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Molybdenum Complexes of an Eight-Electron-Donor Monocarbollide Ligand, Geometrically Constrained by Intramolecular Five-Membered-Ring Formation

Jason A. Kautz, Denis A. Kissounko, Natalia S. Kissounko, and
F. Gordon A. Stone*

Department of Chemistry and Biochemistry, Baylor University, Waco, Texas 76798-7348

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Summary: The complex $[N(PPh_3)_2][1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{10}]$ (**1**) *is obtained by deprotonating* $7\text{-}NH_2C(O)Me\text{-}nido\text{-}7\text{-}CB_{10}H_{12}$ *with NaH in THF, adding NCMe and* $[Mo(CO)_6]$*, and refluxing the mixture, with a final addition of* $[N(PPh_3)_2]Cl$*. Compound* **1** *undergoes cage-substitution reactions with* $O(CH_2)_4$ *or* Me_2S *in the presence of* CF_3SO_3Me *and* H_2SO_4*, respectively, to yield charge-compensated complexes* $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}7\text{-}L\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_9]$ ($L = O(CH_2)_4$ (**2**), SMe_2 (**3**)), *and with* $[Me_2N{=}CH_2]Cl$ *and* $TlPF_6$ *to afford* $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}7\text{-}NMe_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_9]$ (**4**).

Introduction

With the isolation of molybdenum,[1] rhodium,[2] ruthenium, and osmium complexes[3] containing the C-amine and -amino carborane ligands $[7\text{-}NR_3\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{2-}$ and $[7\text{-}NR_2\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ ($R = H$ or alkyl), respectively, it is becoming apparent that a range of transition metal complexes containing these di- and trianions should exist. These anions are isolobal with the groups $[nido\text{-}7,8\text{-}C_2B_9H_{11}]^{2-}$ and $[nido\text{-}7\text{-}CB_{10}H_{11}]^{3-}$, respectively, species known to form a host of metal complexes.[4] However, some recent studies with the C-amine and -amino carborane ligands reveal metal ligating properties that differ significantly from those of their $[nido\text{-}7,8\text{-}C_2B_9H_{11}]^{2-}$ and $[nido\text{-}7\text{-}CB_{10}H_{11}]^{3-}$ counterparts. Two examples suffice to illustrate this.

The exopolyhedral amino groups of $[7\text{-}NR_2\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ ligands can in some instances form intramolecular donor bonds to the metal centers to which the CBBBB ring of the *nido*-carborane fragment is pentahapto coordinated. Thus, oxidation of the trianion $[1\text{-}NHBu^t\text{-}2,2,2\text{-}(CO)_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{10}]^{3-}$ affords $[1,2\text{-}\mu\text{-}NHBu^t\text{-}2,2,2\text{-}(CO)_3\text{-}2,1\text{-}MoCB_{10}H_{10}]^-$, isolated as its $[N(PPh_3)_2]^+$ salt.[1a] In the latter complex the $[7\text{-}NHBu^t\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ moiety functions formally as an eight-electron ($6\pi + 2\sigma$) donor group to the molybdenum. In another study it was shown that the amine substituent on the carbon in the charge-compensated complex $[1\text{-}NH_3\text{-}2\text{-}Cl\text{-}2\text{-}PPh_3\text{-}closo\text{-}2,1\text{-}RhCB_{10}H_{10}]$ reacts with acetic anhydride, affording $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2\text{-}PPh_3\text{-}closo\text{-}2,1\text{-}RhCB_{10}H_{10}]$, a species in which a $[7\text{-}\{NHC(Me){=}O\}\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ ligand is pentahapto coordinated to the rhodium and also attached to the metal by a donor bond from the carbonyl of the amido group.[2d] Herein we report some molybdenum species in which the mode of attachment of this $[7\text{-}\{NHC(Me){=}O\}\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ group to the metal is similar to that observed with rhodium.

