2B_8H_{11}]$[3] in refluxing toluene resulted in evolution of gas and formation of [μ-6,9-AlEt(OEt$_2$)-6,9-C$_2$B$_8$H$_{10}$] (**2**).[4] This reaction presumably occurs according to eq 1.

$$\begin{gathered}Na[5,6\text{-}C_2B_8H_{11}] + Et_2AlCl{\cdot}OEt_2 \xrightarrow[\text{reflux}]{\text{toluene}} \\ nido\text{-}[\mu\text{-}6,9\text{-}AlEt(OEt_2)\text{-}6,9\text{-}C_2B_8H_{10}] + NaCl + C_2H_6\end{gathered} \quad (1)$$

Purification by high vacuum fractional distillation afforded **2**, a colorless, viscous liquid, in 77% yield.[5] Complex **2** is air- and water-sensitive, decomposing visibly within seconds upon exposure to the atmosphere and reacting vigorously with water. Crystals (mp 28–30 °C) could be obtained by layering benzene solutions of **2** with pen-

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(3) Plesek, J.; Hermanek, S. *Collect. Czech. Chem. Commun.* **1973**, *38*, 338.

(4) Preparation of **2**: All manipulations were carried out under dry N_2 or in vacuo by using standard Schlenk line, glovebox, and vacuum line apparatus. In a typical experiment, 16.6 mL of a 0.5 M toluene solution of $Et_2AlCl{\cdot}OEt_2$ (8.3 mmol) was added rapidly to a stirred suspension of freshly prepared $Na[5,6\text{-}C_2B_8H_{11}]$ (1.2 g, 8.3 mmol) in 200 mL of toluene at 0 °C in a 500-mL, round-bottom Schlenk flask. The mixture was stirred at room temperature for 1 h. The flask was then fitted with a condenser and solution was refluxed under N_2 for 16 h. The resulting mixture was filtered and the volume of the filtrate reduced in vacuo to ca. 20 mL. Product **2** was precipitated from the filtrate as a pale yellow oil by addition of 150 mL of dry *n*-pentane. The product was precipitated twice more from benzene/*n*-pentane. Further purification was accomplished by distillation under high vacuum at 60 °C to a 0 °C U-trap to give liquid **2** (1.6 g, 77% yield) as a colorless distillate.

(5) Spectroscopic data for **2**: 160.5 MHz ^{11}B NMR (C_6D_6; referenced to external $BF_3{\cdot}OEt_2$ at 0 ppm with chemical shift values upfield of 0 ppm reported as negative; ambient temperature; splitting patterns and areas given in parentheses) δ 4.3 (d, 1), 2.9 (d, 2), −25.3 (d, 1), singlets upon ^1H decoupling; 200.1-MHz ^1H NMR (C_6D_6; referenced to residual protons in C_6D_6 at 7.15 ppm; ambient temperature) δ 4.69 (s, 2, carboranyl C–H), 2.97 (q, 4, coordinated OEt_2), 0.95 (t, 2, Al-bound ethyl group), 0.51 (t, 6, coordinated OEt_2), −0.38 (q, 3, Al-bound ethyl group), broad B–H resonances in the 5.1–0.1 ppm region; characteristic IR (NaCl; neat press; cm^{-1}) 2943 (vs), 2793 (m), 2548 (vs, B–H stretch), 1468 (s), 1392 (s), 1327 (m), 1287 (m), 1192 (s), 1150 (s), 1090 (s), 1053 (m), 1012 (vs), 953 (m), 890 (s), 833 (m), 770 (s).

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