Communications to the Editor

Solution and Solid-State Structure of a Phosphorus-Stabilized Carbanion

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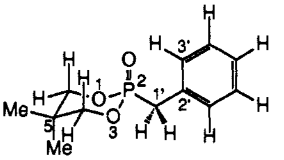
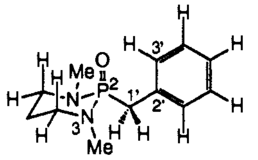
Heteroatom-stabilized anions are among the synthetic chemists' most versatile reagents for carbon–carbon bond formation.[1] Yet, they are also among the most poorly understood. This discrepancy between utility and knowledge has stimulated investigations into the fundamental aspects of carbanion structure and reactivity.[1e,2] We are involved in a program on the development of stereoselective carbon–carbon bond forming reactions using auxiliary-based, chiral phosphorus-stabilized anions.[3] A significant portion of this program is aimed at elucidating the structure of and bonding in such species to allow for rational design of chiral auxiliaries. We report herein the results of solution NMR[4] and X-ray crystallographic analysis of lithiated phosphonyl derivatives.



1



2

The substrates examined are the 2-benzyl-2-oxo-1,3,2-dioxaphosphorinane (**1**) and 2-benzyl-2-oxo-1,3,2-diazaphosphorinane (**2**). ^1H, ^{13}C, and ^{31}P NMR spectra of the neutral materials and their derived lithio anions[5] (Li^+1^-, Li^+2^-) were recorded at temperatures between 20 and −90 °C in THF-d_8.[6] A summary of the relevant spectroscopic data is collected in Table I. The conformational analysis of **1** and related compounds has been well studied.[7] The ring is conformationally mobile and the equilibrium composition of the two chair forms (α and β, Scheme I) is dependent on temperature,[7c] solvent,[7a] and additives.[7i,j] At 20 °C in THF the α-form is preferred (57/43)[8] while at −76 °C the β-form is preferred (70/30).[9] This switch is consistent with increased stabilization of the more polar conformer (β)[10] as the

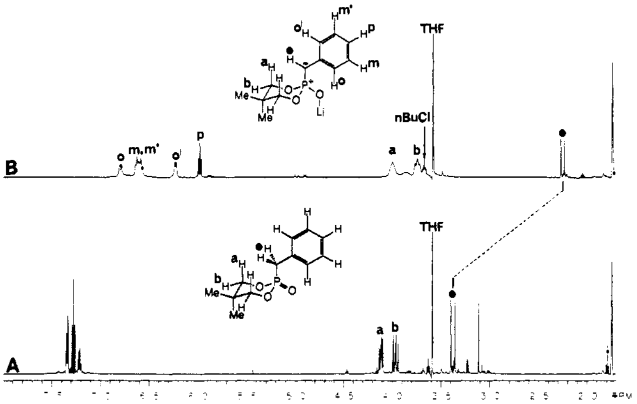


Figure 1. ^1H NMR spectra (500 MHz) of **1** (A) and Li^+1^- (B) in THF-d_8 at −86 °C.

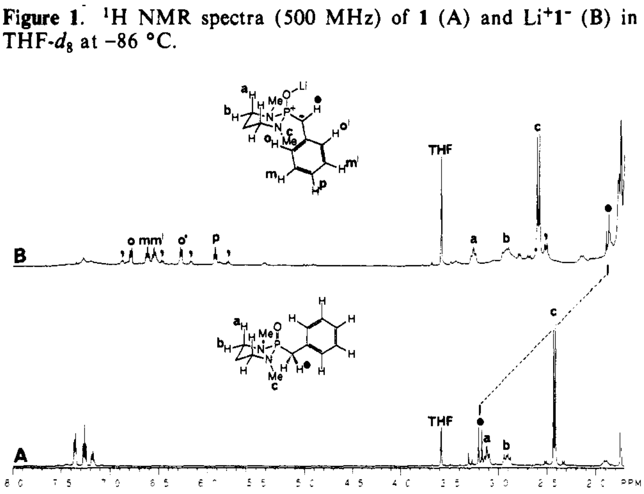
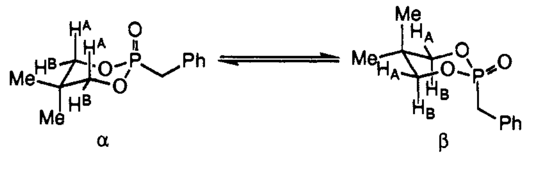


Figure 2. ^1H NMR spectra (500 MHz) of **2** (A) and Li^+2^- (B) in THF-d_8 at −76 °C.