Results and Discussion

Deprotonation of the carborane $7\text{-}NH_2C(Me){=}O\text{-}nido\text{-}7\text{-}CB_{10}H_{12}$[5] with NaH in THF (tetrahydrofuran) followed by addition of $[Mo(CO)_6]$ in NCMe and refluxing the mixture, with a further addition of $[N(PPh_3)_2]Cl$, gave $[N(PPh_3)_2][1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{10}]$ (**1**) purified by column chromatography. The IR spectrum revealed CO bands at 2015 and 1933 cm^{-1}, but significantly no absorption due to the C=O group of the amido substituent was observed. The $^{11}B\{^1H\}$ NMR spectrum exhibited a pattern of four resonances at δ −4.7 (3B), −8.6 (2B), −17.2 (2B), and −3.1 (3B). The $^{13}C\{^1H\}$ NMR spectrum revealed peaks for the cage-carbon and amido C=O nuclei at δ 100.5 (br) and 180.7, respectively. The data support the formulation for **1** depicted with the amido group coordinated to the molybdenum. The intramolecular ring system MoCN(H)C(Me)=O is very similar to that MoCN(H)C(Me)=NH existing in the anion $[1,2\text{-}\mu\text{-}\{NHC(Me){=}NH\}\text{-}2,2,2\text{-}(CO)_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{10}]^-$, formed by insertion of NCMe into an NH bond of the transient species $[1\text{-}NH_2\text{-}2,2,2\text{-}(CO)_3\text{-}2\text{-}NCMe\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{10}]^-$ with concomitant migration of a hydrogen atom.[1b]

Reactions of **1** with electrophilic reagents were next explored. We have recently shown that hydride is readily removed from a BH vertex of the monoanionic complex $[2,2,2\text{-}(CO)_3\text{-}2\text{-}PPh_3\text{-}closo\text{-}2,1\text{-}MoCB_{10}H_{11}]^-$ upon treatment with electrophiles (H_2SO_4, CF_3SO_3Me), and

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10.1021/om020103l CCC: $22.00

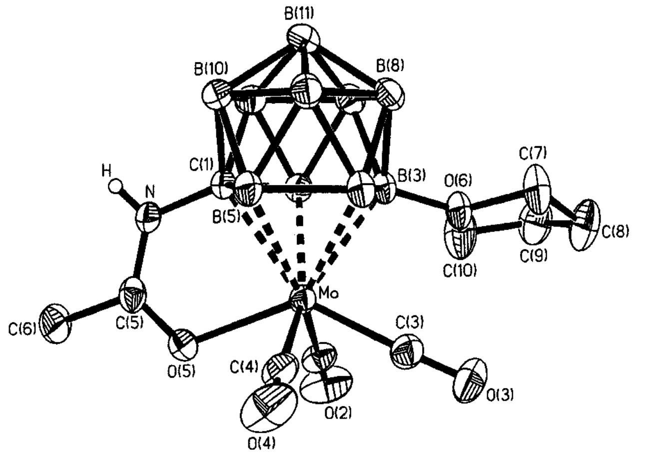

Figure 1. Molecular structure of **2** (40% probability ellipsoids; hydrogens, except for the amido hydrogen (H) are omitted). Selected bond lengths (Å) and angles (deg): Mo−C(3) 1.979(3), Mo−C(4) 2.022(3), Mo−C(2) 2.031(3), Mo−O(5) 2.198(2), Mo−B(3) 2.333(3), Mo−B(4) 2.342(3), Mo−B(5) 2.361(3), Mo−C(1) 2.374(3), Mo−B(2) 2.388(3), C(1)−N 1.455(3), B(3)−O(6) 1.533(4), N−C(5) 1.311(4), C(5)−O(5) 1.256(4), C(3)−Mo−O(5) 135.13(12), C(4)−Mo−O(5) 81.49(12), C(2)−Mo−O(5) 77.78(11), O(5)−Mo−B(3) 139.58(10), O(5)−Mo−C(1) 72.99(9), N−C(1)−Mo 105.3(2), O(6)−B(3)−Mo 114.5(2), C(5)−N−C(1) 121.7(3), O(5)−C(5)−N 120.7(3), O(5)−C(5)−C(6) 119.9(3), C(5)−O(5)−Mo 119.0(2)

