of ammonium chloride (848 mg, 16 mmol), the ammonia was evaporated under a stream of nitrogen. The light yellow residue was dried overnight at 0.1 mmHg and then dissolved in warm methanol previously deaerated with nitrogen. The mixture was filtered rapidly by suction, and the filtrate was cooled at 5 °C for 1 h. The white solid that precipitated, together with additional solid that resulted from concentration of the mother liquor under vacuum, was collected and washed with water and methanol (yield of **2a**: 305 mg, 40%). An analytical sample was obtained by recrystallization from boiling methanol saturated with nitrogen: mp >350 °C dec; UV λ_{max} 226 nm (50 mM KPi, pH 7.42); ^{1}H NMR ($(CD_3)_2SO$) δ 3.52 (m, 2), 3.74 (m, 2), 7.58 (s, 1), 8.09 (s, 1), 12.00 (br s, 1), 12.50 (br s, 1); FABMS, m/z 189 $(M + 1)^+$; HRFABMS, m/z 189.0782 (calcd for $C_9H_9N_4O$ $(M + 1)^+$, 189.0776). Anal. Calcd for $C_9H_8N_4O \cdot 0.5H_2O$: C, 54.82; H, 4.56; N, 28.42. Found: C, 54.47; H, 4.39; N, 28.15.

***lin*-Benzopurine (imidazo[4,5-*g*]quinazoline) (3)** was obtained by removal of solvent in vacuo from the mother liquor as a dark yellow oil. Flash chromatography (silica gel, acetone) gave **3** as a hygroscopic yellow solid: 136 mg (20%); mp 299–300 °C; ^{1}H NMR ($(CD_3)_2SO$) δ 8.15 (s, 1), 8.42 (s, 1), 8.69 (s, 1), 9.15 (s, 1), 9.68 (s, 1); EI mass spectrum (relative intensity) (10 eV), m/z 170 (M^+, 100); HRFABMS, m/z 171.0668 (calcd for $C_9H_7N_4$ $(M + 1)^+$, 171.0671). Anal. Calcd for $C_9H_6N_4 \cdot 0.5H_2O$: C, 60.33; H, 3.91. Found: C, 60.20; H, 3.61.

***lin*-Benzopurine (3)** was also obtained when *lin*-benzoadenine (**1d**) was subjected to the general Birch reduction conditions described above. Via the treatment with ammonium chloride and evaporation of the ammonia, the resulting yellow solid was extracted three times with boiling methanol. Removal of the solvent in vacuo followed by column chromatography (silica gel, acetone) gave **3** in isolated yield of 52%, identical in all respects to the byproduct of the reduction of *lin*-benzohypoxanthine.

4,9-Dihydro-8-(methylthio)imidazo[4,5-*g*]quinazoline (5). A mixture of 4,9-dihydro-*lin*-benzohypoxanthine (**2a**) (94 mg, 0.5 mmol), purified P_2S_5 (222 mg, 1 mmol), and dry pyridine (5 mL) was heated at reflux for 15 h. During this time, dry H_2S was bubbled slowly through the solution. The reaction mixture was allowed to stand at 5 °C overnight. The precipitated solid was collected by filtration and then treated with warm water (5 mL). The grey crystals that were deposited were washed with water and dried in vacuo to give **4,9-dihydro-8-mercaptoimidazo[4,5-*g*]quinazoline (4)** (57 mg, 56%), slightly contaminated with 8-mercaptoimidazo[4,5-*g*]quinazoline according to TLC (silica gel, acetone–methanol, 5:1); ^{1}H NMR ($(CD_3)_2SO$) δ 3.74 (s, 2), 3.86 (s, 2), 7.78 (s, 1), 8.29 (s, 1), 13.00 (br s, 2).

The crude mixture was used directly for conversion to **5**. Methyl iodide (42 mg, 0.3 mmol) was added to a stirred suspension of the mercapto compound (50 mg, 0.25 mmol) and potassium hydroxide (17 mg, 0.3 mmol) in 50% aqueous methanol (3 mL). Stirring was continued under nitrogen for 1 h. The solid that precipitated was collected by filtration, washed with water (1–2 mL), and dried at 0.1 mmHg for 24 h to give **5** (yield, 44 mg, 82%). Further purification was effected by radial preparative-layer chromatography[19] under nitrogen (silica gel, acetone–methanol, 5:1): mp 305 °C; ^{1}H NMR ($(CD_3)_2SO$) δ 2.51 (s, 3, SCH_3), 3.70 (s, 2), 3.97 (s, 2), 7.66 (s, 1), 8.83 (s, 1), 12.00 (br s, 1); FABMS, m/z 219 $(M + 1)^+$; HRFABMS, m/z 219.0702 (calcd for $C_{10}H_{11}N_4S$ $(M + 1)^+$, 219.0704). This compound was not readily convertible into 4,9-dihydro-*lin*-benzoadenine (**6**) by treatment with ammonia in ethanol in a steel bomb at 110–120 °C, nor could **6** be obtained by fusion of **2a** with phenyl phosphorodiamidate (PPDA).

