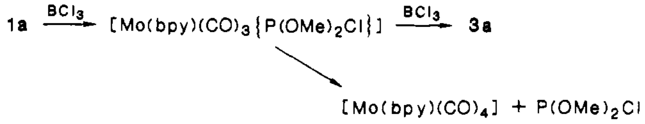
$$fac\text{-}[\mathrm{Mo(bpy)(CO)_3\{P(OMe)_3\}}]\ (\mathbf{1a}) \xrightarrow{2\mathrm{BCl_3}} mer\text{-}[\mathrm{Mo(bpy)(CO)_3\{P(OMe)Cl_3\}}]\ (\mathbf{3a})$$

rearrangement is evidenced by the $^{13}C\{^1H\}$ NMR spectrum.[9] The rearrangement may be due to the strong trans influence of a $P(OMe)Cl_2$ ligand. The reaction of **1a** with 1 equiv of BCl_3 yielded **3a** (25%), $[Mo(bpy)(CO)_4]$ (20%), and free $P(OMe)_2Cl$ (25%). Although the monosubstituted complex $[Mo(bpy)(CO)_3\{P(OMe)_2Cl\}]$ was not obtained, it can be considered that $[Mo(bpy)(CO)_3\{P(OMe)_2Cl\}]$ forms from **1a** and BCl_3, but it is very reactive; thus, some of the molecules react with the BCl_3 still existing in solution to give **3a**, and some of them decompose to afford $[Mo(bpy)(CO)_4]$, releasing free $P(OMe)_2Cl$.



$$\mathbf{1a} \xrightarrow{\mathrm{BCl_3}} [\mathrm{Mo(bpy)(CO)_3\{P(OMe)_2Cl\}}] \xrightarrow{\mathrm{BCl_3}} \mathbf{3a};\quad [\mathrm{Mo(bpy)(CO)_3\{P(OMe)_2Cl\}}] \rightarrow [\mathrm{Mo(bpy)(CO)_4}] + \mathrm{P(OMe)_2Cl}$$

The mechanism of replacement of OR group(s) on the phosphorus ligating to the Mo atom with X is not clear now, but it may involve an ionic intermediate containing a cationic phosphenium ligand or it may be of a concerted nature. Shriver et al. proposed the dihalocarbene complexes as an intermediate in the halogen exchange reaction of transition-metal perfluoroalkyl carbonyl complexes with BX_3 (eq 3).[3] If the electrophilic alkoxide abstraction from phosphorus by BX_3 takes place in the same manner as the halogen abstraction, then the cationic phosphenium complex is a more probable candidate as an intermediate.

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(7) For **3a**: IR (in dichloromethane) ν(CO) 1960, 1880, 1840 cm^{-1}; ^1H NMR (in acetone-d_6) δ 7.60–9.23 (m, 8 H, bpy), 3.85 (d, J = 14.8 Hz, 2 H, OCH_3), 3.62 (d, J = 12.1 Hz, 1 H, OCH_3); $^{31}P\{^1H\}$ NMR δ 146.0 (s). Anal. Calcd for $C_{14}H_{11}Cl_2MoN_2O_4P$: C, 35.83; H, 2.35; N, 5.97; Cl, 15.14. Found: C, 35.94; H, 2.62; N, 5.91; Cl, 15.98.

(8) The reactions of **1b** and **1c** with 2 equiv of BCl_3 produce analogues of **3a**, though these reactions are not so clean.