in the presence of donor molecules (L) charge-compensated species [2,2,2-$(CO)_3$-2-PPh_3-7-L-*closo*-2,1-$MoCB_{10}H_{10}$] are formed.[6a,b] A similar reactivity pattern is displayed by [2,2,2,2-$(CO)_4$-*closo*-2,1-$MoCB_{10}H_{10}]^-$ on treatment with iodine in the presence of ethers or sulfides.[6c] Treatment of **1** with CF_3SO_3Me in a CH_2Cl_2−THF solvent mixture afforded [1,2-*μ*-{NHC(Me)=O}-2,2,2-$(CO)_3$-7-$O(CH_2)_4$-*closo*-2,1-$MoCB_{10}H_9$] (**2**). In the fully coupled ^{11}B NMR spectrum a diagnostic singlet peak appears at δ 22.7 for the $BO(CH_2)_4$ nucleus.[6] Resonances for the cage-C and C=O nuclei occur at δ 98.9 (br) and 182.3, respectively, in the $^{13}C\{^1H\}$ NMR spectra. In the ^1H NMR spectrum the NH signal is broad at δ 6.74. Suitable crystals of **2** were available for an X-ray diffraction study, revealing the structure shown in Figure 1. The presence of the intramolecular $\overline{\mathrm{MoCN(H)C(Me){=}O}}$ feature in **2** is confirmed and thus may be inferred for **1** also. The exopolyhedral cage-$O(CH_2)_4$ substituent is bonded to a boron β to the carbon atom in the $\overline{\mathrm{CB}\mathit{B}\mathrm{BB}}$ ring coordinated to the molybdenum. This site for substitution by the donor molecule is the preferred one in reactions of this type.[6]

The reaction between **1** and SMe_2 in CH_2Cl_2 on addition of H_2SO_4 gives [1,2-*μ*-{NHC(Me)=O}-2,2,2-$(CO)_3$-7-SMe_2-*closo*-2,1-$MoCB_{10}H_9$] (**3**). In the fully coupled ^{11}B NMR spectrum there is a singlet resonance for the $BSMe_2$ nucleus at δ 5.0. The corresponding peak in the spectrum of [2,2,2-$(CO)_3$-2-PPh_3-7-SMe_2-*closo*-2,1-$MoCB_{10}H_{10}$] is at δ 3.4.[6b] Other significant peaks in the NMR are at ^1H, δ 6.12 (br, NH); $^{13}C\{^1H\}$, δ 101.2 (br, cage-C), 182.7 (C=O). In the presence of $TlPF_6$, the reagent $[Me_2N{=}CH_2]Cl$ reacts with **1** in CH_2Cl_2 to afford [1,2-*μ*-{NHC(Me)=O}-2,2,2-$(CO)_3$-7-NMe_3-*closo*-2,1-$MoCB_{10}H_9$] (**4**), albeit in low yield. Significant peaks observed in the NMR spectra are seen at ^1H, δ 2.64 (NMe_3), 6.86 (br, NH); $^{13}C\{^1H\}$, δ 99.3 (br, cage-C), 182.9 (C=O); ^{11}B, 18.3 (s, $BNMe_3$).

Oxidation of the Mo^{II} compound **1** with iodine in the presence of excess $CNBu^t$ gives the Mo^{IV} complex [1,2-*μ*-{NHC(Me)=O}-2,2-$(CNBu^t)_2$-2-CO-2-I-*closo*-2,1-$MoCB_{10}H_{10}$] (**5**). The latter is akin to the recently described species [1,2-*μ*-$NHBu^t$-2,2,2-$(CNBu^t)_3$-2-I-*closo*-2,1-$MoCB_{10}H_{10}$].[1a]

Conclusions

The new complexes **1**−**5** are to the best of our knowledge the first molybdenacarboranes in which an exopolyhedral amido group forms a bridge from the cage carbon to the metal, thereby augmenting the pentahapto ligation of the *nido*-CB_{10} moiety. A similar bonding to the metal was recently observed with [1,2-*μ*-{NHC(Me)=O}-2-PPh_3-*closo*-2,1-$RhCB_{10}H_{10}$].[2d] The bonding of the C=O linkage of the amido group to the molybdenum in **1**−**5** is evidently strong, since this interaction is not lifted on treatment with phosphines, nor by Me^+ or H^+ in the formation of **2** and **3**. In **1** and **5** the $[7\text{-}\{N(H)C(Me){=}O\}\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ ligand formally functions as an eight-electron ($6\pi + 2\sigma$) donor to the Mo^{II} and Mo^{IV} centers, respectively. A similar role is played by the $[7\text{-}NHBu^t\text{-}nido\text{-}7\text{-}CB_{10}H_{10}]^{3-}$ moiety in $[1,2\text{-}\mu\text{-}NHBu^t\text{-}2,2,2\text{-}(CO)_3\text{-}2,1\text{-}MoCB_{10}H_{10}]^-$.[1a] These results show how the customary six-electron-donor capability of the $[nido\text{-}7\text{-}CB_{10}H_{11}]^{3-}$ group may be increased to eight by attachment at the cage-carbon vertex of a suitable donor group.

