$(OC_2H_4OMe)_6]_\infty$,[13] respectively. The triply bridging chelating coordination mode has been found in the tetranuclear cluster $Ba_2Cu_2(OC_2H_4OMe)_4(acac)_4 \cdot 2OHC_2H_4OMe$.[5] The Y–O alkoxide bond distances are significantly longer for the μ_3 moieties than for those of μ_2 (2.44 (2)–2.50 (2) Å vs 2.28 Å av), and longer than those observed for terminal methylmethoxide groups.[4] The Y–O bond lengths involving the ether functionality are quite long (2.52 (2)–2.57 (2) Å). The μ_3-oxygen atoms are clearly pyramidal (sum of the Y–O–Y angles is 285.3° average), and located at 1.230 and 1.287 Å above and below the Y_3 planes, respectively. The Y–O(acac) distances (2.25 (2)–2.35 (2) Å) are comparable to those reported in the literature.[14] These results show the preference of the 2-methoxyethoxide groups over the acetylacetonate ones to act as bridging ligands.

$Y_3(\mu_3,\eta^2\text{-}OC_2H_4OMe)_2(\mu_2,\eta^2\text{-}OC_2H_4OMe)_2(\mu_2,\eta^1\text{-}OC_2H_4OMe)(acac)_4$ (**2**) displays a complex behavior in solution, and several molecular species are observed in $CDCl_3$ solution for instance, as evidenced by ^1H and ^{13}C NMR. Several hypotheses such as a nonchelating behavior of some μ_3 or μ_2 groups could account for the spectroscopic observations, but detailed analysis has not been carried out. By contrast to $Y_2(OAc)_2(acac)_4(H_2O)_2$,[14] no free acetylactone was detected in solution.

The nature of the isolated yttrium and copper products appears to be independent of the stoichiometry of the reaction and/or the solvents used (toluene, pentane, additional methoxyethanol). While the method explored here yielded no isolable mixed-metal Y/Cu compounds, the intermediacy of such a derivative(s) is implicit in the synthetic reaction (eq 1). Indeed, no reaction proceeds between $Cu(acac)_2$ and anhydrous 2-methoxyethanol under similar conditions. These results also suggest that ligand metathesis can be a complicating factor in building multimetallic systems.

Direct reaction between **1** and acacH (1:9 molar ratio) in toluene offers an alternative route to **2**. This novel yttrium precursor displays a volatility and a thermal stability (as established by thermal gravimetric analysis) comparable to that of $[Y(acac)_3]_m$, although the presence of the functional alkoxide ligands reduces its sensitivity to moisture. The copper acetylacetonate alkoxide **3** is highly soluble as well as hydrolyzable, while the homoleptic Cu(II) parent alkoxide $[Cu(OC_2H_4OMe)_2]_m$ is polymeric and insoluble.[15] It is therefore an attractive precursor for sol–gel applications, and further experiments are in progress.

Acknowledgment. This work was supported by the ARC "CVD Supraconducteurs" PIRMAT-CNRS.

Supplementary Material Available: Tables of a full summary of the crystallographic data, positional and isotropic thermal parameters, anisotropic thermal parameters, positional parameters for hydrogen atoms, and bond lengths and angles (7 pages); a listing of observed and calculated structure factors (6 pages). Ordering information is given on any current masthead page.

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Synthesis, Structure, and Reactivity of the Nitrosyl Ligand/Lewis Acid Adduct $(\eta^5\text{-}C_5H_5)Re(NO \cdot BCl_3)(PPh_3)(SiMe_2Cl)$

Lewis acids form a rich array of adducts with carbonyl ligands of transition-metal complexes.[1] Some are isolable, whereas others can only be detected spectroscopically. Such interactions catalyze or promote a variety of transformations.[1] However, less is known about nitrosyl ligand/Lewis acid adducts.[2-4] Some have been characterized in solution by IR spectroscopy,[3] and Legzdins has recently isolated $AlMe_3$ and MgI_2 complexes of $(\eta^5\text{-}C_5H_5)W(R)_2(NO)$ in analytically pure form.[4] Nitrosyl ligand/Lewis acid interactions have also been shown to play a key role in the catalytic oxidation of alcohols by a cobalt nitro/nitrosyl couple.[5] However, structurally characterized adducts have not to our knowledge been described. In this communication, we report the synthesis, crystal structure, and reactions of the nitrosyl ligand/BCl_3 adduct $(\eta^5\text{-}C_5H_5)Re(NO \cdot BCl_3)(PPh_3)(SiMe_2Cl)$ (**1**).

