desirable, since the stereochemical fates of subsequent reactions will lead inextricably to **1**, **2**, and **3**.

Alkylation of **14** using lithium diisopropylamide in THF at −70 to −20 °C and quenching with ethyl bromoacetate gave **15** (82%)[7] as a mixture of epimers (~1:1 by NMR) at C-6.[9] Reduction of the C-7 carbonyl was achieved using sodium borohydride in 3 N NaOH-MeOH, and proceeded via the keto acid **16**,[7] to give the cis lactone **17** as a mixture of epimers at C-1 (60%). The epimeric lactones **17** appeared as a single compound on chromatography.

The mixture of epimeric lactones **17** was treated with lithium diisopropylamide in THF at −70 °C, followed by Eschenmoser's salt ($Me_2N^+CH_2I^-$),[10] and then warmed to 20 °C; workup with MeI followed by Na_2CO_3 gave the α-methylene lactones isolinderalactone (**2**)[11] and *epi*-isolinderalactone (**4**). When the mixture of **2** and **4** was heated to 160 °C for a few minutes, **4** was irreversibly Cope rearranged into neolinderalactone (**3**), and **2** was reversibly Cope rearranged into linderalactone[11] (**1**). Prolonged heating at 260 °C slowly and irreversibly transformed neolinderalactone (**3**) into **2**[5] which again Cope rearranged to give linderalactone (**1**).[4] In this way the unnatural isomer **4** is in effect epimerized at the quaternary C-1 position through two Cope rearrangements, and consequently the synthesis leads to an equilibrium mixture of **1** and **2**.[2b,4]

In conclusion this first synthesis of the linderalactones **1**, **2**, and **3** provides a unique opportunity to epimerize at a quaternary carbon atom using two Cope rearrangements, and thus avoid stereochemical considerations at C-1.

Acknowledgments. Dr. Clifford Burford, Dr. Ed Ehlinger, and Mr. Wilson Hsin are thanked for their contributions to parts of this work.

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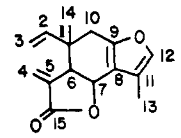

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(11) Linderalactone (**1**), isolinderalactone (**2**), and neolinderalactone (**3**) were readily (PLC) separated and identified. Comparison (TLC) of **1** and **2** was made with authentic samples kindly provided by Dr. Takeda. Spectra agreed with those reported,[1-3] and the Cope rearrangement[4] further demonstrated the identities of **1**, **2**, and **3**.

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Stereoelectronic Effects in the Reactions of Phosphate Diesters, Phosphoramidates, and Phosphonates. 3. Ab Initio Molecular Orbital Calculations of Transition States

Sir:

The hypothesis of stereoelectronic control in the reactions of tetracovalent carbon and phosphorus species and pentacovalent phosphoranes has recently received both experimental and theoretical support.[1-10] Deslongchamps[1] and co-workers have demonstrated selective cleavage of bonds which are antiperiplanar (app) to lone pairs on directly bonded oxygen and nitrogen atoms in tetrahedral carbon species. For phosphate ester hydrolysis Gorenstein et al.[8,9] have calculated reaction profiles and pentacovalent phosphorane transition-state energies which support a large stereoelectronic acceleration in the breaking of a P-O ester bond which is app to lone pairs on directly bonded oxygen atoms. In this communication we explore in further ab initio molecular orbital calculations the stereoelectronic effect on phosphorane transition states which do not have an app ester oxygen lone pair.

In an S_N2(P) mechanism, the base-catalyzed hydrolysis of dimethyl phosphate, *O*-methyl-*N*-methyl phosphoramidate, and methyl ethylphosphonate (X = O, NH, or CH_2, respectively in eq 1) proceeds via transition states **1-3** shown in Figure 1.

$$CH_3O(CH_3X)PO_2^- + {}^-OH \rightarrow \underset{\mathbf{1\text{-}3}}{CH_3O(CH_3X)PO_3H^{2-}} \rightarrow CH_3XPO_3H^- + CH_3O^- \quad (1)$$

Geometries for the two trigonal bipyramidal conformations t,g,−g and g,t,t shown were identical with the extensively optimized geometries for the dimethyl phosphoranes **1a** and **1b** previously calculated, except P-N, P-C, N-C, and C-C bond lengths of 1.740, 1.850, 1.40, and 1.48 Å, respectively, and a PNC bond angle of 115.3° were substituted where appropriate. In all cases a collinear ^-OH attack and MeO^- leaving were assumed. The dimethyl phosphate reaction was shown to