Experimental Section

General Considerations. Experimental procedures have been described previously.[1a,6] The NMR measurements were recorded at the following frequencies: ^1H, 360.13; ^{13}C, 90.56; ^{11}B, 115.55 MHz. A complete listing of NMR data for the new compounds is given in the Supporting Information. The carborane 7-$NH_2C(Me){=}O$-*nido*-7-$CB_{10}H_{12}$ was synthesized according to the literature procedure.[5] All other reagents were used as supplied. Chromatography columns (ca. 20 cm in length and 2 cm in diameter) were packed with silica gel (Acros, 60−200 mesh).

Synthesis of 1. The carborane 7-$NH_2C(Me){=}O$-*nido*-7-$CB_{10}H_{12}$ (0.62 g, 3.24 mmol) in THF (10 mL) was treated with NaH (3.2 mmol, 60% suspension in mineral oil). The mixture was stirred (10 min), then MeCN (30 mL) and $[Mo(CO)_6]$ (0.85 g, 3.22 mmol) were added. After refluxing the mixture for 3 h the salt $[N(PPh_3)_2]Cl$ (1.86 g, 3.24 mmol) was added, and refluxing continued for a further 1 h. Solvents were removed in vacuo, and the oily residue was extracted with the minimum amount of CH_2Cl_2 (ca. 3 mL). The extract was chromatographed, eluting with CH_2Cl_2. A purple fraction was collected, from which solvent was removed in vacuo, affording purple microcrystals of $[N(PPh_3)_2]$[1,2-*μ*-{NHC(Me)=O}-2,2,2-$(CO)_3$-*closo*-2,1-$MoCB_{10}H_{10}$] (**1**) (2.11 g, 72%). The compound was sufficiently pure for further syntheses. Analytically pure samples were obtained by crystallization from a CH_2Cl_2 solution layered with petroleum ether. IR (CH_2Cl_2): ν_{max}(CO) 2015 s, 1933 s cm^{-1}. Anal. Calcd for $C_{42}H_{44}B_{10}MoN_2O_4P_2\cdot 0.5\,CH_2Cl_2$: C, 53.8; H, 4.8; N, 3.0. Found: C, 53.8; H, 4.9; N, 3.0.

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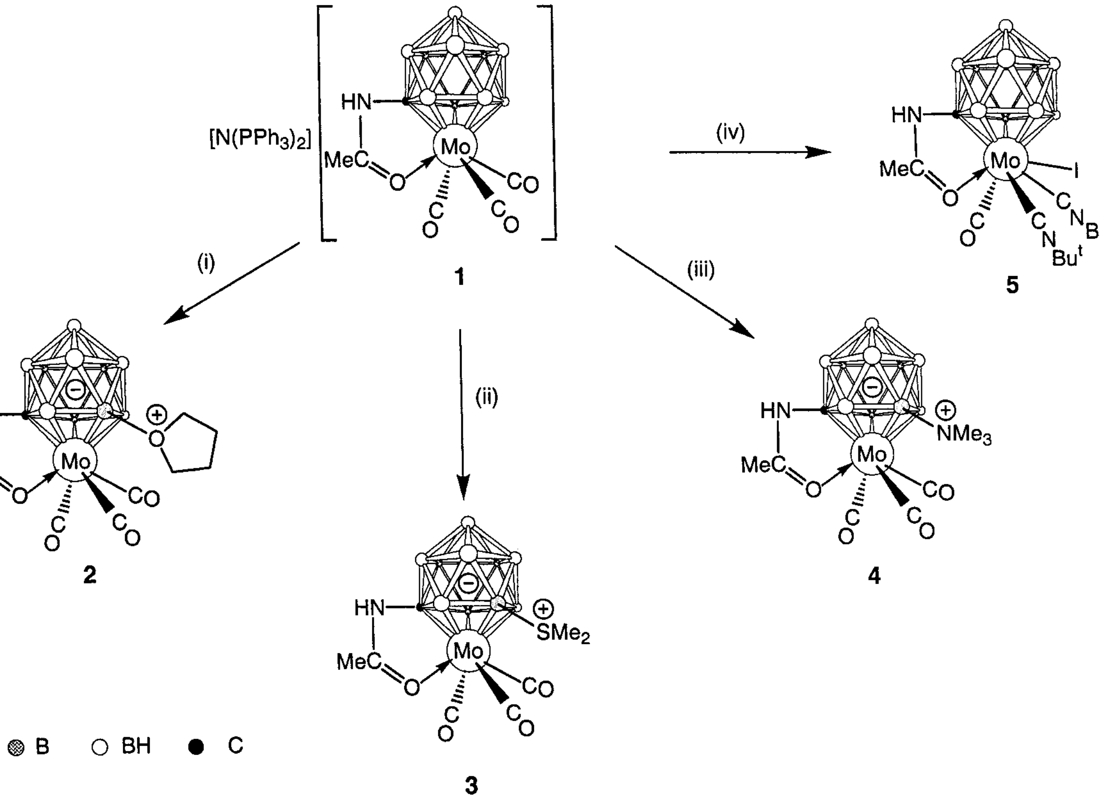
Scheme 1