4,9-Dihydro-*lin*-benzoadenine (6). In a dry 50-mL, three-necked flask, equipped with a mechanical stirrer, a dry ice condenser with a moisture trap, and a septum, methylamine (25–30 mL) was condensed directly under argon from a cylinder. *lin*-Benzoadenine (**1d**) (185 mg, 1 mmol) was dissolved in 10 mL of hot, freshly distilled HMPT and purged with argon. Lithium (27 mg, 4 mmol) was added to the methylamine, followed by the hot solution of **1d** with stirring. The solution of **1d** was added at such a rate that the blue color of the lithium solution persisted. After the addition was complete (30 min), the reaction mixture turned from blue to light yellow. Ammonium chloride (213 mg, 4 mmol) was added slowly, the dry ice suspension was removed from the condenser, and argon was bubbled through the solution to remove as much methylamine as possible. After 2 h, 15 mL of methanol, purged with argon, was added. During the next 2 h, a cream-colored precipitate separated. The precipitate was filtered, washed with 3 mL of methanol, and dried under vacuum to give **6** (44 mg, 23%). The compound was recrystallized from DMF: mp >360 °C; UV λ_{max} 231 and 265 nm (50 mM KPi, pH 7.42); ^{1}H NMR ($(CD_3)_2SO$) δ 3.50 (m, 2), 3.78 (m, 2), 6.75 (s, 2, NH_2, ex), 7.58 (s, 1), 8.21 (s, 1), 12.00 (br s, 1, NH); FABMS, m/z 188 $(M + 1)^+$; HRFABMS, m/z 188.0934 (calcd for $C_9H_{10}N_5$ $(M + 1)^+$, 188.0936).

Enzyme Studies. For the enzyme studies, adenosine deaminase (type VII, from calf intestinal mucosa) and xanthine oxidase (grade I, from buttermilk) were purchased from Sigma Chemical Co.

Adenosine Deaminase. 4,9-Dihydro-*lin*-benzoadenine (**6**) (2.1 mg) was dissolved in 250 mL of 50 mM KPi buffer (pH 7.42), and a 3-mL aliquot of the solution was transferred into a cuvette. A UV spectrum was taken, and the cuvette was placed in a water bath at 25 °C. When the thermal equilibrium was reached, 4 μL of a water solution of adenosine deaminase (100 units/200 μL) was added, the solution was mixed well, and a UV spectrum was taken. The solution was incubated 24 h at 25 °C. No change in the UV spectrum was observed during this time. In a control experiment under the same conditions, adenosine deaminase converted *lin*-benzoadenine (**1d**) into *lin*-benzohypoxanthine (**1a**), as previously reported.[12]

Xanthine Oxidase. *lin*-Benzohypoxanthine (**1a**) and 4,9-dihydro-*lin*-benzohypoxanthine (**2a**) were dissolved separately in 50 mM KPi buffer (pH 7.42) to make 50 μM solutions. In each experiment 1 mL of the substrate solution was mixed with the enzyme (3.2 μL of 13 units/mL suspension) and the change in absorbance (at 300 nm for **1a** and 264 nm for **2a**) was recorded as a function of time. The final absorbance was recorded after 4 h. From a plot of absorbance versus time, the half reaction times were determined. The comparison of the half reaction times showed that **2a** was transformed to **2b** at approximately $^2/_3$ the rate of transformation of **1a** to **1b**. The product of the xanthine oxidase promoted reaction of **2a** was identified by comparison of the UV spectra. 4,9-Dihydro-*lin*-benzoxanthine (**2b**) was not altered by the enzyme under the same enzyme oxidation conditions as described above, proving that it was the final product of the oxidation of **2a** and that further oxidation had not occurred in the imidazole ring.

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(19) Mikes, F.; Boshart, G.; Gil-Av, E. *J. Chromatogr.* **1976**, *122*, 205.