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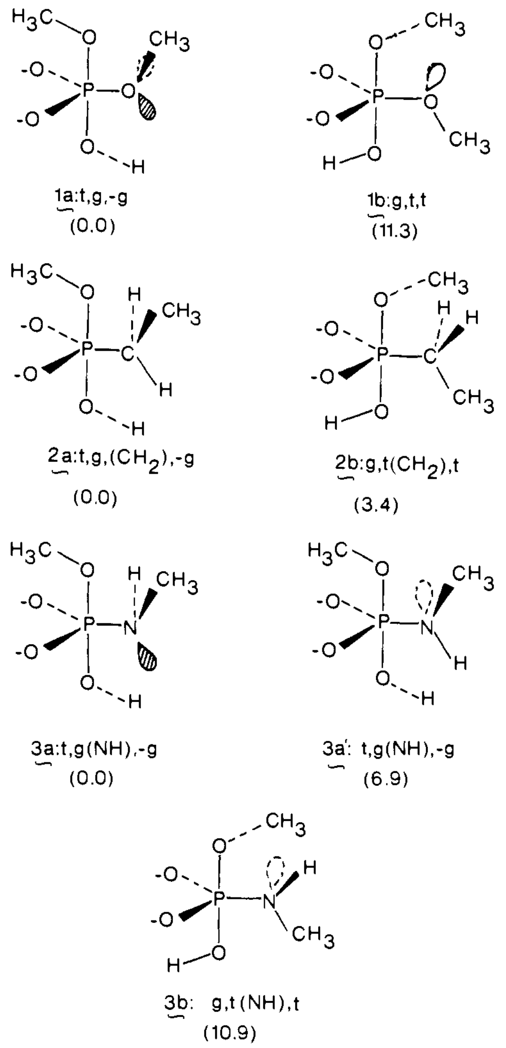

Figure 1. Structures and relative energies for the trigonal bipyramidal transition states for apical methoxyl leaving for the hydroxide catalyzed hydrolysis of dimethyl phosphate (**1a** and **1b**), methyl ethylphosphonate (**2a** and **2b**), and *O*-methyl-*N*-methyl phosphoramide (**3a**, **3b**, and **3b'**). Torsional angles about the P-X bonds for **1-3** are defined by the MeOPX structural fragment, and the torsional angle about the P-OH bond is defined by the POH fragment (X = O, CH_2, NH). Conformers are defined by the following order for the torsional angles: apical P-O ester bond, equatorial P-X bond, and apical P-OH bond. The t,g,−g conformation is shown with basal antiperiplanar lone pair to axial bond methoxyl shaded. Energy (in parentheses) in kilocalories/mole for the g,t,t conformation is shown relative to the t,g,−g conformation of the same molecule. Total energy of **1a** is −784.429 57. Total energy of **2a** is −749.140 977. Total energy of **3b** is −764.873 92. Relative energies for **3a** and **3b'** with PNC bond angle of 120° are 8.9 and 6.6 kcal/mol, respectively.

proceed via a metastable pentacovalent intermediate with separate transition states for hydroxide attack and methoxide displacement.

For the methoxide elimination transition states **1-3** a P-OCH_3 apical bond length of 2.5 Å was chosen based upon previous studies.[8,9] Calculations used the Gaussian 70 series of programs with the STO-3G basis set[11] as prescribed in ref 9. Relative energies for the two conformations of the transition states **1-3** are shown in Figure 1. The g,t,t methoxide leaving transition state **1a** is 11.3 kcal/mol higher in energy than the t,g,−g transition state **1b**.[8,9] In **1a** the apical methoxyl P-O bond is app to a lone pair on the basal gauche methoxyl oxygen (shown shaded in **1a**, Figure 1). According to the stereoelectronic theory this facilitation of bond translation and weakening of the apical methoxyl bond by an app lone pair on the basal methoxyl atom is attributable to mixing of the nonbonding app lone pair with the antibonding σ^* apical orbital.[12,13]

It is important to note that the conformation of *both* axial bonds is not very important in determining the relative transition-state energies.[9] Thus a g,g,−g dimethoxyphosphorane transition state has nearly the same energy as the t,g,−g. Similarly the g,t,−g transition state is also about the same energy as g,t,t. The energy difference is insensitive to whether optimized geometries for both **1a** and **1b** or a fixed standard geometry are used.