The previously reported functionalized silyl complex $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(SiMe_2H)$ (**2**; ν_{NO} 1636 cm^{-1} (KBr))[6] was dissolved in $CHCl_3$ at 50 °C (Scheme I). Workup gave the chlorosilyl complex $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(SiMe_2Cl)$ (**3**)[7] as orange needles (84%). Complex **3** exhibited a strong IR ν_{NO} at 1656–1657 cm^{-1} (CH_2Cl_2, KBr)—somewhat low for a linear nitrosyl ligand,[2] but typical for neutral $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(X)$ compounds.[8]

In connection with another synthetic objective, a CD_2Cl_2 solution of **3** was treated with BCl_3 (1.1 equiv, 1.0 M in CH_2Cl_2) at −78 °C. NMR spectra (−72 °C) showed downfield shifts of the cyclopentadienyl and methyl ^1H resonances and an upfield shift of the PPh_3 ^{31}P resonance.[9] A ^{11}B NMR spectrum showed a broad peak (7.1 ppm vs $BF_3 \cdot OEt_2$) that was upfield of BCl_3 and in a range characteristic of Lewis base/BCl_3 adducts.[10]

A second equivalent of BCl_3 was added to the **3**/BCl_3 mixture. Extremely air sensitive, orange microcrystals precipitated. In a separate experiment, a CH_2Cl_2 solution of **3** and BCl_3 (2 equiv) was kept at −25 °C. Orange cubes formed (72%).[11] NMR

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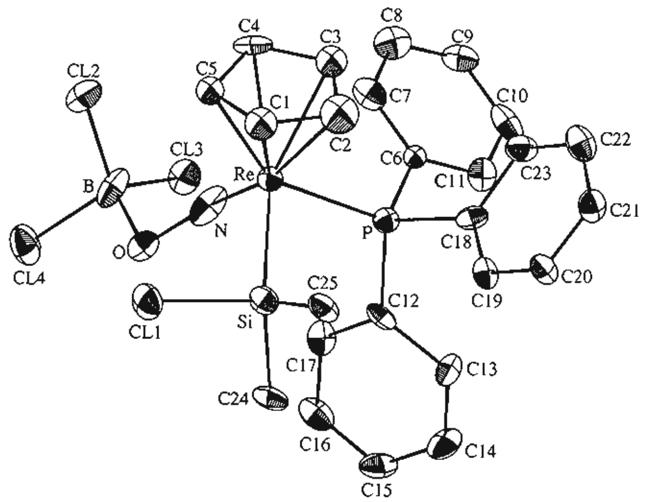
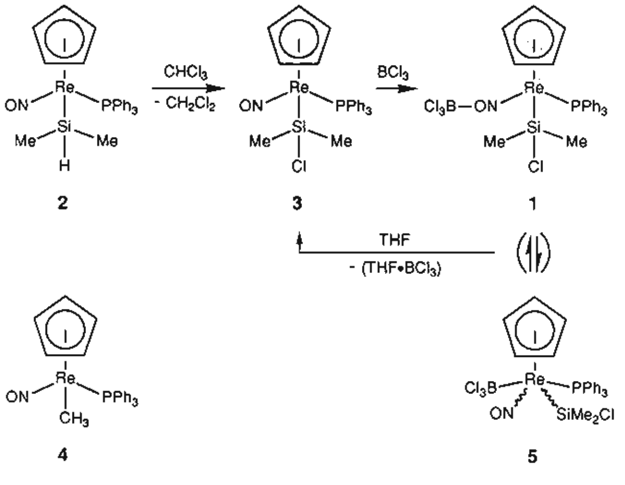


Figure 1. Structure of **1**·CH_2Cl_2. Key bond lengths (Å) and angles (deg): Re–N 1.568 (9), N–O 1.47 (1), O–B 1.53 (1), B–Cl2 1.78 (1), B–Cl3 1.77 (2), B–Cl4 1.88 (2), Re–Si 2.476 (3), Si–Cl1 2.113 (4), Si–C24 1.87 (1), Si–C25 1.86 (1), Re–P 2.382 (3); N–Re–P 97.4 (3), P–Re–Si 94.6 (1), N–Re–Si 97.4 (4), Re–N–O 163.7 (8), N–O–B 106.8 (8), O–B–Cl2 110.5 (9), O–B–Cl3 109.9 (8), O–B–Cl4 100 (1), Re–Si–Cl1 104.5 (2), Re–Si–C24 115.6 (3), Re–Si–C25 120.8 (3).