Do Simple Optically Active Phase-Transfer Agents Catalyze Enantioselective Ether Formation?[1]

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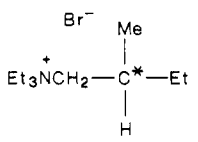
Much evidence has been accumulated showing that enantioselective phase-transfer catalysis (PTC) of reactions is successful only if there is a multipoint interaction be-

(1) Applications of Phase Transfer Catalysis, Part 39. Part 38: Dehmlow, E. V.; Rao, Y. R. *Synth. Commun.* **1988**, *18*, 487.

0022-3263/88/1953-3875$01.50/0

tween the reacting species and the catalyst in the transition state[2] as seems to be the case in enzyme-catalyzed conversions. It was surprising therefore that Verbicky and O'Neil[3] reported enantioselective ether formation with a nonfunctional PT catalyst, (+)-triethyl(2-methylbutyl)-ammonium bromide (**1**). Purported ee values up to 43%



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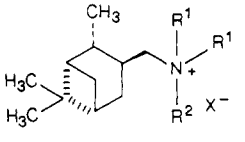

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were recorded for the reaction of eq 1 with a catalyst bearing substituents of as little distinction as ethyl and methyl at a chiral carbon atom which is two atoms removed from the ionic center. *Should this effect be real,*

$$\mathrm{PhCHOHCH_3} \xrightarrow[\mathrm{Me_2SO_4\ (0.5\ equiv),\ room\ temperature}]{\text{50\% NaOH, pentane, catalyst}} \mathrm{PhC^*H(OMe)CH_3} \quad (1)$$

many mechanistic concepts of PTC would need revision as the interaction of anions and cations would be much stronger than suspected hitherto. We have cautioned workers in the field before that impurities or decomposition products of chiral catalysts can mimic optical yields of reactions.[4] Presently, we prepared the sterically more demanding quaternary ammonium salts (+)-triethyl(isopinocampheylmethyl)ammonium bromide (**2a**, $R^1 = R^2 =$ Et, X = Br) and (+)-dibenzylethyl(isopinocampheylmethyl)ammonium bromide (**2b**, $R^1 = CH_2Ph$, R^2 = Et, X = Br) from the commercial amine **2c** ($R^1 = R^2 = H$, X = Cl).[5] Using these as catalysts for the conversion of eq 1 under the conditions of Verbicky,[3] we obtained α-phenethyl methyl ether in 74 and 60% chemical yields, but without any optical activity. Thereafter, the repetition of the original authors' work[3,6] was attempted. Again the ether (obtained in 74% yield) had no rotation when freshly prepared optically active **1** was the catalyst. It must be mentioned, however, that some of the physical data for the very hygroscopic **1** were quite different from the ones published by Verbicky.[3] Our twice-recrystallized **1** had mp 147 °C (from EtOAc/little MeOH) and $[\alpha]^{20}_D$ −0.40° (*c* 7.6, MeCN), the racemic **1** having mp 136 °C, whereas Verbicky's sample had mp 97–98 °C and $[\alpha]^{25}_D$ +3.15 (MeCN). We find that the rotation decreases to almost 0 when a few drops of water are added to the absolute MeCN solvent.[7] ^{13}C NMR spectra of both our (−)-**1** and racemic **1** are almost identical with the values published[3] except for the fact that Verbicky and O'Neil give eight signals whereas structure **1** should have only seven. In unpurified samples of **1** we find a resonance at δ 46.5 (Verbicky gives δ 46.13) and another one at δ 9.0, both of which disappear after repeated recrystallization. Another curious divergence is in the rotation of the expected product. When (*S*)-(−)-1-methoxy-1-phenylethane was prepared by phase-transfer catalysis from commercial (*S*)-(−)-1-phenylethanol ($[\alpha]^{26}_D$ −38.1° (neat); 94.3% ee[8]), the ether had $[\alpha]^{26}_D$ −114.0° (neat), $[\alpha]^{25}_D$ −117.3° (*c* 0.8; MeCN), and $[\alpha]^{25}_D$ −118.0° (*c* 7, MeCN). As the highest known rotation of the (+) enantiomer is $[\alpha]^{25}_D$ +120° (neat),[9] there cannot be any racemization during our workup. Verbicky and O'Neil give $[\alpha]^{25}$ +38.3° (MeCN, no concentration given) for a product they presumed to be optically pure.[3] Furthermore, we observed no dependence of product rotation upon the reaction temperature during dimethyl sulfate addition, and there was no racemization of the ether once formed by traces of acid.

