Table I. IR Spectra of Digallane in the Vapor Phase at ca. 270 K and Isolated in Solid Ar or N_2 Matrices at ca. 20 K

vapor: digallane-h_6		vapor: digallane-d_6[b]			digallane-h_6 trapped in an Ar matrix		digallane-h_6 trapped in an N_2 matrix		
$\tilde{\nu}$/cm^{-1}	intens[a]	$\tilde{\nu}$/cm^{-1}	intens[a]	$\tilde{\nu}_H/\tilde{\nu}_D$	$\tilde{\nu}$/cm^{-1}	intens[a]	$\tilde{\nu}$/cm^{-1}	intens[a]	assignment[a]
					2015	m } *c*			
1998	m	1446	m }	1.3817	1996	m	2000	m	ν_8 (b_{1u}), ν(Ga-H_t)
		1434	m, sh						
1981	R } m	1421	R } m	1.3956[d]	1985	s } *c*	1985	m	ν_{16} (b_{3u}), ν(Ga-H_t)
1970	P	1410	P		1968	m			
					1283	s } *c*	1282	m } *c*	
1278	R } s				1278	m			
1267	Q	914	Q s	1.3862[d]	1253	m	1258	w	ν_{13} (b_{2u}), ν(Ga-H_b)
					1234	w	1243	w	
					1221	s } *c*			
1205	R } m	865	R } m	1.3953[d]	1218	sh			
1195	P	855	P		1213	m	1220	s	ν_{17} (b_{3u}), ν(Ga-H_b)
					1208	m			
					1195	m			
1046	vw				1080	vw	1075	vw	combination
		555	R } m	1.3945[d]	773	m } *c*			
760	Q w	545	Q		761	m	770	m	ν_{14} (b_{2u}), ρ(GaH_2)
700	sh				695	w			combination
676	R } vs	488	R } vs	1.3878[d]	676	vs } *c*	673	vs	ν_{18} (b_{3u}), δ(GaH_2)
666	P	479	P		666	s			
					659	m } *c*			
	e	439	mw		655	m	655	s } *c*	ν_9 (b_{1u}), ρ(GaH_2)[f]
					653	m	647	w	
					648	m			

[a] s strong, m medium, w weak, v very, sh shoulder, t terminal, b bridging. [b] The IR spectrum of this sample also included a number of weak absorptions attributable to $Ga_2H_nD_{6-n}$ (n = 1, 2...) or impurities containing both H and D. [c] Matrix splitting. [d] Product rule calculations give for b_{2u} fundamentals $P_{obsd} = \nu_{13}(H)\cdot\nu_{14}(H)/\nu_{13}(D)\cdot\nu_{14}(D) = 1.9331$ vs $P_{calcd} = 1.9581$ and for b_{3u} fundamentals $P_{obsd} = \nu_{16}(H)\cdot\nu_{17}(H)\cdot\nu_{18}(H)/\nu_{16}(D)\cdot\nu_{17}(D)\cdot\nu_{18}(D) = 2.7026$ vs $P_{calcd} = 2.7681$. The deviations of 1.3 and 2.4%, respectively, fall in the range customarily found when observed (anharmonic) vibration frequency data are used. [e] Too weak to be observed. [f] Tentative assignment.

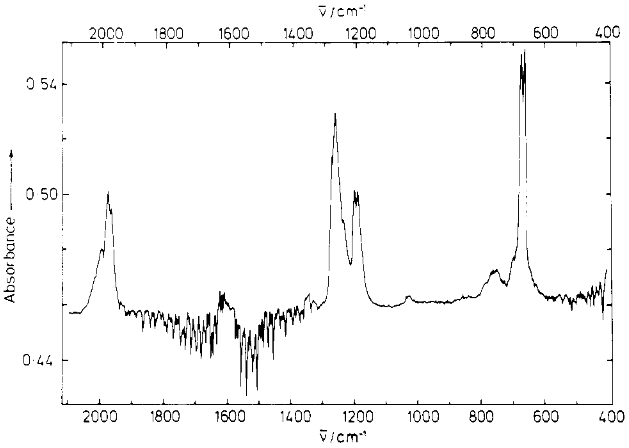

Figure 1. FT-IR spectrum of gallane vapor in a Pyrex glass cell with a pathlength of 10 cm, fitted with CsI windows and cooled to ca. 270 K; the record corresponds to the difference in absorbance between the initial, partially decomposed and final, fully decomposed sample (with appropriate scaling).