(9) The $^{13}C\{^1H\}$ NMR spectrum for **3a** exhibits 10 sets of signals in the bpy region, out of which six sets are doublets. In the CO region, three singlets are observed. If the $P(OMe)Cl_2$ ligand rotates freely along the P–Mo bond, then carbonyl carbons cis to the both nitrogen atoms of the coordinated bpy would be magnetically equal. Thus, some interamolecular interaction between one of the carbonyl groups and presumably the OCH_3 group on the phosphorus atom may be expected. This is compatible with the fact that in the ^1H NMR spectrum one methoxy hydrogen is magnetically different from the other two methoxy hydrogens. The $^{13}C\{^1H\}$ NMR spectrum for **1a** containing facial geometry exhibits three singlets and two doublets with small coupling constants in the bpy region and two doublets in the terminal CO region. For **3a**: $^{13}C\{^1H\}$ NMR (in THF-d_8) δ 54.7 (d, J = 20.3 Hz, OCH_3) 122.6 (s, bpy), 123.3 (d, J = 61.8 Hz, bpy), 125.2 (s, bpy), 125.5 (d, J = 27.1 Hz, bpy), 137.4 (s, bpy), 138.4 (d, J = 108.5 Hz, bpy), 152.5 (d, J = 6.8 Hz, bpy), 152.6 (d, J = 13.6 Hz, bpy), 154.4 (d, J = 47.5 Hz, bpy), 154.7 (s, bpy), 219.8 (s, CO), 222.0 (d, J = 20.3 Hz, CO). For **1a**: $^{13}C\{^1H\}$ NMR (in THF-d_8) δ 49.5 (d, J = 1.6 Hz, OCH_3), 121.7 (s, bpy), 124.0 (s, bpy), 135.9 (s, bpy), 152.2 (d, J = 2.1 Hz, bpy), 154.7 (d, J = 1.0 Hz, bpy), 213.7 (d, J = 64.9 Hz, CO), 226.2 (d, J = 14.0 Hz, CO).

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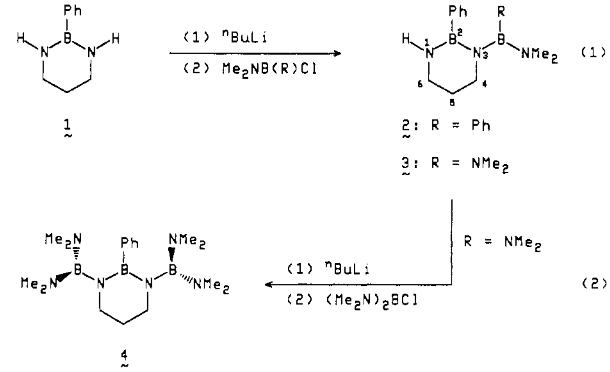
Boron–Nitrogen Polymer Precursors. Synthesis, Structure, and Stereochemistry of *N*-Boryl Derivatives of the 1,3,2-Diazaboracyclohexane Ring System

Sir:

In recent years, there has been an increasing level of interest in the use of small-molecule boron–nitrogen compounds as potential precursors to B–N polymers, BN-based ceramics, and other solid-state materials.[1] The possibility of preparing *linear* B–N polymers [i.e., poly(iminoboranes),[2] $(RBNR)_n$] is especially intriguing since such materials are not only preceramic polymers[3] but are also the isoelectronic analogues of polyacetylene and related organic conductors. In the 1950s and 1960s, the syntheses of numerous amine- and aminoboranes were reported along with many unsuccessful attempts to polymerize such reagents.[4] The high thermal stability of the cyclic trimers [i.e., borazines, $(RBNR)_3$] is generally cited as the reason for the failure of the B–N "monomers" to polymerize. In order to overcome the problem of borazine ring formation, two different approaches, both involving diborylamines as condensation monomers, are being studied in our laboratory. The incorporation of a linear N–B–N–B unit along with other structural features is intended to prevent these systems from condensing to the six-membered borazine rings upon thermolysis. In the first method, acyclic diborylamines containing both Si–N and B–X groups are the starting compounds. A few such species, having the sterically protecting *t*-Bu group on boron, have recently been prepared and characterized.[5]

This study is related to the second synthetic approach which involves linking the nitrogen atoms of the N–B–N–B backbone through bridging $-(CH_2)_n-$ units by use of the 1,3,2-diazaboracycloalkane[6] ring systems (e.g., **1**, eq 1). The bridges are intended to provide structural rigidity in order to prevent the boron–nitrogen backbone from condensing to the cyclic trimer. Specifically, we report here our preliminary findings on the synthesis, structural characterization, and dynamic stereochemistry of some new *N*-boryl derivatives of the 2-phenyl-1,3,2-diazaboracyclohexane ring system **1**.



