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Lithium hydroxide as base in the Wittig reaction. A simple method for olefin synthesis

R. Antonioletti [a,*], F. Bonadies [b,*], A. Ciammaichella [b], A. Viglianti [a]

[a] C.N.R. Istituto di Chimica Biomolecolare-Sezione di Roma c/o Dip. di Chimica, Università di Roma ''La Sapienza'', P.le A.Moro 5, I-00185, Roma, Italy
[b] Dipartimento di Chimica, Università di Roma ''La Sapienza'', P.le A. Moro 5, I-00185, Roma, Italy

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Abstract

A mild and practical procedure for the Wittig olefination, promoted by lithium hydroxide and triphenylbenzyl phosphonium bromide, has been set up for the synthesis of stilbenes and styrenes. The experimental conditions allow aromatic, heteroaromatic, unsaturated and saturated aliphatic aldehydes to give final products in good yields.

1. Introduction

Although lithium hydroxide has been widely employed in organic synthesis for the hydrolysis of acid derivatives, few reports have been published for carbanion formation. In 1992, Scettri and co-workers showed that active methylene compounds such as β-diketones, β-keto esters and β-diesters are deprotonated by LiOH to yield the corresponding lithium salts. This methodology allows alkylation with halides,[1] or Michael addition with enones.[2] Later on, the same authors extended this technique to the Horner–Wadsworth–Emmons reaction since this alkaline base can deprotonate organo-phosphorus compounds such as phosphonoacetate,[3] ketophosphonates[4] and cyanophosphonates;[5] the reaction with aldehydes and ketones gives olefins in very good yields. Moreover, in 1998, Takacs and co-workers published the synthesis of (*E*,*E*)-α,β-γ,δ-unsaturated esters by reaction of 4-phosphonocrotonate with LiOH and carbonyl compounds.[6] They showed that LiOH provides higher yields than LDA (LiOH=86%, LDA=70%).

The pK_a values of the active methylene compounds[7] and the organo-phosphonates[8] (Fig. 1) suggest that LiOH, also in the case of benzyl triphenylphosphonium bromide (pK_a=12.6),[9] must be able to generate an ylide for the Wittig reaction.[10] Therefore, benzyl triphenylphosphonium bromide (TPBPB) and aromatic aldehydes were subjected to the action of a slight excess of LiOH and the effect of different solvents, under the conditions given in Table 1, was examined.

2. Results and discussion

As shown, isopropyl alcohol seems to be the best among the solvents examined, producing high yields of stilbenes. The other oxygenated solvents either achieved lower conversions or required longer reaction times. Under the same conditions we investigated the reaction of *p*-methylbenzaldehyde with TPBPB at various temperatures and found that reflux temperature was optimum. At lower temperature, lower chemical yields were observed without any improvement of *E*/*Z* ratio. Using these reaction conditions, as shown in Tables 2 and 3, aldehydes (aromatic and heteroaromatic) are smoothly converted into the corresponding olefins in high yields, but as *E*/*Z* mixtures.

Moreover, there is no significant influence of ring substituents. In the case of entry g (Table 2), the low yield could be due to the phenoxy ion that hampers the ylide attack on to the carbonyl.

When we extended the procedure to alkyl aldehydes (Table 4), the reaction times were longer according to their steric hindrance. The stereoselectivity, low in all cases, is independent of steric effects. Only in the case of entry d, the *Z*-isomer was the major product because of the effect of twisting of

* Corresponding authors.
E-mail address: francesco.bonadies@uniroma1.it (F. Bonadies).

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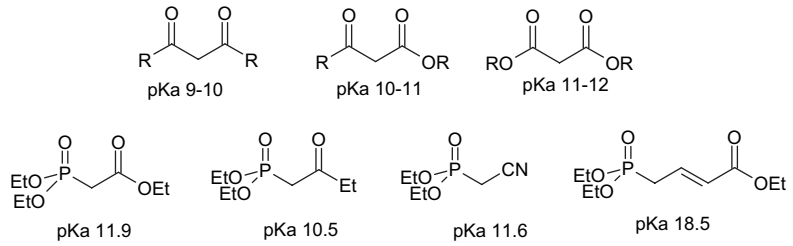
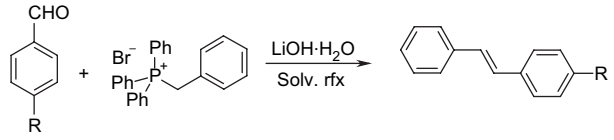


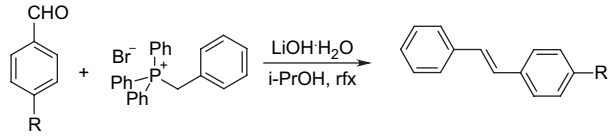
Figure 1.