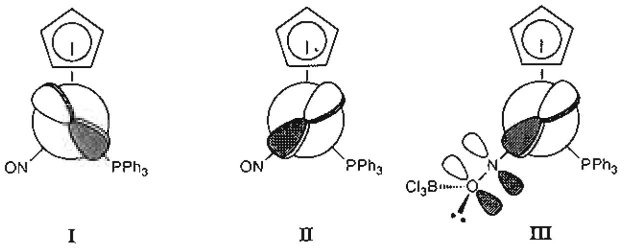
Scheme I. Reactions of Functionalized Silyl Complexes



spectra were recorded in CD_2Cl_2 and closely matched those obtained above. IR spectra were acquired in CH_2Cl_2 and KBr and showed an extremely low ν_{NO} (1395 cm^{-1}). In both IR media, a second, less intense ν_{NO} (1759 cm^{-1}) was also observed.

X-ray data were collected on the orange cubes as described in the supplementary material. Refinement showed the product to be a CH_2Cl_2 monosolvate of the nitrosyl ligand/BCl_3 adduct $(\eta^5\text{-}C_5H_5)Re(NO\cdot BCl_3)(PPh_3)(SiMe_2Cl)$ (**1**). The structure (Figure 1) exhibited several interesting features. First, the Re–N bond (1.57 Å) was distinctly shorter than those in other neutral $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(X)$ complexes (1.78–1.74 Å, average 1.75 Å),[8] whereas the N–O bond (1.47 Å) was longer (1.22–1.19 Å, average 1.21 Å). Second, the N–O–B linkage was sharply bent (107°), and an oxygen lone-pair position was calculated on the basis of an idealized trigonal geometry. Third, the Re–N–O linkage was slightly bent (164°) relative to those in other $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(X)$ complexes but still in the range of linear nitrosyl ligands.[2]

The highest occupied molecular orbitals of the $[(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)]^+$ fragment have been shown to be I (HOMO) and II (SHOMO).[12] SHOMO II is of appropriate symmetry



to back-bond into the nitrosyl ligand, and its plane in **1** can be approximated by the Si/Re/N/O least-squares plane. As would be expected for a structural model with a formal N=O double bond and sp^2-hybridized oxygen (III), the Si/Re/N/O and B/O/lone pair planes are essentially coplanar (∠7°). Finally, the P–Re–Si–Cl1 and N–Re–Si–Cl1 torsion angles are 162.0 (3) and 63.9 (8)°. This places the Si–Cl bond nearly in the plane of HOMO I.

We sought to extend the preceding chemistry. First, CD_2Cl_2 solutions of methyl complex $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(CH_3)$ (**4**; ν_{NO} 1623 cm^{-1}) were treated with BCl_3 or $AlCl_3$ (1 equiv) at −78 °C. NMR spectra resembled those of **3**/BCl_3.[13] The IR spectrum of **4**/BCl_3 showed ν_{NO} at 1392 (major) and 1752 cm^{-1} (minor). Five-coordinate rhenium complexes $[(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(H)(R)]^+X^-$ exhibit IR ν_{NO} of 1750–1769 cm^{-1},[14] and the binding of Lewis acids to the *metal* in nitrosyl complexes should *increase* ν_{NO}.[3] Accordingly, when **3**/BCl_3 and **4**/BCl_3 were treated with the Lewis base THF (2 equiv), the 1392–1395- and 1752–1759-cm^{-1} ν_{NO} were replaced by those of precursors **3** and **4**. Thus, we propose that **1** equilibrates with the Re/BCl_3 tautomer $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(SiMe_2Cl)(BCl_3)$ (**5**; ν_{NO} 1759 cm^{-1}) in CH_2Cl_2 and that methyl complex **4** forms analogous adducts.