Scheme I



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[3] (a) Denmark, S. E.; Marlin, J. E. *J. Org. Chem.* **1987**, *52*, 5742. (b) Denmark, S. E.; Rajendra, G.; Marlin, J. E. *Tetrahedron Lett.* **1989**, *30*, 2469. (c) Denmark, S. E.; Dorow, R. L. *J. Org. Chem.* **1989**, *54*, 3. (d) Denmark, S. E.; Cramer, C. J. *J. Org. Chem.*, in press.

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[5] Generated with either 10 M *n*-BuLi or 1.5 M *s*-BuLi. Final concentrations 0.1–0.14 M.

[6] Spectra were recorded at 500 MHz (^1H), 125.8 MHz (^{13}C), and 121.4 MHz (^{31}P) with calibrated probes.

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[9] This is in good agreement with the estimates of Edmundson[7e] and Katritzky.[7f]

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Table I. Selected NMR Data for $Li^+\mathbf{1}^-$ and $Li^+\mathbf{2}^-$ [a]

	$\Delta\delta$ ^1H (ppm)[b]			$\Delta\delta$ ^{13}C (ppm)[c]			ΔJ^n (Hz)		
species	H(1′)	H_{ax} (4)	H_{para}	C(1′)	C_{para}	$\Delta\delta$ ^{31}P (ppm)[d]	$J^1_{C(1')H}$	J^1_{PC}	$J^2_{PH(1')}$
$Li^+\mathbf{1}^-$	−1.13	−0.17	−1.23	3.79	−15.45	18.2	18.2	96.4	−2.3
$Li^+\mathbf{2}^-$	−1.30	0.14	−1.26	4.58	−16.35	18.4	21.3	105.7	−3.2

[a] $\Delta\delta$ = (δ anion) − (δ neutral), positive numbers are downfield shifts. [b] 500 MHz, −86 °C ($Li^+\mathbf{1}^-$); −76 °C ($Li^+\mathbf{2}^-$); THF-d_8. 125.8 MHz, 20 °C; THF-d_8. [d] 121.4 MHz, −72 °C; 1:1 THF/THF-d_8.

Table II. Selected Bond Lengths (Å) and Angles (deg) for $(Li^+\mathbf{2}^-\cdot 2THF]_2$

C(1′)–P	1.689	P–N(1)	1.681	O(1)–Li	1.912
C(1′)–C(2′)	1.433	P–N(2)	1.675	O(2)–Li	1.981
P–O(1)	1.516			O(3)–Li	1.969

P–C(1′)–C(2′)	130.7	N(1)–P–O(1)	105.6
C(1′)–P–O(1)	112.8	N(2)–P–O(1)	112.3
P–O(1)–Li	140.2	N(1)–P–N(2)	101.1

dielectric constant of the solvent increases with decreasing temperature.[11]

Formation of the anion is accompanied by dramatic upfield shifts of all aromatic protons, the ortho and para carbons, and the benzylic (carbanionic) proton (Figure 1). The ortho protons are highly anisochronous at −86 °C due to slow rotation about C(1′)–C(2′). However, we were unable to detect conformational isomerism about the P–C(1′) bond; only one benzylic proton was detected at all temperatures. $Li^+\mathbf{1}^-$ behaves analogously to **1** in the ring conformational inversion upon lowering temperature. In $Li^+\mathbf{1}^-$ the α conformer is more strongly favored (65/35, 20 °C) due to the increased basicity of the phosphonyl oxygen.[7j] The increase in $^1J_{C(1')H}$ and $^1J_{PC}$ upon deprotonation is characteristic for the change in hybridization from sp^3 to sp^2.[12] Finally, the downfield shift of the ^{31}P resonance is indicative of the polarization of the phosphonyl group to stabilize the anion.[3d,13]

Ring conformational analysis of the diaza analogue **2** is complicated by the coupling due to H–C(5) and the flattening of the phosphonamide moiety.[14] Nevertheless, only chair–chair equilibria are expected.[14c] No conformational inversion is seen upon lowering the temperature. Changes reminicent of those in **1** were observed upon anion formation (Table I). The upfield shifts of the para proton and carbon are larger, indicative of greater charge displacement toward the benzene ring. The most striking aspect of the anion spectrum (Figure 2) is the *persistence of a single (^{31}P-coupled) methyl resonance down to −76 °C.* Thus, the symmetry of the molecule is maintained, suggesting either a low rotational barrier about P–C(1′) or a high barrier in an anacomeric C_s symmetrical anion. A small barrier is expected by analogy to P-ylides[15] and from theoretical considerations.[16] On the other hand, HC(1′) and H_{ortho} are not broadened. Resolution of this question will require a symmetrically substituted anion. The aggregation state of the anions in solution is uncertain. However, we have demonstrated that the anions do form *mixed* aggregates with both educts as well as with *n*- or *s*-BuLi (see arrowheads, Figure 2).