Table 1
Wittig reaction using various solvents



Entry	R	Solvent	Time (h)	Yield (%)	*E*/*Z*
a	H	THF	3	70	17/83
b	H	DME	3	94	45/55
c	H	Dioxan	2	80	33/67
d	H	MeOH	0.25	66	50/50
e	H	*i*-PrOH	0.75	96	47/53
f	CH_3	THF	3	70	34/66
g	CH_3	DME	7	>98	42/58
h	CH_3	Dioxan	7	>98	44/56
i	CH_3	MeOH	24	55	45/55
j	CH_3	*i*-PrOH	0.75	>98	39/61

Table 2
Wittig reaction with *p*-substituted benzaldehydes



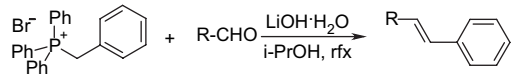
Entry	R	Time (h)	Yield (%)	*E*/*Z*
a	H	0.75	96	47/53
b	CH_3	0.75	>98	39/61
c	OCH_3	1	>98	20/80
d	NO_2	0.75	>98	35/65
e	Br	0.75	>98	44/56
f	$N(CH_3)_2$	2	89	58/42
g	OH	24	21	47/53
h	3,5-OCH_3	0.75	>98	44/56

aromatic ring in respect to the plane of double bond that is smaller than a steric interaction between the vinylic hydrogen and the *tert*-butyl group in *E*-isomer. In no case we have noticed by-product deriving from aldol condensation that, normally, benzyl aldehyde suffers in alkaline medium.

In the case of the most reactive α,β-unsaturated aldehydes (Table 5), we found 25 °C as the best temperature to run the experiments. In effect, at this temperature, the substrates (except for entry c) were converted into the final dienyl products in an acceptable time, good yields and without by-products deriving from conjugate addition.

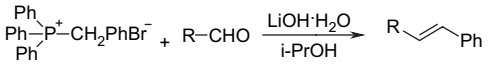
To test the ability of LiOH to deprotonate phosphonium salts having different values of pK_a, we treated all of them

Table 3
Synthesis of 2-heteroarylstyrenes



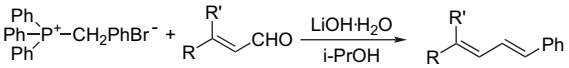
Entry	R	Time (h)	Yield (%)	*E*/*Z*
a		0.75	>98	56/44
b		0.75	>98	51/49
c		0.75	95	55/45

Table 4
Synthesis of β-alkylstyrenes



Entry	R	Temperature (°C)	Time (h)	Yield (%)	*E*/*Z*
a	*n*-C_8H_{17}	Reflux	1.5	86	51/49
b	*c*-C_6H_{11}	Reflux	3	80	57/43
c	$CH(CH_3)_2$	50	4.5	95	67/37
d	$C(CH_3)_3$	70	5	74	27/73
e	CH_2–Ph	Reflux	1	80	43/57

Table 5
Synthesis of dienes

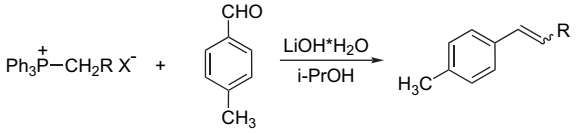


Entry	R	R^1	Temperature (°C)	Time (h)	Yield (%)	*E*/*Z*
a	CH_3	H	0	12	65	48/52
b	CH_3	H	25	1	41	45/55
c	CH_3	H	Reflux	<0.5	—	—
d	CH_3	CH_3	25	5	86	46/54
e	Ph	H	25	5	81	52/48

with LiOH in isopropyl alcohol and reacted them with 4-methyl-benzaldehyde (Table 6).

The results prove, as expected, that lithium hydroxide is able to generate ylides deriving from phosphonium salts having pK_a<12.6 (entry a, b) and pK_a=12.6. When the reactions are performed with salts having pK_a>12.6, we obtain olefins in low yield at room temperature, whereas, the reactions afford the corresponding alkyl-diphenyl-phosphinoxides at reflux. The low reactivity of ethyl triphenylphosphonium bromide

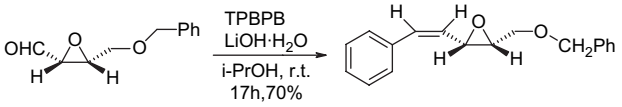
Table 6
Wittig reaction using various salts



Entry	R	Temperature (°C)	Time (h)	Yield (%)	*E*/*Z*
a	CN	rt	1.5	>98	55/45
b	$COCH_3$	rt	3	>98	10/90
c	C_6H_4–*p*-OMe	Reflux	6	95	20/80
d	C_6H_4–*p*-NO_2	Reflux	0.5	>98	35/65
e	CH_3	rt	42	30	80/20
f	$CH(CH_3)_2$	rt	27	10	80/20

(entry e) was confirmed by performing the reaction in different solvents; the better yield (41%) was obtained with a biphasic mixture (H_2O/CH_2Cl_2 1/1), only after a 4-day reaction.