Synthesis. Typically, the diazaboracyclohexane **1** [100 mmol, in THF (150 mL)] was deprotonated by treatment with an equimolar amount of *n*-BuLi at 0 °C (eq 1). Subsequent addition of the appropriate chloroborane, $Me_2NB(R)Cl$[7] (100 mmol),

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(1) See, for example, the following and references cited therein: (a) Narula, C. K.; Schaeffer, R.; Paine, R. T.; Dayte, A.; Hammetter, W. F. *J. Am. Chem. Soc.* **1987**, *109*, 5556. (b) Narula, C. K.; Paine, R. T.; Schaeffer, R. *Polym. Prepr.* (*Am. Chem. Soc., Div. Polym. Chem.*) **1987**, *28*, 454. (c) Shaw, Y. S.; DuBois, D. A.; Neilson, R. H. *ACS Symp. Ser.*, in press. (d) Paciorek, K. J. L.; Harris, D. H.; Kratzer, R. H. *J. Polym. Sci., Polym. Chem. Ed.* **1986**, *24*, 173. (e) Narula, C. K.; Janil, J. F.; Duesler, E. N.; Paine, R. T.; Schaeffer, R. *Inorg. Chem.* **1986**, *25*, 3346. (f) Komm, R.; Geanangel, R. A.; Liepins, R. *Inorg. Chem.* **1983**, *22*, 1684.

(2) For the synthesis and structures of the *monomeric* and *dimeric* iminoboranes, see: Paetzold, P.; von Plotho, C. *Chem. Ber.* **1982**, *115*, 2819 (and references cited therein).

(3) Wynne, K. J.; Rice, R. W. *Annu. Rev. Mater. Sci.* **1984**, *14*, 297.

(4) For a review, see: Atkinson, I. B.; Currell, B. R. *Inorg. Macromol. Rev.* **1971**, *1*, 203.

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(6) Niedenzu, K.; Fritz, P.; Dawson, J. W. *Inorg. Chem.* **1964**, *8*, 1077.

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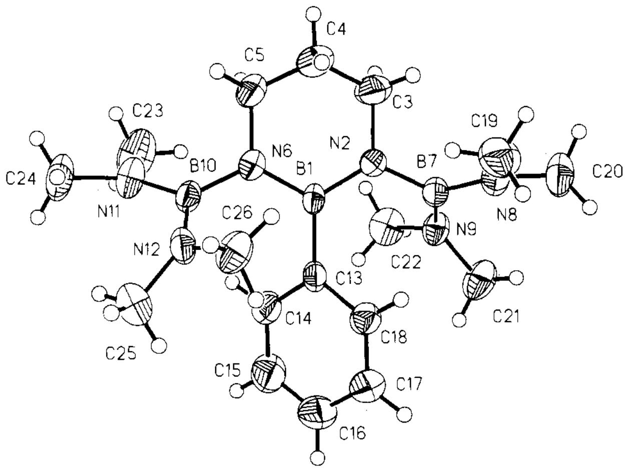

Figure 1. ORTEP drawing of **4**. Thermal elipsoids are drawn at the 25% probability level. Chemically equivalent bonds are statistically equivalent in length. The two ring B–N bonds average 1.428 (5) Å, the two exocyclic B–N bonds average 1.463 (13) Å, and the four B–NMe_2 bonds average 1.418 (8) Å. The eight N–CH_3 distances average 1.456 (9) Å, and B(1)–C(13) = 1.574 (7) Å. The BN_2C_3 ring is folded by 5.4 (7)° along a line through N(2)–N(6); the B(1)B(7)B(10)N(2)N(6)C(13) moiety is planar (0.04 Å rmsd) and makes angles of 57.4 (6) and 56.8 (6)° with the planes B(10)N(11)N(12) and B(7)N(8)N(9), respectively. The H_3C–N–CH_3 groups are twisted by an average of 28.8(1.4)° with respect to the NBN planes, e.g., N(8)B(7)N(9). The phenyl ring makes an angle of 46.7 (8)° with the heterocyclic ring.

followed by filtration and vacuum distillation, gave the diborylamines **2** and **3**. These compounds were obtained in good yields as colorless liquids and were characterized by elemental analyses and NMR spectroscopy (^1H, ^{11}B, and ^{13}C).[8,9] The proton NMR of the starting material **1** shows a well-resolved triplet/quintet pattern for the trimethylene bridge. Upon monosubstitution, however, three distinct multiplets are observed, indicative of the unsymmetrical structures of these derivatives. Also, the nonequivalence of the N–C carbon atoms (C^4 and C^6) is clearly apparent in the ^{13}C NMR spectra of **2** and **3**. The ^{11}B NMR spectrum of **2** consists of two separate signals (ca. δ 32, 36) as expected, while that of **3** is a very broad resonance centered at δ 30 ($\nu_{1/2} \sim 200$ Hz).