(c) ^{1}H NMR Spectrum. The ^{1}H NMR spectrum of the gallane dissolved in $C_6D_5CD_3$ at −65 °C consisted of two singlets at δ 4.41 and 1.11 with relative intensities 2:1, both showing the broadness characteristic of protons directly bound to gallium atoms. This too is consistent with the structure **Ib**. Coalescence to a single very broad resonance occurred when the sample was warmed to −30 °C; decomposition set in at ca. 0 °C with the appearance of gallium metal.

(d) Chemical Trapping with Trimethylamine. The gallane reacted with an excess of trimethylamine at −95 °C. At temperatures below −30 °C, there was *but a single product* identified by its IR, Raman, and ^{1}H NMR spectra as the adduct $(Me_3N)_2GaH_3$;[10] this dissociated at ambient temperatures to trimethylamine and $Me_3N\cdot GaH_3$ characterized by its vibrational spectra.[10,11]

Acknowledgment. We thank the SERC for supporting this research and for funding studentships for M.J.G. and C.R.P.

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Enantioselective Syntheses of Secondary Homoallyl Alcohols with Optically Active η^3-Allylmolybdenum Complexes

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The enantioselective synthesis of secondary homoallylic alcohols is of interest in the context of acyclic stereoselective synthesis, with particular emphasis on their utility as biosynthetic intermediates.[1] The condensation of carbonyl compounds with main group organometallic allyl reagents has been a successful strategy in this respect. In particular, the use of chiral metal templates for asymmetric induction during the condensation of aldehydes with metal allyls has been developed. Chiral organometallic complexes including allylstannanes,[2] allylaluminum,[3] and allylboranes[4-6] react with aldehydes generating nonracemic homoallyl

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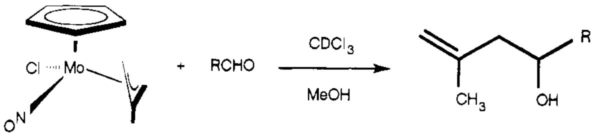
alcohols. However, the high reactivity of these main group organometallic reagents frequently results in a loss of regio- and stereoselectivity, reducing their utility as reagents for asymmetric synthesis. *B*-allylboranes have been extensively studied owing to the relative ease of incorporation of chiral moieties derived from naturally occurring materials. Homochiral *B*-allylboranes derived from camphor glycols have been successfully used by Hoffmann et al.[4] for condensation with aldehydes, generating homoallyl alcohols in 45–77% ee. More recently others have developed α-pinene and tartrate analogues which add in 83–96% ee.[5,6]

With the exception of allylnickel reagents,[7] attention has focussed on main group derivatives. Our preliminary studies of homoallylic alcohol synthesis via condensation of aldehydes with CpMo(NO)(Cl)(η^3-allyl) reagents indicate that η^3-allylmolybdenum complexes have great potential in the synthesis of this important class of compounds. These neutral halides are robust air-stable complexes which can be handled with no special precautions. The resolution of the chiral metal center by incorporation of a neomenthyl unit allowed us to demonstrate that the condensations of benzaldehyde and propionaldehyde proceed with 97% stereoselectivity. This suggests that the enantioselectivity is independent of the nature of the aldehyde.

Preliminary studies were carried out in $CDCl_3$ in NMR tubes in the presence of methanol. The CpMo(NO)(X)(η^3-2-methallyl) complexes were treated with excess aldehyde (2–3 equiv) to yield the corresponding homoallyl alcohols in high yield (90–100%).[8] The reaction rates were highly dependent on the halide ligand. For CpMo(NO)(Cl)(η^3-methallyl), the reaction with benzaldehyde required ~1 day at room temperature, generating 3-methyl-1-phenyl-3-buten-1-ol, **1**, whereas use of CpMo(NO)(I)(η^3-methallyl) required ~1 week for completion. Aliphatic aldehydes react faster (~8 h), as shown by the clean reaction of propionaldehyde with CpMo(NO)(Cl)(η^3-methallyl), to generate 5-methyl-5-hexen-3-ol, **2**.