We must conclude that the apparent optical yields reported[3] should be attributed to some byproduct introduced from use of an impure catalyst. Cursory investigations into the more obvious possible contaminants did not result in any explanation of the literature rotations. It must be stressed again that the utmost care is necessary in the execution of chiral PTC reactions and in the interpretation of the results.[2a,4]

Experimental Section

Equipment and Material. ^1H (300 MHz) and ^{13}C (75 MHz) NMR spectra were recorded on a Bruker AM 300 instrument in $CDCl_3$. Chemical shifts δ are relative to TMS. Acetonitrile was dried by refluxing over P_2O_5 or CaH_2 or 5 h and subsequent distillation.

(*S*)-(+)-Triethyl(2-methylbutyl)ammonium Bromide (1). A solution of 5.0 g (33 mmol) of (*S*)-1-bromo-2-methylbutane[6] and 3.4 g (33 mmol) of triethylamine in 50 mL of absolute CH_3CN was refluxed for 24 h. The solvent was evaporated and the residue was washed with EtOAc, filtered, and dried at 0.01 Torr/room temperature: yield 6.2 g (74%); mp 127–128 °C. Twice-recrystallized material (EtOAc/trace MeOH) after extensive drying had mp 147 °C, $[\alpha]^{25}_D$ −0.40° (*c* 7.6, MeCN). The compound is very hygroscopic, and its rotation is very dependent on the dryness of the solvent.[7] ^{13}C NMR: δ 64.0 (t), 53.9 (t), 29.6 (t), 29.0 (d), 19.7 (q), 11.1 (q), 8.3 (q). Racemic **1**, mp 136 °C, has a virtually identical spectrum.

(+)-Triethyl(isopinocampheylmethyl)ammonium Bromide (2a). A mixture of 10.17 g (50 mmol) of (+)-3-(aminomethyl)-pinane hydrochloride,[5] 142 g (1.3 mol) of ethyl bromide, and 100 g (0.72 mmol) of potassium carbonate was refluxed for 7 days. The solid was filtered and washed with CH_2Cl_2. The organic phase was evaporated, and the residue was dissolved in 10 mL of absolute methanol. Precipitation with absolute ether was followed by suspension of the solid in 50 mL of toluene and stirring of the suspension at 100 °C for 2 h. After cooling to 0 °C, the solid was filtered off and dried at 0.01 Torr/room temperature: mp 175 °C; 4.15 g (25%) yield; $[\alpha]^{20}_D$ +39.6° (*c* 1.5, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.0–2.5 (m, 26 H), 3.5 (q, 8 H). Anal. Calcd for $C_{17}H_{34}BrN$ (332.4): C, 61.43; H, 10.31; N, 4.21. Found: C, 61.54; H, 10.54; N, 3.96.

(+)-Dibenzylethyl(isopinocampheylmethyl)ammonium Bromide (2b). A mixture of 4.37 g (21.3 mmol) of (+)-3-(aminomethyl)pinane hydrochloride,[5] 17 g (135 mmol) of benzyl chloride, and 18.6 g (135 mmol) of K_2CO_3 was refluxed in 50 mL of acetonitrile for 2 weeks. After cooling, filtration, and washing of the solid with CH_2Cl_2, the organic phase was concentrated and fractionated. At 120 °C/0.005 Torr, 6.8 g (19.6 mmol) of dibenzyl(pinan-3-ylmethyl)amine was obtained. This was heated

(2) (a) Review: Dehmlow, E. V.; Dehmlow, S. S. *Phase* Transfer *Catalysis*, 2nd ed.; Verlag Chemie: Weinheim, 1983; p 69 ff. (b) For recent striking examples, cf.: Bhattacharya, A.; Dolling, U.-H.; Grabowski, E. J. J.; Karady, S.; Ryan, K. M.; Weinstock, L. M.; *Angew. Chem.* **1986**, *98*, 442; *Angew. Chem., Int. Ed. Engl.* **1986**, *25*, 476. Dolling, U.-H.; Hughes, D. L.; Bhattacharya, A.; Ryan, K. M.; Karady, S.; Weinstock, L. M.; Grabowski, E. J. J. In *Phase Transfer Catalysis: New Chemistry, Catalysts, and Applications*; Starks, C. M., Ed. ACS Symposium Series 326; American Chemical Society: Washington, DC, 1987; p 67.

(3) Verbicky, J. W., Jr.; O'Neil, E. A. *J. Org. Chem.* **1985**, *50*, 1786.