Further substitution of the diazaboracyclohexane ring system may be accomplished by a similar reaction sequence. Starting, for example, with compound **3**, the second deprotonation/substitution process (eq 2) gave the *N*,*N'*-bis[bis(dimethylamino)boryl] derivative **4** in 82% yield.[10] Compound **4** is a crystalline solid whose symmetrical structure is consistent with the simplicity

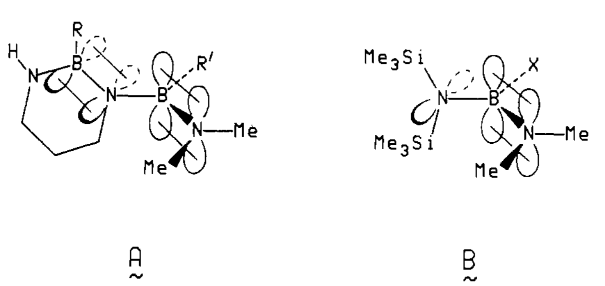

Figure 2. Ground-state conformations (based on variable-temperature NMR studies): (A) *N*-boryl derivatives of 1,3,2-diazaboracyclohexanes (e.g., **2**: *R* = *R'* = Ph); (B) (dimethylamino)[bis(trimethylsilyl)amino]boranes (e.g., X = Cl, Ph, alkyl).[13]

of its NMR spectra relative to those of **2** and **3**.

Structure. The molecular structure of the bis(boryl) derivative **4** was determined by single-crystal X-ray diffraction.[11] This structure (Figure 1) is significant since it contains a *linear backbone* of six B–N bonds and, as such, may serve as a good model for the structure of a linear high polymer. The diazaboracyclohexane ring is only slightly puckered and the phenyl group is rotated out of the plane of the ring by about 47°. The BN framework, defined by B(1)–B(10), is planar as would be expected for sp^2 hybridization of the 3-coordinate B and N atoms. The B–N bond lengths within this skeleton are similar to those of other aminoboranes (e.g., 1,8,10,19-triazaboradecalin[12]). However, the B–N bonds branching from the ring [i.e., N(6)–B(10) and N(2)–B(7)] are longer than those within the ring by 0.02–0.05 Å. Apparently, there is less dative π interaction within these bonds due to the rotation of the $B(NMe_2)_2$ moieties out of the plane of the BN_2C_3 ring by approximately 57°. The H_3C–N–CH_3 groups are twisted by an average of 29° with respect to the NBN planes [e.g., N(8)–B(7)–N(9)].

Stereochemistry. Interestingly, the solid-state structure of **4** is consistent with and provides corroboration for the ground-state structures of the *N*-boryl derivatives **2** and **4**, as determined by variable-temperature ^1H NMR measurements. The ^1H NMR spectra of **2** and **4** revealed a temperature dependence similar to that observed previously for the related compounds $(Me_3Si)_2NB(X)NMe_2$ (X = Cl, Ph, NH_2, alkyl).[13] Thus, at room temperature, the NMe_2 protons of **2** are nonequivalent as a result of restricted rotation about the $BNMe_2$ bond (Figure 2A). Compound **4** exhibits a similar splitting of the NMe_2 signal at lower temperatures. The free energies of activation ($\Delta G^{\ddagger}$),[14] determined by measurement of the coalescence temperatures, were found to be 17.3 and 12.9 kcal/mol, respectively, for **2** and **4**. The

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(7) Chloro(dimethylamino)phenylborane was prepared by the addition of Me_3SiNMe_2 (225 mmol) to a stirred solution of $PhBCl_2$ (225 mmol) in pentane (300 mL) at 0 °C (90% yield). Bis(dimethylamino)chloroborane was prepared via the disproportionation reaction of BCl_3 and $(Me_2N)_3B$ according to the published procedure: Niedenzu, K.; Dawson, J. W. *Boron-Nitrogen Compounds*; Springer-Verlag: West Berlin, 1965. See also: Niedenzu, K.; Dawson, J. W. *J. Am. Chem. Soc.* **1960**, *82*, 4223.