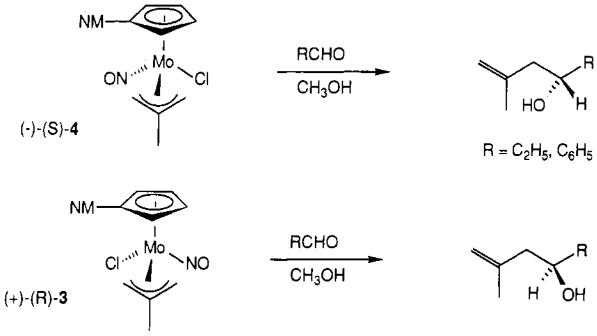


1 (R = Ph)
2 (R = Et)

In order to assess the ability of homochiral molybdenum η^3-methallyl complexes to enantioselectively generate the homoallyl alcohols, the neomenthyl-substituted complexes were studied. Owing to the reduced reactivity and relative ease of purification, the neutral iodides were prepared[9] initially. *Samples of both diastereomers were readily isolable with very high diasteromeric excess* (>97% *de*) via fractional crystallization. The homochiral iodides were then converted to chlorides by sequential addition of Ag^+ and Cl^- yielding the epimers of NMCpMo(NO)(Cl)(η^3-methallyl), (+)-**3** and (−)-**4**, which differ in the configuration at the stereogenic metal center. Treatment of >98% de (−)-NMCpMo(NO)(Cl)(η^3-methallyl), (−)-**4**, with benzaldehyde gave (+)-(*R*)-3-methyl-1-phenyl-3-buten-1-ol in >98% ee.[10]

To eliminate the possibility of diastereomer fractionation in intermediates and to facilitate accurate determination of the stereoselectivity of the reaction, samples of NMCpMo(NO)(Cl)(η^3-methallyl) with lower de were used in other experiments. The % de were determined by ^{1}H NMR integration prior to aldehyde addition. The course of the reaction was monitored by ^{1}H NMR to assure that reaction was complete. This was necessary since the starting materials are diastereomers and presumably react with slightly different rates with the aldehydes. Thus, stopping the reaction before completion could result in a nonracemic sample of the corresponding homoallyl alcohol for which the % ee does not reflect the true stereoselectivity of the reaction.

NMCpMo(NO)(Cl)(η^3-methallyl) containing 80% (+)-**3** and 20% (−)-**4** (60% de) yielded (−)-(*S*)-3-methyl-1-phenyl-3-buten-1-ol[11] when treated for 2 days with excess benzaldehyde. A chiral shift reagent experiment showed the product to be 78% (−)-(*S*)-**1** and 22% (+)-(*R*)-**1** (56% ee).[12] This implies a 97% stereoselectivity during the reaction with benzaldehyde. When propionaldehyde was treated with a mixture of 63% (−)-**4** and 37% (+)-**3** (26% de), (−)-(*S*)-5-methyl-5-hexen-3-ol was isolated in 24% ee, indicating a stereoselectivity of 98%.[12]



An obvious mechanism of formation would involve attack of an η^1-allyl on an η^1-aldehyde through a chair-like transition state, followed by hydrolysis. A detailed analysis is premature, but it is clear that the allyl group adds to a specific enantioface of the aldehyde for a given absolute stereochemistry at the metal. Given the absolute configuration at the metal center for (−)-NMCpMo(NO)(Cl)(η^3-methallyl)[13] and the anticipated structure of an η^1-aldehyde intermediate, the same sense of product chirality

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[8] The yields were calculated by integration of ^{1}H NMR spectra with an internal standard of 1,2-dichloroethane (δ 3.70). The yields ranged between 90 and 100%, with isolated yields between 40 and 50% on small scale reactions. A description of a typical larger scale reaction follows: to a CH_2Cl_2 solution of 206 mg of CpMo(NO)(CO)(η^3-methallyl) was added 118 mg (1.5 equiv) of benzaldehyde and 85 mg (3.6 equiv) methanol. The solution, which was initially yellow, was stirred for 24 h at room temperature. A resulting deep red precipitate was removed by filtration through alumina and elution with CH_2Cl_2. Although the amount of impurities were small, purification on preparative TLC (silica gel, CH_2Cl_2, R_f(**1**) = 0.4) yielded 75 mg (63%) of **1**. The low isolated yields are presumably the consequence of purifying small amounts of relatively volatile products.

[9] The preparation of similar NMCp compounds are reported in the following: Faller, J. W.; Shvo, Y.; Chao, K.-H.; Murray, H. H. *J. Organomet. Chem.* **1982**, *226*, 251.