(4) Dehmlow, E. V.; Singh, P.; Heider, J. *J. Chem. Res., Synop.* **1981**, 292.

(5) Available from BASF, Ludwigshafen, Germany, and from Merck-Schuchardt, Darmstadt, Germany. $[\alpha]^{26}_D$ + 41.5° (*c* 3.6, MeOH). Highest known rotation $[\alpha]^{25}_D$ +44.5° (*c* 4, MeOH) (Paust, J.; Pfohl, S.; Reif, W.; Schmidt, W. *Justus Liebigs Ann. Chem.* **1978**, 1024).

(6) Our starting material (*S*)-(+)-1-bromo-2-methylbutane (Aldrich) had $[\alpha]^{20}_D$ +3.9° (neat); 4.9° (*c* 5.3, MeCN); 4.5° (*c* 5, $HCCl_3$). The highest known value is 4.69° (neat) (Frohardt, R. P.; Dion, H. W.; Jakubowski, Z. L.; Ryder, A.; French, J. C.; Bartz, C. R. *J. Am. Chem. Soc.* **1959**, *81*, 5500). Verbicky and O'Neil apparently used starting material of *lesser* rotation.

(7) Addition of one drop of water to the MeCN solution gives $[\alpha]^{25}_D$ −0.25° (*c* 7.6, MeCN); in water $[\alpha]^{25}_D$ +0.01° (*c* 8.0, H_2O) is found.

(8) Product from Aldrich. Maximum known rotation: $[\alpha]^{25}_D$ +40.4° (neat) Mislow, K. *J. Am. Chem. Soc.* **1951**, *73*, 4043.

(9) Cram, D. J.; Kingsbury, C. C.; Rickborn, B. *J. Am. Chem. Soc.* **1959**, *81*, 5835.

with 58 g (270 mmol) of ethyl bromide in 40 mL of acetonitrile in an autoclave at 100 °C for 2 days. The solvent was evaporated, and the residue was washed with ether and ethyl acetate successively. Recrystallization from acetonitrile yielded 580 mg (6%) of **2b** hydrate: mp 211 °C; $[\alpha]^{25}_D$ +55.6° (*c* 2.2, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 0.8–3.0 (m, 20 H), 4.0–4.9 (m, 8 H), 7.3–7.7 (m, 10 H), 10.0 (br, 2 H). Anal. Calcd for $C_{27}H_{38}NBr \cdot H_2O$ (474.5): C, 68.34; H, 8.43; N, 2.95. Found: C, 64.18; H, 8.15; N, 2.92.

Phase Transfer Catalytic Ether Formation. To a mixture of 10 g of 50% NaOH, 40 mL of petroleum ether (boiling range 30–60 °C), 4.88 g (40 mmol) of 1-phenylethanol, and 0.4 mmol of catalyst was added 2.52 g (20 mmol) of Me_2SO_4 (40 mmol in the case of optically active compound). Thereafter a slight exothermic reaction occurred. The mixture was stirred at room temperature for 1 h, 8 mL of 50% aqueous ammonia was added, and the mixture was stirred again for 5 min. The phases were separated, and the organic layer was washed three times with 20-mL portions of H_2O and dried over Na_2SO_4. Removal of the solvent and fractional distillation gave the ether, bp 68 °C/16 Torr. Yields with 1, **2a**, **2b** as catalysts: 74, 60, or 74%, respectively, $[\alpha]^{25}_D$ 0.0° (*c* 13.6, MeCN). Yield with optically active alcohol and tetrabutylammonium chloride as catalyst: 82%, $[\alpha]^{26}_D$ −114.0° (neat) (in solution, see text).

Acknowledgment. We thank the BASF for the donation of **2c**. Our research was supported by Deutsche Forschungsgemeinschaft and Fonds der Chemischen Industrie.

Mechanistic Study of the Wittig Reaction of Benzophenone with a Nonstabilized Ylide