(8) Characterization data for **2** (bp 130–174 °C, 77% yield): Anal. Calcd: C, 70.15; H, 7.97. Found: C, 70.31; H, 8.08. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.92 (q, $^3J_{HH}$ = 5.7 Hz, C^5H_2), 3.02 (t, $^3J_{HH}$ = 5.7 Hz, C^6H_2), 3.27 (m, C^4H_2), 2.24 and 2.52 (s, NMe_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz): δ 28.18 (s, C^5), 40.67 (s, C^6), 45.03 (s, C^4), 40.05 and 40.10 (s, NMe_2). ^{11}B NMR ($CDCl_3$, 96 MHz): δ 31.7 (br, s, B^2), 36.1 [br, s, $B(Ph)NMe_2$].

(9) Characterization data for **3** (bp 88–92 °C, 69% yield): Anal. Calcd: C, 60.49; H, 9.39. Found: C, 60.56; H, 9.56. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.92 (q, $^3J_{HH}$ = 5.6 Hz, C^5H_2), 3.12 (t, $^3J_{HH}$ = 5.2 Hz, C^6H_2), 3.24 (m, C^4H_2), 2.42 (s, NMe_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz): δ 28.20 (s, C^5), 40.85 (s, C^6),43.63 (s, C^4), 39.38 (s, NMe_2). ^{11}B NMR ($CDCl_3$, 96 MHz): δ 29.7 (br, s).

(10) Characterization data for **4** (bp 106–119 °C, mp 85–87 °C, 82% yield): Anal. Calcd: C, 57.37; H, 9.91. Found: C, 58.24; H, 9.91. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.83 (q, $^3J_{HH}$ = 5.5 Hz, C^5H_2), 3.01 (t, $^3J_{HH}$ = 5.4 Hz, $C^{4,6}H_2$), 2.32 (s, NMe_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz): δ 28.66 (s, C^5), 44.09 (s, $C^{4,6}$), 39.28 (s, NMe_2). ^{11}B NMR ($CDCl_3$, 96 MHz): δ 29.3 (br, s, B^2), 31.0 [br, s, $B(NMe_2)$].

(11) Crystal of dimensions 0.3 × 0.4 × 0.5 mm, triclinic space group $P1$, with a = 10.986 (2) Å, b = 11.077 (3) Å, c = 11.396 (4) Å, α = 67.57 (2)°, β = 61.30 (2)°, γ = 76.96 (2)°, V = 1122.9 (6) $Å^3$, Z = 2, D(calcd) = 1.05 g cm^{-3}, $F(000)$ = 388, λ(Mo Kα) = 0.710 73 Å, μ = 0.7 cm^{-1}. A total of 2257 independent reflections were collected ($3 \leq 2\theta \leq 45°$) on a Nicolet R3M/μ update of a $P2_1$ diffractometer; 1391 were observed [$I \geq 3\sigma(I)$] (Lorentz, polarization, and Ψ-scan empirical corrections applied). The structure was solved by direct methods. Anisotropic block-cascade, least-squares refinement (H atoms allowed to ride on carbon atoms with fixed isotropic thermal parameters) led to R = 0.0776, R_w = 0.0727, S = 1.675, $(\Delta/\sigma)_{max}$ = 0.08. Largest peaks in the final difference map of +0.20 e $Å^{-3}$. $\sum w(|F_o| - |F_c|)^2$ minimized with w = $[\sigma^2(F_o) + 0.00057F_o^2]^{-1}$. Atomic scattering factors and anomalous dispersion corrections were taken from: *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1974; Vol. IV. Atomic coordinates, thermal parameters, bond lengths, and valence angles are listed in Supplementary Tables 1–5. Lists of structure factors are also available.