Several earlier literature reports deserve emphasis. First, Legzdins has crystallized 2:1 nitrosyl ligand/MgI_2 adducts $[(\eta^5\text{-}C_5H_5)M(NO)(R)_2]_2MgI_2\cdot OEt_2$ (M = Mo, W) from Grignard displacement reactions.[4a] Although disorder precluded complete structural characterization, the N–O bonds (1.20 Å, M = W, R = CH_2SiMe_3) were not significantly lengthened, and IR ν_{NO} were only 35–70 cm^{-1} lower than those of uncomplexed analogues. Second, when nitrosyl complexes $(\eta^5\text{-}C_5H_5)M(NO)(PPh_3)(CO)$ (M = Mo, W; ν_{NO} 1606–1595 cm^{-1}) were treated with $AlCl_3$ in CH_2Cl_2, IR ν_{NO} similar to those of **1** and **5** appeared.[3b] Although no products were isolated, analogous structures were proposed. Third, hydride transfer from alkyl complexes $(\eta^5\text{-}C_5H_5)Re(NO)(PPh_3)(R)$ to Ph_3C^+ has been shown to involve initial electron transfer.[15] Complex **1** provides an attractive model for a possible inner-sphere redox mechanism. Finally, there have been many attempts to prepare cationic silylene complexes $[L_nM{=}SiR_2]^+$ by the abstraction of leaving groups from functionalized silyl complexes $L_nM{-}SiR_2X$.[14b,16] Equilibria of the types shown in Scheme I should serve to diminish the reactivity of Lewis acidic abstracting reagents.

Acknowledgment. We thank the NSF for support of this research.

Supplementary Material Available: A discussion of the crystal structure and tables of crystallographic data, atomic coordinates, bond lengths, bond and torsion angles, and anisotropic thermal parameters for **1**·CH_2Cl_2 (9 pages); a table of calculated and observed structure factors

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(11) (a) Crystallization is considerably slower if only 1.5 equiv of BCl_3 is present. (b) When microanalyses of these crystals are attempted, values that are intermediate between those calculated for **1** (C, 39.80; H, 3.47; Cl, 18.80) and **1**·CH_2Cl_2 (C, 37.21; H, 3.36; Cl, 25.34) are found: C, 38.41; H, 3.47; Cl, 18.61.

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(13) NMR data for **4**/BCl_3 (CD_2Cl_2, −72 °C): ^1H (δ) 5.28 (s, C_5H_5), 1.77 (d, CH_3); ^{31}P (ppm) 13.8 (s); ^{11}B (ppm) 6.8 (br, s).

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for **1**·CH_2Cl_2 (9 pages). Ordering information is given on any current masthead page.

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Structure and Magnetic Moment of Pyrrolidinium Copper Trichloride, a Linear Ferromagnet

Pyrrolidinium copper trichloride, $(C_4H_8NH_2)CuCl_3$, was synthesized in an attempt to find another example of a spin $^1/_2$, Heisenberg, linear ferromagnetic copper salt. The pyrrolidinium salt was chosen for its expected similarities to the well-characterized linear-chain compound cyclohexylammonium copper trichloride (CHAC).[1] The latter contains chains of square-pyramidal $CuCl_5$ polyhedra that share basal plane edges. A folding of the chains along these shared edges leads to bridging Cu–Cl–Cu angles near 85°, a value favoring ferromagnetic coupling.[2] The $-NH_3$ moiety of the cyclohexylammonium ion hydrogen bonds to axial chloride ions in adjacent chains, both providing stability to the structure and stabilizing the square-pyramidal coordination geometry. The possibility of the $-NH_2$ portion of the pyrrolidinium cation performing the same function led to the present investigation. The structure of $(C_4H_8NH_2)CuCl_3$ was thus determined, and its powder magnetic susceptibility was examined with the result being indeed a ferromagnetic chain but one with much stronger antiferromagnetic interchain coupling than was expected.

The compound was prepared by mixing solutions of hydrated $CuCl_2$ with solutions of pyrrolidine in dilute HCl. The HCl was always in excess because this seems to help with crystal growth in many of these compounds. Several preparations were done and quantities varied from about 6 to about 15 mmol of $CuCl_2$; the quantity of pyrrolidine was always at least equivalent to the amount of $CuCl_2$, and in some cases it was doubled to see if we could form a tetrachlorocuprate (the title compound was the only product observed in any of the preparations). The molar excess of HCl was always about a factor of 2 larger than that required to completely protonate the amine. The components were each dissolved in the minimum amount of water and mixed; the resultant solution was filtered to remove dust and other nucleation centers and set to evaporate slowly in a petri dish. Crystals were collected on a suction filter, washed sparingly with ice cold ethanol and then with ethyl ether, and air-dried. Note that air drying is appropriate in the dry Montana climate but may not be successful when the humidity is high!