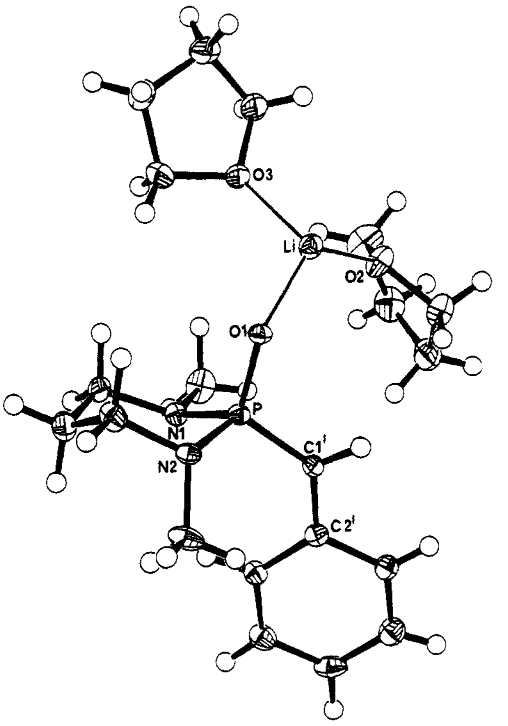

Figure 3. Monomeric unit of $[Li^+\mathbf{2}^-\cdot 2THF]_2$ from X-ray crystallographic analysis.

We have successfully crystallized $Li^+\mathbf{2}^-$ from THF solution and determined its structure by X-ray crystallography (Figure 3).[17] Selected bond lengths and angles are collected in Table II. The anion crystallizes as a centrosymmetric dimer defining a planar Li–O–Li–O rhombus with two THF molecules ligated to each Li atom. The Li–C(1′) distance (3.88 Å) is greater than the sum of van der Waals radii as is also the case in a lithio sulfoxide[18a] and in lithio sulfones.[18b,c] The carbanionic carbon is planar and at nearly a 0° angle to the P=O to maximize p-type interaction. There is a considerable shortening of the P–C(1′) bond (0.11 Å) and a lesser lengthening of the P–O (0.04 Å) and P–N (0.02 Å) bonds compared to average literature values for neutral phosphonamides.[19] Perhaps the most remarkable feature of the anion

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is the pyramidality of the nitrogens,[20] clearly disposing the methyl groups to axial and equatorial positions. We assume the nitrogens are more nearly trigonal in **2** by analogy to most phosphonamides,[19] phosphoramides, and 2-oxo-1,3,2-oxazaphosphorinanes unconstrained by small rings.[21]

These structural insights are valuable for the understanding of bonding in these systems and for the rational design of chiral adjuvants. Further studies on the structure and reactivity of P-stabilized anions are in progress.

Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research. S.E.D. acknowledges the A. P. Sloan foundation for a Fellowship (1985–1989). We are also grateful to the National Science Foundation (Presidential Young Investigator Award, NSF CHE-8451321) for support. Dr. Scott R. Wilson and Charlotte Stern are thanked for their help with the X-ray structure analysis.

Supplementary Material Available: A listing of crystal and positional parameters, bond lengths, bond angles, and torsional angles for $[Li^+2^- \cdot 2THF]_2$ (24 pages). Ordering information is given on any current masthead page.

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(20) Sum of the angles: N(1) 346.2°; N(2) 346.4°.

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Diastereoselective Aldol Reactions Using β-Keto Imide Derived Enolates. A Versatile Approach to the Assemblage of Polypropionate Systems

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We have recently described complementary methods for the synthesis of β-keto imides such as **1** wherein full control of the potentially labile C_2 methyl-bearing stereocenter can be maintained.[1] The surprising attenuation of the kinetic lability of these substrates has been attributed to the intervention of local nonbonding interactions of the A(1,3) type[2] which inhibit the stereoelectronically required alignment of both carbonyls with the C_2 hydrogen. We now describe the unprecedented enolization of this substrate and the diastereoselective aldol reactions of the derived Sn(II) and Ti(IV) enolates which provide access to the complementary diastereomeric β-keto imide adducts **2** and **3** (Scheme I).