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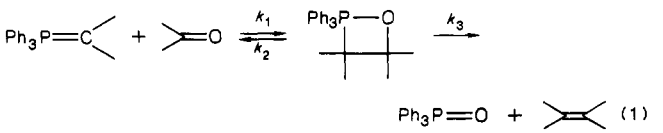
The Wittig reaction is one of the most important reactions in organic synthesis, and extensive studies have been performed to elucidate the mechanism.[1] In spite of the amount of investigation, however, some important points still remain unclear, e.g., the origin of cis preference in reactions of nonstabilized ylides, the intermediacy of betaine, and the nature of the rate-determining step of the multistep reaction. Reactions of nonstabilized ylides with simple aldehydes or ketones have been shown by ^{31}P NMR to go through oxaphosphetanes as an only detectable intermediate.[2,3] Thus, ethylidenetriphenylphosphorane reacted with cyclohexanone at −78 °C in THF to give a sharp signal, attributable to oxaphosphetane, at 66.5 ppm relative to external 85% phosphoric acid. At higher temperature above −15 °C, the oxaphosphetane was converted into triphenylphosphine oxide.[2] This finding as well as others was interpreted in terms of the scheme shown in eq 1; oxaphosphetane forms rapidly and then decomposes to phosphine oxide and alkene in a slow step. In the



(1)

reaction of benzaldehyde and *n*-butylidenetriphenylphosphorane, both *cis*- and *trans*-oxaphosphetanes have been observed and the rates of decomposition of these intermediates have been determined by the ^{31}P NMR method.[3] The intermediacy of the betaine species was questioned for the reactions of nonstabilized ylides.

However, studies by use of ^{31}P NMR are so far limited to the reaction systems in which the oxaphosphetane is far more stable than the reactants (ylide + aldehyde or ketone) and therefore only the decomposition of the oxaphosphetane could be followed kinetically. In spite of that, the oxaphosphetane formation step was suggested to be crucial in determining the stereochemistry of the overall reaction. In the present paper, we report the results of an NMR study for the reaction of benzophenone with a nonstabilized ylide, wherein both formation and decomposition of oxaphosphetane could be monitored for the first time. Substituent and kinetic isotope effect (KIE) experiments were also carried out, which are known to be useful in obtaining information on the rate-determining step of reactions.

A THF solution of isopropylidenetriphenylphosphorane (0.1 M), prepared from isopropyltriphenylphosphonium iodide and sodium hexamethyldisilazide, was placed in a flame-dried NMR tube and cooled by liquid N_2. To this was added a benzophenone solution, and the tube was sealed.[4] The THF solution containing ylide (0.052 M), benzophenone (0.075 M), and THF-d_8 (8.3% v/v, internal D-lock) was then allowed to react at 0 °C and examined at 40.5 MHz FT-NMR with proton noise decoupling. Figure 1 shows the time dependence of the spectrum. The signal at −55.3 ppm was assigned to oxaphosphetane from analogy with the literature values.[2,3] The other five signals were assigned by using authentic samples and/or by ^1H NMR analysis to those of triphenylphosphine (external standard, adjusted to −5.5 ppm), ylide (10.8), triphenylphosphine oxide (27.3), isopropyltriphenylphosphonium iodide (33.1), and isopropyldiphenylphosphine oxide (36.9). No peak attributable to betaine was observed, although a very short-lived betaine may escape detection.

From the results in Figure 1 the kinetic analysis was made and illustrated in Figure 2. The variation of oxaphosphetane in Figure 2 suggests that the reaction proceeds consecutively and that the olefin-formation step is not a single rate-determining step in sharp contrast to the reaction of cyclohexanone.[2] Thus, the reaction profile for benzophenone is quite different from that for cyclohexane or various aldehydes. By assuming the reaction scheme of eq 1, we carried out computer simulation of the plots with k_1, k_2, and k_3 being adjustable parameters. As Figure 2 shows, theoretical lines fit nicely with the experimental points. A series of parameter sets were found to reproduce the observed data. The conclusions obtained from the simulation are (1) k_3 is in the order of 7×10^{-4} s^{-1}, (2) k_1

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(2) Vedejs, E.; Snoble, K. A. J. *J. Am. Chem. Soc.* **1973**, *95*, 5778. Vedejs, E.; Meier, G. P.; Snoble, K. A. J. *J. Am. Chem. Soc.* **1981**, *103*, 2823.

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(4) Olah et al. have reported that the reaction of isopropylidenetriphenylphosphorane, prepared with *n*-BuLi as a base, with benzophenone gave benzhydrol in addition to the expected alkene product in toluene. The formation of the reduced product was ascribed to an electron-transfer mechanism. We repeated the experiments and indeed obtained benzhydrol although in a lower yield. However, when PhLi or sodium hexamethyldisilazide was used in place of *n*-BuLi, the reaction gave the expected alkene exclusively either in toluene, ether, or THF, and the material balance in these reactions was excellent: Olah, G. A.; Krishnamurthy, V. V. *J. Am. Chem. Soc.* **1982**, *104*, 3987.

0022-3263/88/1953-3877$01.50/0