A crystal that was a dark red hexagonal prism 0.12-mm thick by 0.48-mm long was mounted on a glass fiber for data collection. Axial photographs showed monoclinic symmetry. Unit cell dimensions were obtained by least-squares refinement using 20 centered reflections for which $27° < 2\theta < 33°$ (graphite-monochromatized Mo Kα radiation). Data collection was carried out on a Nicolet R3m/E four-circle diffractometer.[3] Three check reflections were monitored every 93 reflections, showing no loss of intensity during the course of data collection. Tables of crystal and refinement data, observed and calculated structure factors, anisotropic thermal parameters, and hydrogen positions are given in the supplementary material (vide infra).

Table I. Crystallographic Data for $(C_4H_8NH_2)CuCl_3$

a	9.3456 (12) Å	fw	242.03
b	6.5097 (7) Å	space group	$P2_1/n$ (No. 14)
c	14.1307 (13) Å	radiation, λ	Mo Kα, 0.71069 Å
β	102.93 (1)°	D_{calc}	1.92 g/cm^3
V_{cell}	837.9	abs coeff	35.0 cm^{-1}
Z	4	R^a	0.0412
T	24 °C	$R_w{}^b$	0.0429

$^a R = \sum(|F_o - F_c|)/\sum|F_o|$. $^b R_w = \sum\{w(|F_o| - |F_c|)^2\}/\sum(w|F_o|^2)$.

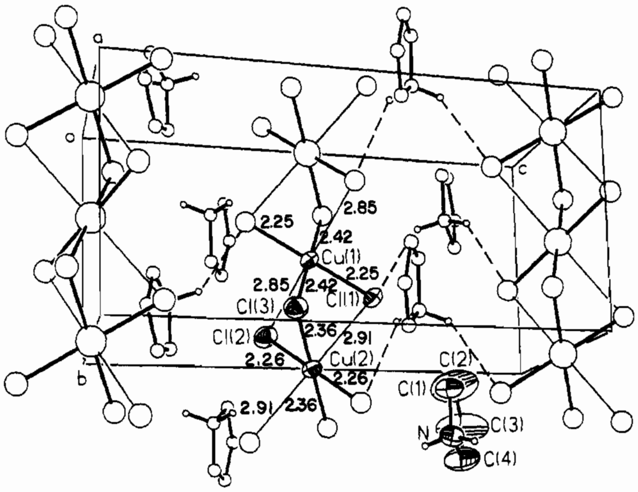

Figure 1. Unit cell, showing the chain structure and probable hydrogen bonding. Bond lengths are given in angstroms.

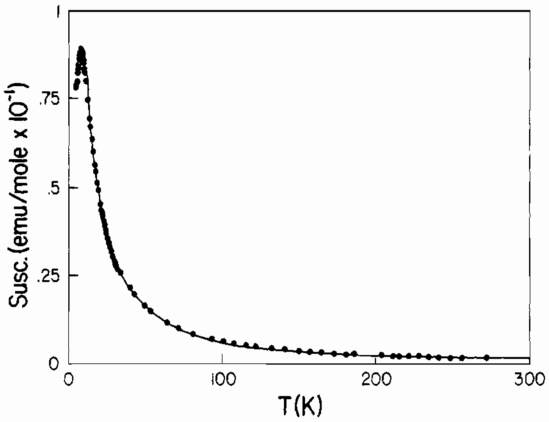

Figure 2. Powder magnetization data (circles) and high-temperature series expansion fit (solid line) with intrachain $J/k = +56$ K and interchain $J'/k = -1.2$ K.

Data reduction,[3] including corrections for Lorentz and polarization effects, gave 2448 independent reflections in the range $3° < 2\theta < 60°$, of which 1264 with $I > 3\sigma(I)$ were used for structure refinement. A brief summary of unit cell and refinement data is given in Table I; a more complete listing is given in the supplementary material. A Patterson synthesis gave the copper positions, and the remaining non-hydrogen atoms were located by F_o–F_c difference maps. These positions were refined with anisotropic thermal parameters by blocked-cascade least squares, minimizing $w\Delta^2$, with all 85 parameters refined in each cycle. Not all hydrogen atom positions could be located reliably in the final difference map, and the final cycles of refinement were done with a riding model in which the hydrogens could move about the attached atom but were fixed at 0.96 Å from each carbon and 0.93 Å from each nitrogen. Atomic scattering factors were taken from Cromer and Waber.[4] Absorption factors were calculated by Gaussian integration using measured distances between indexed crystal faces. Maximum and minimum electron densities in the final difference map were 0.71 and −0.79 e/Å^3, respectively. Refined atomic coordinates are given in Table S2 (supplementary

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