[10] Following the procedure for **1**,[8] using pure (−)-**4** with $[\alpha]_D^{23}$ −159° (*c* 0.8, $HCCl_3$) (517 mg, 1.23 mmol) gave an 87% yield of crude product. Column chromatography (alumina, hexane, CH_2Cl_2, Et_2O) gave 0.120 mg (0.74 mmol, 60%) of (+)-(*R*)-3-methyl-1-phenyl-3-buten-1-ol with $[\alpha]_D^{23}$ +51° (*c* 1.55, benzene), for which the concentration of (*S*)-alcohol was below that detectable by $Eu(tfc)_3$ shift experiments. [Compare with $[\alpha]_D^{23}$ −44.9 (*c* 7.38 benzene) reported for the (*S*)-alcohol 96% ee].[4b]

[11] Signs of rotation and absolute configurations of the homoallylic alcohols have been correlated elsewhere.[4b] In order to unambiguously assign the configuration of the alcohols generated with CpMo(NO)(Cl)(η^3-methallyl), samples of (−)-(*S*)-**1** and (+)-(*R*)-**2** were prepared from the chiral borane complex (−)-*B*-methoxydiisopinocampheylborane, which is derived from (+)-α-pinene.[4b] The relative position of the enantiomer resonances in the ^{1}H NMR in the presence of the $Eu(tfc)_3$ chiral shift reagent were used as a reference for the determination of the configuration. This method confirmed the assignment based on the rotation of the samples.

[12] The ee of the product alcohols were determined by addition of the $Eu(tfc)_3$ to a benzene-d_6 solution of the nonracemic alcohols. The olefinic protons at ~4.8 ppm were monitored until a sufficient downfield shift (~0.8 ppm) split the broad singlets to allow integration.

[13] The absolute configuration of the metal in (−)-**4** was determined to be (*S*) by X-ray crystallography by using the known configurations within the neomenthyl group. This complex crystallizes in the monoclinic space group $P2_1$ with a = 6.410 (2) Å, b = 9.774 (2) Å, c = 16.012 (3) Å, β = 91.14 (2)°, and V = 1002.8 (6) Å^3.

will generally be imparted to the products for a given hand of the stereogenic metal center. Thus, (–)-**4** ultimately yields the product which would be expected from allyl addition to the *re*-face of the aldehyde. Consequently, this system should provide a general and predictable route for the formation of homoallylic alcohols with high enantioselectivity.

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Catalytic Conversion of Molecular Nitrogen into Silylamines Using Molybdenum and Tungsten Dinitrogen Complexes[1]

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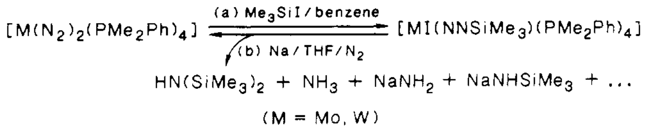
Although many attempts have been made to develop a system in which molecular nitrogen is catalytically converted into nitrogen hydrides or organonitrogen compounds, the examples reported to date of the effective catalysis by transition-metal complexes under mild conditions are quite rare.[2] Several groups including ours have been studying the reactivities of molybdenum and tungsten dinitrogen complexes of the type $[M(N_2)_2(L)_4]$ (L = tertiary phosphine) and have clarified the details of the attractive reactions such as the protonation and the C–N bond formation at the dinitrogen ligand in these complexes. However, none of these reactions have led to the development of any catalytic nitrogen-fixing systems, since it is difficult to establish the methods to protonate dinitrogen or to form a C–N bond at the dinitrogen ligand accompanied by reducing in situ the high-valent metal species produced by those reactions to the lower oxidation state under the same conditions of the protonation and C–N bond formation reactions.[3] Here we wish to describe initially the formation of the N_1 products concurrent with regeneration of the parent dinitrogen complexes, when trimethylsilylated dinitrogen complexes of Mo and W are reduced with excess Na. This finding has finally led to development of the catalytic system in which molecular nitrogen is converted into silylamines promoted by these dinitrogen complexes.

Previously we reported the preparation of the trimethylsilyldiazenido complexes *trans*-$[WI(NNSiMe_3)(PMe_2Ph)_4]$ (**1**) and $[WI(NNSiMe_3)(dpe)_2]$ by the reactions of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$ (**2**) and *trans*-$[W(N_2)_2(dpe)_2]$ (dpe = $Ph_2PCH_2CH_2PPh_2$) with Me_3SiI.[4] Analogous treatment of *cis*-$[Mo(N_2)_2(PMe_2Ph)_4]$ (**3**) and *trans*-$[Mo(N_2)_2(dpe)_2]$ with Me_3SiI afforded the corresponding molybdenum complexes *trans*-$[MoI(NNSiMe_3)(PMe_2Ph)_4]$ (**4**) and $[MoI(NNSiMe_3)(dpe)_2]$, respectively.[5] These reactions serve as the first example of the Si–N bond formation by reaction at a coordinated dinitrogen moiety.