An important test of the validity of the stereoelectronic effect theory is whether the app lone pair in a t,g,−g transition state is essential for these large energy differences. Substitution of a methylene group for the basal oxygen in **2** (Figure 1) should therefore largely eliminate the 11-kcal/mol difference in energy between the t,g,−g and g,t,t transition states. As shown in Figure 1, the g,t(CH_2),t transition state is now only 3.4 kcal/mol higher energy than the t,g(CH_2),−g. A t,t(CH_2),t transition state is also of similar energy to the g,t(CH_2),t structure and these results are independent of whether we use the standard geometry derived from the oxy transition states **1** or whether we partially optimize the *lower* energy methylene transition state **2a**. In addition the 3.4-kcal/mol transition-state energy difference in **2** is about the same as the energy difference between conformations for the metastable *intermediates*. Thus, with an apical P-OCH_3 bond length of 1.82-1.85 Å the g,t,t oxy intermediate is only 4.0-kcal/mol higher energy than the t,g,−g oxy intermediate and the g,t,t methylene intermediate is 4.4-kcal/mol higher energy than the t,g,−g methylene intermediate. (Geometries were the same as reported in ref 9.) These ~4-kcal/mol differences reflect steric interactions since in t,g,−g the X-CH_3 bond (X = O, C) is staggered relative to the P-O bonds while, in the g,t,t structures, the X-CH_3 bond is cis to the apical P-OH bond.

A final test of the stereoelectronic effect is provided by the amido transition states **3**. Again the g,t(NH),t **3b** transition state is 9-11-kcal/mol higher energy than t,g(NH),−g **3a** with the lower energy transition state having a nitrogen lone pair app to the leaving methoxyl group. There are actually two t,g(NH),−g conformations since with a gauche methyl group on the trigonal nitrogen the hydrogen on the basal nitrogen can be either gauche (as in **3a**) or trans (as in **3a'**) to the apical leaving group. Only in **3a** is the nitrogen lone pair app to the leaving group. Although **3a** and **3a'** differ only by the relative orientation of the nitrogen lone pair and hydrogen, **3a** is still 6.9 kcal/mol lower in energy than **3a'**. Finally, the 3-4-kcal/mol energy difference between the **3a'** and **3b** transition states is attributable to the same steric interactions as suggested for the methylene transition states **2a** and **2b**.[14]

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(14) The amidophosphorane intermediate **3a** is only 2.4-kcal/mol higher energy than **3b** and 1.67-kcal/mol higher energy than **3b′**.
(15) Fellow of the Alfred P. Sloan Foundation, 1975–1979.

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Reaction of Group 6 Organoimido Complexes with Organozinc. Reductive Elimination across a Metal-Nitrogen Multiple Bond

Sir:

Sharpless and co-workers have suggested that the addition of bis(alkylimido)osmium complexes to olefins proceeds via migration of an organic ligand to an electron deficient imido nitrogen atom.[1] The industrially important ammoxidation of propylene to acrylonitrile may similarly involve transfer of an allyl moiety to $^{VI}Mo{=}NH$.[2] Unfortunately appropriate model compounds for studying this type of process[3] have not been available: no complex containing both imido and hydrocarbyl ligands [nor any organomolybdenum(VI) species whatsoever] has been reported. We now report that such complexes have been prepared and present preliminary evidence that the proposed rearrangements do occur.

Treatment of $(^tBuN)_2W(O^tBu)_2$ or $(^tBuN)_2Mo(OSiMe_3)_2$[4] with dimethylzinc in hexane, followed by cooling to −40 °C in the latter case,[5] follows eq 1. Red-orange crystals of Ia (60%) or violet crystals of Ib (52% after recrystallization) were collected by filtration.[6] The color of solutions of compounds I discharged to pale yellow upon addition of potential donor ligands (PMe_3, pyridine, $AlMe_3$, O_2) with a concomitant upfield shift of the methyl NMR resonances.[7]

$$2(^tBuN)_2M(OR)_2 + 4Me_2Zn \rightarrow [(^tBuN)_2M(CH_3)_2]_2 + 4MeZnOR \qquad (1)$$

$$\text{Ia, } M = W \qquad \text{b, } M = Mo$$

The X-ray crystal structure[8,9] of Ib (Figure 1) shows it to contain the first example[10] of an unsymmetrically bridging imido ligand. The dimer is situated on a crystallographic center of symmetry and each molybdenum exhibits distorted trigonal bipyramidal coordination. The three Mo–N bond lengths in Ib [Mo–N (1) = 1.730 (2), Mo–N (2) = 1.819 (2), Mo′–N(2) = 2.322 (2) Å] roughly correspond to those expected[10] for triple-, double-, and single-bonded nitrogen, respectively. However, Mo–N(1) is ~0.02 Å longer[10] than is anticipated for a Mo–N triple bond and the Mo–N–C bond angle also deviates somewhat from linearity [Mo–N(1)–C(11) = 167.4 (1)°].