(i) CF_3SO_3Me in $O(CH_2)_4$-CH_2Cl_2. (ii) H_2SO_4 in Me_2S-CH_2Cl_2.
(iii) $[Me_2N{=}CH_2]Cl$ and $TlPF_6$ in CH_2Cl_2. (iv) I_2 and $CNBu^t$ in CH_2Cl_2.

Reactions of 1 with Electrophilic Reagents. (i) Complex **1** (0.16 g, 0.18 mmol) in CH_2Cl_2−THF (20 mL, 1:1) was stirred with CF_3SO_3Me (20 μL, 0.18 mmol) at ambient temperatures for 3 h. All volatile material was removed in vacuo and the residue dissolved in the minimum amount of CH_2Cl_2 (ca. 3 mL). Column chromatography eluting with CH_2Cl_2−petroleum ether (4:1) afforded a purple fraction. Removal of solvent in vacuo gave purple $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}7\text{-}O(CH_2)_4$-*closo*-$2,1\text{-}MoCB_{10}H_9]$ (**2**) (0.06 g, 76%). IR (CH_2Cl_2): ν_{max} (CO), 2035 s, 1952 s, 1918 m (sh) cm^{-1}. Analytically pure samples were obtained by crystallization from CH_2Cl_2 solutions layered with petroleum ether. Anal. Calcd for $C_{10}H_{21}B_{10}MoNO_5$: C, 27.3; H, 4.8; N, 3.2. Found: C, 27.4; H, 4.9; N, 3.2.

(ii) Complex **1** (0.18 g, 0.20 mmol) in CH_2Cl_2−SMe_2 (15 mL, 2:1) was treated with concentrated sulfuric acid (0.2 mL), and the mixture stirred overnight. All volatile material was removed in vacuo, and the residue dissolved in CH_2Cl_2 (ca. 3 mL) and chromatographed. Elution with CH_2Cl_2−petroleum ether (4:1) afforded a purple eluate from which was obtained $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}7\text{-}SMe_2$-*closo*-$2,1\text{-}MoCB_{10}H_9]$ (**3**) (0.02 g, 23%). IR (CH_2Cl_2): ν_{max} (CO), 2039 s, 1960 s, 1927 m (sh) cm^{-1}. Anal. Calcd for $C_8H_{19}B_{10}MoNO_4S$: C, 22.4; H, 4.5; N, 3.3. Found: C, 22.3; H, 4.6; N, 3.3.

(iii) The reagent $[Me_2N{=}CH_2]Cl$ (0.018 g, 0.19 mmol) in CH_2Cl_2 (20 mL) was stirred with **1** (0.19 g, 0.21 mmol) and $TlPF_6$ (0.074 g, 0.21 mmol) at room temperature (3 h). Volatiles were removed in vacuo, and the residue was taken up in CH_2Cl_2 (ca. 3 mL) and chromatographed. Elution with CH_2Cl_2−petroleum ether (4:1) gave a purple fraction, which after removal of solvent in vacuo afforded $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2,2\text{-}(CO)_3\text{-}7\text{-}NMe_3$-*closo*-$2,1\text{-}MoCB_{10}H_9]$ (**4**) (0.01 g, 12%), crystallized from a CH_2Cl_2 solution layered with petroleum ether. IR (CH_2Cl_2): ν_{max} (CO), 2034 s, 1965 s, 1923 m (sh) cm^{-1}. Anal. Calcd for $C_9H_{22}B_{10}MoN_2O_4\cdot0.5\ CH_2Cl_2$: C, 24.3; H, 5.0; N, 6.0. Found: C, 24.3; H, 5.2; N, 6.2.