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(14) The equation $\Delta G^{\ddagger} = T_c[45.67 + 4.58 \log (T_c/\Delta\nu)]$ gives $\Delta G^{\ddagger}$ in cal/mol with T_c in K and $\Delta\nu$ in Hz (see ref 13c for further discussion of this method of calculating $\Delta G^{\ddagger}$ values). Variable-temperature ^1H NMR (300 MHz) data: for **2**, C_6D_6, T_c = 78 °C, $\Delta\nu$ = 60.5 Hz (30 °C); for **4**, $CDCl_3$, T_c = −17 °C, $\Delta\nu$ = 24.6 Hz (−55 °C). The estimated experimental uncertainty in $\Delta G^{\ddagger}$ is ~0.3 kcal/mol.

value observed for **2** is, in fact, typical of aminoboranes in which only one nitrogen is oriented properly for π-bonding to boron[13] {e.g., [bis(trimethylsilyl)amino]boranes, Figure 2B}. The $\Delta G^{\ddagger}$ values indicate a substantial degree of (p–p)π interaction in the B–NMe_2 bond(s) and show that the ground-state configurations of **2** and **4** in solution are probably the same as the structure that is adopted in the solid state of **4**. The B–NMe_2 rotational barrier of **4** is significantly lower than that of **2**, indicative of a lowering of the B–N π bond order due to competition of two nitrogen electron pairs for the empty p orbital of boron.[13]

More detailed studies of the preparative chemistry, molecular structures, and thermal condensation reactions of the *N*-boryl derivatives of the 1,3,2-diazaboracycloalkane ring systems are currently in progress.

Acknowledgment. We thank the U.S. Office of Naval Research, The Robert A. Welch Foundation (Grants P-759 and P-074), and the TCU Research Fund for financial support.

Supplementary Material Available: Tables of atomic positional parameters and U_{eq} values, hydrogen atom positions and isotropic U values, anisotropic thermal parameters, interatomic distances, and valence angles (4 pages); a table of observed and calculated structure factors (14 pages). Ordering information is given on any current masthead page.

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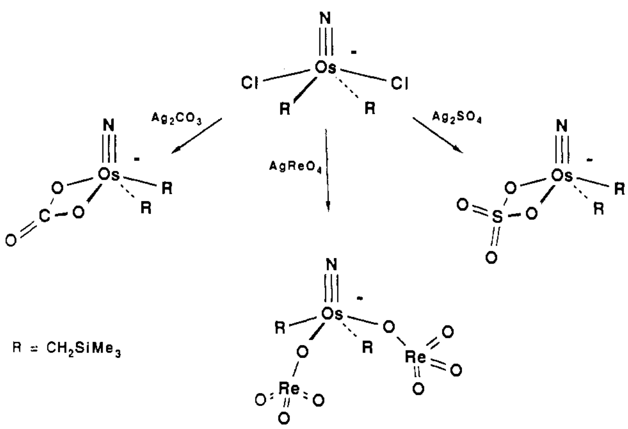
Halide Substitution Reactions of *trans*-$[Os(N)(CH_2SiMe_3)_2Cl_2]^-$. Synthesis of Carbonate, Sulfate, and Perrhenate Complexes of Osmium(VI)

Sir:

Complexes of ruthenium and osmium(VI) have been studied recently as oxidation reagents or catalysts.[1-3] Certain transition-metal oxyanions, MnO_4^-, for example, are strong oxidants toward both organic and inorganic molecules.[4] Coordination of oxyanions to coordinatively unsaturated osmium(VI) or ruthenium(VI) centers could lead to the development of new, bimetallic oxidation catalysts. Transition-metal complexes of the sulfate, carbonate, and perrhenate oxyanions are well-known in inorganic chemistry. Coordination compounds containing monodentate, bidentate, and tridentate sulfate and carbonate groups have been characterized.[5] An osmium cluster complex containing a tridentate sulfate ligand has been reported.[6] Although organometallic complexes of these ligands are rare, (pentamethylcyclopentadienyl)rhenium(V) complexes with bidentate sulfate and carbonate, $(C_5Me_5)Re(O)(CO_3)$ and $(C_5Me_5)Re(O)(SO_4)$, have recently been reported.[7] A copper complex of perrhenate and several rhenium complexes of perrhenate have been prepared and structurally characterized.[8-11]