When diphenylzinc was added to a solution of $(^tBuN)_2Cr(OSiMe_3)_2$ in hexane, $PhZnOSiMe_3$ precipitated as expected but no complex analogous to Ib could be isolated. Instead, upon hydrolysis, organic products[11] including biphenyl and *N*-*tert*-butylaniline were identified by GLC as summarized in Table I. Thus, under conditions where diphenylzinc is added dropwise *to* the chromium complex at 80 °C, up to 70% *tert*-butylaniline is observed. It is noteworthy that the ratio of *tert*-butylaniline to biphenyl in the product decreases when (1) the reagents are added in inverse order, (2) the reaction is carried out at reduced temperature, (3) the reaction is carried out in the presence of added pyridine, or (4) complex Ib is substituted for the chromium complex in this reaction.

Table I. Yield of *N*-*tert*-Butylaniline and Biphenyl from Reaction of 1 mmol of $(Me_3SiO)_2Cr(N^tBu)_2$ with Diphenylzinc

Ph_2Zn, mmol	temp, °C	addition[a]	additive	$PhNH^tBu$[b]	Ph–Ph[c]
2.0	80	normal		0.704	0.293
2.0	25	normal		0.334	0.377
2.0	80	inverse		0.258	0.638
2.0	80	normal	pyridine[d]	0.174	0.660
2.0	−78	inverse		0.038	0.687
1.0	80	normal		0.699	0.096
2.0	80	normal	Mo[e]	0.015	0.352

[a] In normal addition the Ph_2Zn in 10 mL of toluene was added to the Cr complex in 10 mL of toluene. [b] Yield of *N*-*tert*-butylaniline in millimole (by GLC). [c] Yield of biphenyl in millimole (by GLC). [d] The Cr complex was dissolved in 9 mL of toluene and 1 mL of pyridine. [e] Compound Ib was substituted for the Cr complex.

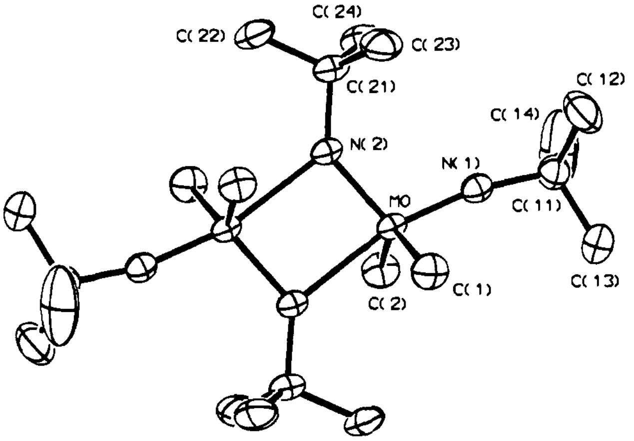
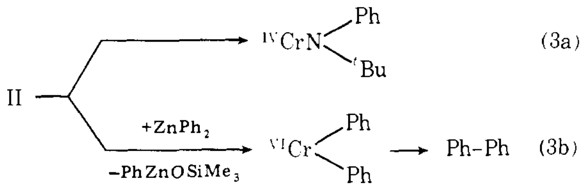


Figure 1. Structure of bis(μ-*N*-*tert*-butylimido)-(*N*-*tert*-butylimido)-tetramethyldimolybdenum.

The observation of different products depending on the order of addition of the reactants requires formation of at least one intermediate. In light of the facile substitution of compounds I, a reasonable intermediate is a monophenyl substituted derivative such as II.[12] Intermediate II may then undergo phenyl migration to the electron-deficient imido nitrogen affording a chromium(IV) amide (eq 3a). Alternatively, II can react with a second equivalent of zinc reagent to afford a diphenyl derivative which subsequently reductively eliminates biphenyl (eq 3b). In accord with this model, factors 1–4 noted above can be expected in each case to increase the likelihood that II will survive to undergo reaction 3b.[13]

$$(^tBuN)_2Cr(OSiMe_3)_2 + Ph_2Zn \longrightarrow (^tBuN)_2Cr(OSiMe_3)Ph + PhZnOSiMe_3 \qquad (2)$$

$$\text{II}$$



Supplementary Material Available: Final positional parameters (as fractional coordinates), table of thermal parameters, and structure factor amplitudes (observed and calculated) of $C_{20}H_{48}Mo_2N_4$ (21 pages). Ordering information is given on any current masthead page.

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