Efforts to effect the regioselective enolization of **1** toward the less substituted ethyl substituent initially focused on the use of Lewis acids in conjunction with tertiary amine bases. After an examination of a number of Lewis acids, it was observed that the Mukaiyama protocol employing $Sn(OTf)_2$[3] with triethylamine (CH_2Cl_2, −20 °C, 1 h) provided a stereochemically homogeneous *Z* enolate which could be trapped by chlorotrimethylsilane.[4] This and related proton quenching experiments established that there is no detectable loss of stereochemistry at C_2 under these enolization conditions. The resultant aldol addition reactions of this enolate proceeded in good yields (71–86%) and stereoselectivities to give the aldol adducts **2** possessing the anti relationship between the C_2 and C_4 methyl groups (Scheme I, Table I).[5] The stereochemical assignments of the aldol adducts **2** and **3** were established by independent unambiguous synthesis. These assignments were confirmed by stereoselective ketone reduction (vide infra) and δ-lactonization to provide substrates from which the four stereocenters could be correlated by ^1H NMR spectroscopy.

In order to amplify the utility of **1** as a dipropionate synthon, we sought to discover other types of metal enolates that might proceed via the complementary aldol reaction to deliver the all-syn aldol diastereomer **3**.[6] This objective was realized when it was

Scheme I

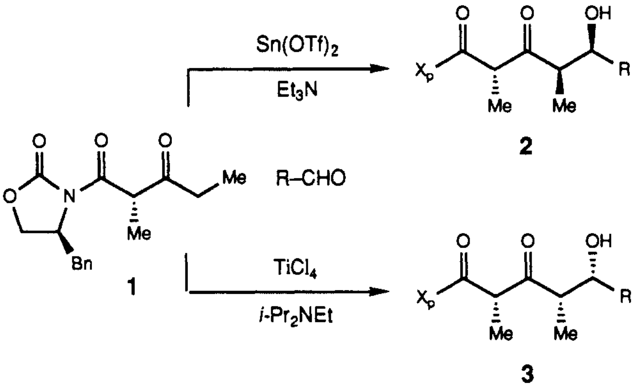
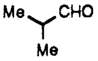
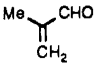
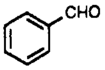


Table I. Aldol Reactions of **1** with Representative Aldehydes (Scheme I)

enolizatn conditns	RCHO[a]	yield,[a] %	ratio[c] **2**:**3**
$Sn(OTf)_2$, Et_3N		83	95:5
$TiCl_4$, iPr_2NEt		86	<1:99
$Sn(OTf)_2$, Et_3N		77[d]	95:5[d]
$TiCl_4$, iPr_2NEt		64[d]	2:98[d]
$Sn(OTf)_2$, Et_3N		71	79:21
$TiCl_4$, iPr_2NEt		86	<1:99
$Sn(OTf)_2$, Et_3N		85	89:11
$TiCl_4$, iPr_2NEt		81	4:96

[a] Unless noted, 1–1.1 equiv of aldehyde was employed in the reaction. [b] Isolated yield of major diastereomer of >99% purity. [c] Ratios determined by HPLC. [d] Yield obtained by using 3–5 equiv of RCHO.

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(3) Mukaiyama, T.; Iwasawa, N.; Stevens, R. W.; Haga, T. *Tetrahedron* **1984**, *40*, 1381–1390. For a detailed procedure for the synthesis of stannous triflate, see: Evans, D. A.; Weber, A. E. *J. Am. Chem. Soc.* **1986**, *108*, 6757–6761. It is imperative that the reagent must be free of triflic acid, which may be effected by carefully washing the salt with anhydrous ether.

(4) The assignment of the *Z* stereochemistry of this enol ether was made through an unambiguous ^1H NMR NOE study.

(5) General stannous triflate mediated aldol procedure: A magnetically stirred suspension of anhydrous, acid-free $Sn(OTf)_2$ (1.05 equiv, ca. 0.25 M in CH_2Cl_2, +25 °C) is treated with triethylamine (1.05 equiv) and then immediately cooled to −20 °C. (Note: the addition of base sometimes causes the initially white suspension to turn pale yellow.) After 5 min, a solution of the β-keto imide (1.00 equiv, 0.35–0.75 M in CH_2Cl_2) is added dropwise over a 5-min period. The resultant suspension is stirred for 1 h and then cooled to −78 °C prior to treatment with freshly distilled aldehyde (1.00 equiv, neat or in CH_2Cl_2). The reaction mixture is stirred at −78 °C for 30 min and then transferred rapidly by cannula to a cool and vigorously stirred 1:1 mixture of CH_2Cl_2/1 N aqueous $NaHSO_4$ (ca. 130 mL/1 mmol of β-keto imide). After 5–10 min of vigorous stirring at 0 °C, the mixture is diluted with additional 1:1 CH_2Cl_2/1 N aqueous $NaHSO_4$, and the aqueous layer is extracted several times with CH_2Cl_2. The combined organic layers are washed with aqueous $NaHCO_3$, dried over Na_2SO_4, and concentrated in vacuo. The product is purified by flash chromatography and/or recrystallization.

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