Reaction of 1 with I_2 and CNBu^t. A solution of **1** (0.14 g, 0.15 mmol) in CH_2Cl_2 (20 mL) and CNBu^t (67 μL, 0.60 mmol) at −78 °C was treated with I_2 (0.038 g, 0.15 mmol) with vigorous stirring. The Schlenk tube was warmed gently until the first evidence of a color change from purple to red and then immediately returned to the cooling bath. The mixture was then warmed to room temperature (1 h). All volatiles were removed in vacuo, and the residue was taken up in CH_2Cl_2 (ca. 3 mL) and chromatographed. Elution with CH_2Cl_2−petroleum ether (3:1) afforded two fractions, of which the initial purple fraction did not contain any molybdenacarborane species (IR spectroscopy) and was discarded. The second red fraction was collected, and solvent removed in vacuo to give red $[1,2\text{-}\mu\text{-}\{NHC(Me){=}O\}\text{-}2,2\text{-}(CNBu^t)_2\text{-}2\text{-}CO\text{-}2\text{-}I$-*closo*-$2,1\text{-}MoCB_{10}H_{10}]$ (**5**) (0.07 g, 77%), crystallized from a CH_2Cl_2 solution layered with petroleum ether. IR (CH_2Cl_2): ν_{max} 2185 s (C≡N), 2012 m (CO) cm^{-1}. NMR: ^{1}H, δ 6.64 (br, NH); ^{13}C-{^{1}H}, δ 105.4 (br, cage-C), 146.5 (br, CN), 180.2 (C=O), 216.9 (C≡O). Anal. Calcd for $C_{14}H_{32}B_{10}IMoN_3O_2$: C, 27.8; H, 5.3; N, 6.9. Found: C, 27.8; H, 5.4; N, 6.8.

Crystallographic Structural Determination. Crystal data collection and refinement parameters are given in Table 1. Intensity data were measured on an Enraf-Nonius CAD4 diffractometer using Mo Kα X-radiation (λ = 0.71073 Å). Data were corrected for Lorentz, polarization, and absorption affects, the last with a semiempirical method based on azimuthal scans of ψ-data.

The structure was solved by direct methods, and successive difference Fourier syntheses were used to locate all non-hydrogen atoms using SHELXTL version 5.03.[7] Refinements were made by full-matrix least-squares on all F^2 data using

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(7) *SHELXTL*, version 5.03; Bruker AXS: Madison WI, 1995.

Table 1. Crystallographic Summary

chemical formula	$C_{10}H_{21}B_{10}MoNO_5$
M	439.32
cryst syst	monoclinic
space group	$P2_1/n$
a, Å	13.3159(7)
b, Å	9.8420(12)
c, Å	15.000(2)
β, deg	92.100(6)
V, Å^3	1964.6(3)
Z	4
d_{calc}, g cm^{-3}	1.485
T, K	295
μ(Mo Kα), cm^{-1}	6.87
wR2 (all data), R1 [a]	0.0814, 0.0356

[a] $wR2 = [\sum\{w(F_o^2 - F_c^2)^2\}/\sum w(F_o^2)^2]^{1/2}$; $R1 = \sum||F_o| - |F_c||/\sum|F_o|$ with $F_o > 4\sigma(F_o)$.

SHELXL-97.[8] Anisotropic thermal parameters were included for all non-hydrogen atoms. The cage-carbon atom was assigned by comparison of the bond lengths to adjacent boron atoms in conjunction with the magnitudes of the isotropic thermal parameters. The amido hydrogen (H) was located in a difference Fourier map; the coordinates of this hydrogen were allowed to refine with a fixed isotropic thermal parameter, $U_{iso}(H) = 1.2U_{iso}(N)$. All remaining hydrogen atoms were included in calculated positions and allowed to ride on their parent boron or carbon atoms with fixed isotropic thermal parameters [$U_{iso}(H) = 1.2U_{iso}(\text{parent})$ or $U_{iso}(H) = 1.5U_{iso}(C)$ for methyl hydrogens]. The methyl hydrogens on C(6) were refined with an ideally disordered model, that is, with two sets of hydrogens rotated 60° from one another, each with a fixed occupancy of 0.50.

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Supporting Information Available: Atomic fractional coordinates, bond distances and angles, and anisotropic thermal parameters for **2**, as well as a listing of NMR data for new compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

OM020103L

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