Scheme I



Tetra-*n*-butylammonium or tetraphenylphosphonium salts of the coordinatively unsaturated nitridooxmium(VI) and nitridoruthenium(VI) dialkyl dichloride complexes, $[M(N)R_2Cl_2]^-$ (M = Ru, Os; R = Me, CH_2SiMe_3), have been prepared by reaction of $[M(N)R_4]^-$ with HCl.[12,13] Substitution of the halides in $[M(N)R_2Cl_2]^-$ for other anionic ligands provides a route to a wide variety of high-oxidation-state organometallic complexes. The preparation of osmium(IV) complexes containing sulfur donor ligands has recently been reported.[14] Thiols, such as 1,2-ethanedithiol, readily displace halides at osmium to form thiolato complexes in which the thiolato ligands are strong σ donors to the metal. Surprisingly, the oxygen analogue, ethylene glycol, does not react under the same conditions.

We were interested in the preparation of nitridoosmium dialkyl complexes with oxygen-containing ligands for two reasons. We wanted to see how they affect the electron density at the metal center and the π-bonding interaction between the nitrogen and the osmium or ruthenium centers. We were also interested in organometallic osmium and ruthenium complexes with oxygen-containing ligands in connection with our goal of preparing high-oxidation-state heterobimetallic oxidation catalysts. There should be similarities in the structures of the complexes and properties of the M–O bond between osmium complexes of the tetrahedral sulfate and chromate dianions and between complexes of the tetrahedral perrhenate and permanganate monoanions. Sulfate and perrhenate are much less oxidizing than chromate and permanganate, so complexes of these should be more stable and easier to study.

The reaction of *trans*-$[N\text{-}n\text{-}Bu_4][Os(N)(CH_2SiMe_3)_2Cl_2]$ with silver sulfate in methylene chloride produced *cis*-$[N\text{-}n\text{-}Bu_4][Os(N)(CH_2SiMe_3)_2(SO_4)]$ (**1**) and precipitates of silver chloride. In a typical preparation, a solution of *trans*-$[N\text{-}n\text{-}Bu_4][Os(N)(CH_2SiMe_3)_2Cl_2]$ (0.090 g, 0.13 mmol) in 5mL of CH_2Cl_2 was added dropwise at room temperature to a suspension of Ag_2SO_4 (0.08 g, 0.26 mmol) in 50 mL of CH_2Cl_2. After the mixture was stirred for 12 h in the light (fluorescent lamp), AgCl was removed by filtration. The solvent was removed from the filtrate under vacuum, and the residue was crystallized from hexane/diethyl ether at −30 °C. Yellow crystals of **1** (0.089 g, 97%) were collected.[15] A similar reaction between *trans*-$[N\text{-}n\text{-}Bu_4][Os(N)$-

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(15) IR (KBr pellet, cm^{-1}): Os≡N obscured; 1119 vs, br (S=O). ^1H NMR (C_6D_6, 200 MHz, 293 K): δ 3.62 (d, 1 H, $OsCH^a$), 2.83 (m, 4 H, NCH_2), 2.42 (d, 1 H, $OsCH^b$), 1.30 (m, 8 H, $NCH_2CH_2CH_2CH_3$), 0.96 (t, 6 H, $NCH_2CH_2CH_2CH_3$), 0.41 (s, 9 H, $OsCH_2Si(CH_3)_3$. $^{13}C\{^1H\}$ NMR (C_6D_6, 50.3 MHz, 295 K): δ 58.5 (NCH_2), 23.9 ($NCH_2CH_2CH_2CH_3$), 20.0 ($NCH_2CH_2CH_2CH_3$), 13.9 ($NCH_2CH_2CH_2CH_3$), 8.7 ($OsCH_2Si(CH_3)_3$), 0.6 ($OsCH_2Si(CH_3)_3$). Anal. Calcd for $OsSN_2Si_2O_4C_{24}H_{58}$: C, 40.19; H, 8.09; N, 3.90. Found: C, 40.38; H, 8.04; N, 3.70. MS: anion, m/z 476, 18%; cation, m/z 242, 100%.

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