When complex **1** was treated with excess Na sand (0.5–1 mm diameter) in THF at 30 °C under Ar, $HN(SiMe_3)_2$ and NH_3 were produced in substantial yields accompanied by the formation of free N_2 and a small amount of the parent dinitrogen complex **2**. Further NH_3 was detected in moderate yield after hydrolysis of the evaporated reaction mixture residue. The total nitrogen balance of the products based on complex **1** as a unit of dinitrogen was 0.95. When this reaction was carried out under N_2, the parent dinitrogen complex **2** was regenerated in moderate yield. The reactions with Na metal proceeded analogously for complex **4**, the results of which are summarized in Table I.



$$[M(N_2)_2(PMe_2Ph)_4] \underset{\text{(b) Na/THF/N}_2}{\overset{\text{(a) Me}_3\text{SiI/benzene}}{\rightleftarrows}} [MI(NNSiMe_3)(PMe_2Ph)_4]$$

$$HN(SiMe_3)_2 + NH_3 + NaNH_2 + NaNHSiMe_3 + \ldots$$

$$(M = Mo, W)$$

As a result, about half of the nitrogen atoms in the trimethylsilyldiazenido complexes are converted into the N_1 products in this reaction system, with the remainder forming N_2 gas. Among these products, $HN(SiMe_3)_2$ might be formed by the disproportionation reaction between two unstable H_2NSiMe_3 molecules, the protons of which may be derived from THF and/or a trace amount of H_2O still remaining despite the employment of rigorously dry conditions. Major N_1 products which were detected as NH_3 after hydrolysis are presumably present as the sodium salts such as $NaNH_2$ or $NaNHSiMe_3$, since the addition of Et_3SiCl to the resultant reaction mixture of complex **1** with Na resulted in the formation of Et_3SiNH_2 and $(Et_3Si)(Me_3Si)NH$, which were detected by GC-MS. When complex **1** was reduced with Na under Ar in the presence of excess Me_3SiCl, $N(SiMe_3)_3$ was formed as the principal product in a yield of 0.42 mol per W atom.

These observations led us to investigate the reactions of Me_3SiX and Na in THF in the presence of a catalytic amount of these Mo and W dinitrogen complexes under dinitrogen. To enhance the reaction rate, Na microdispersion (8–10 μ diameter) was used in place of Na sand. The trimethylsilylation of the dinitrogen ligands in complexes **2** and **3** proceeds cleanly for Me_3SiI as described above. However, the reactions using Me_3SiBr and Me_3SiCl were undertaken because Me_3SiI is highly reactive toward THF.[6] When an equimolar amount of Me_3SiBr and Na were reacted at 30 °C in the presence of 1 mol% of complex **3** under dinitrogen, $N(SiMe_3)_3$ and $HN(SiMe_3)_2$ were obtained in 10% (3.2 mol/Mo atom) and 4% (1.8 mol/Mo atom) yields, respectively, accompanied by the formation of $Me_3SiSiMe_3$ and the ring-opening product of THF, $Me_3Si(CH_2)_4OSiMe_3$, as byproducts in substantial yields. When Me_3SiCl was used in place of Me_3SiBr, the yields of the silylamines increased to 24% (7.5 mol/Mo atom) for $N(SiMe_3)_3$ and 1.2% (0.6 mol/Mo atom) for $HN(SiMe_3)_2$. All charged Me_3SiCl was consumed in 4 h under these reaction conditions, and the major byproduct was $Me_3SiSiMe_3$. Therefore the reactions of Me_3SiCl with Na were investigated in further detail.

$$Me_3SiCl + N_2 + Na \xrightarrow[\text{THF}]{\text{catalyst}} N(SiMe_3)_3 + HN(SiMe_3)_2 + Me_3SiSiMe_3$$

As shown in Table II, complex **3** showed the highest catalytic activity for formation of the silylamines among the complexes examined here, and when the reaction system containing complex **3** (0.05 mmol) was diluted 5 times with THF, the yield of the silylamines was enhanced to 38% (25 mol/Mo atom) accompanied

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(6) The trimethylsilylation of the dinitrogen ligand in complex **3** also takes place by the reaction with Me_3SiBr, which was confirmed by the isolation of the trimethylsilylhydrazido(2–) complex *mer*-$[MoBr_2(NNHSiMe_3)(PMe_2Ph)_3]$ from the reaction mixture: IR (KBr disk, cm^{-1}) ν(NH), 3180 w; ν(NN), 1341 s; ν(SiN), 843 s; $\delta(SiMe_3)$, 1252 m.

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