In Scheme 1, we report a further example, as conclusive test of the wide versality of this protocol. 4-Benzyloxy-2,3-epoxybutanal was stirred under the reaction conditions for 17 h. The product, purified by chromatography, was obtained in a 70% yield. The ^{1}H NMR spectrum[11] showed exclusive formation of the *Z*-isomer. The configuration was assigned from the coupling constant between olefinic protons (J=11.7 Hz). Furthermore, the signals due to the epoxy protons at 3.4–3.9 ppm indicated no oxirane ring opening.



Scheme 1.

3. Conclusion

In conclusion, in spite of low stereoselectivity, LiOH has been shown to be an efficient base[12] in the Wittig reaction since it is able to extract the proton from the benzyl triphenylphosphonium bromide reagent giving the ylide. Reaction of this ylide with aldehydes of various type (aromatic, heteroaromatic, saturated and α,β-unsatured alkyl) provides olefin derivatives in high yields. Moreover, this method can be applied also to highly functionalised aldheydes with good results. No side reactions such as condensation, conjugate addition or Cannizzaro reaction were observed. Lithium hydroxide has the further advantage of low cost, stability and low toxicity. Unfortunately, ketones do not undergo the Wittig reactions; under these experimental conditions we obtained only low yields (<30%) with acetophenone, cyclohexanone and 2-pentanone.

4. Experimental section

4.1. General

^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ with either Varian XL-300 or Varian 'Gemini' 200-MHz instruments, while IR spectra were recorded in CCl_4 and $CHCl_3$ with a Shimadzu IR-740 instrument. HRMS spectra were recorded by Micromass Q-TOF micro. MS spectra were recorded by an HP5971A/MS detector coupled with an HP5890 gas chromatograph. The stereoisomeric ratios were determined on HP5880 and HP5890 gas chromatographs equipped with capillary columns. Column chromatography was carried out on Kieselgel Merck (70–230 mesh and 230–400 mesh).

4.2. General procedure

Lithium hydroxide (1.6 mmol) was added to a stirred solution of phosphonium salt (1.2 mmol) in isopropyl alcohol (4 mL). After 15 min aldehyde (1.1 mmol) was added to the mixture and the reaction was carried out at several temperatures (Tables 1–6). The reaction was continued until complete consumption of the aldehyde, which is monitored by TLC and GC, then quenched with water and extracted with ethyl acetate (3×50 mL). The organic phase was washed with brine until neutrality, dried with Na_2SO_4 and evaporated in vacuum. The residue was purified by flash chromatography (SiO_2) eluting with hexane/Et_2O to afford the product as a mixture of two isomers. The isomeric ratios were determined by GC and their structures were confirmed by spectral data. All alkene products are known and their spectroscopic data are identical to that reported in the literature, except for (*Z*)-4-methoxy-4′ methyl stilbene, (*Z*)-1-(*p*-tolyl)-3-methyl-1-butene and (*E*)-1-(*p*-tolyl)-3-methyl-1-butene.

4.2.1. (Z)-Stilbene and (E)-stilbene
See Refs. 13 and 14, respectively.

4.2.2. (Z)-4-Methoxy-stilbene and (E)-4-methoxy-stilbene
See Refs. 15 and 16, respectively.

4.2.3. (Z)-4-Nitro-stilbene and (E)-4-nitro-stilbene
See Refs. 17 and 18, respectively.

4.2.4. (Z)-4-Methyl-stilbene and (E)-4-methyl-stilbene
See Ref. 14.

4.2.5. (Z)-4-Bromo-stilbene and (E)-4-bromo-stilbene
See Ref. 14.

4.2.6. (Z)-4-Hydroxy-stilbene and (E)-4-hydroxy-stilbene
See Refs. 19 and 20.

4.2.7. (Z)-4-(N,N-Dimethylamino)-stilbene and (E)-4-(N,N-dimethylamino)-stilbene
See Ref. 21.

4.2.8. (Z)-3,5-Dimethoxy-stilbene and (E)-3,5-dimethoxy-stilbene
See Ref. 22.

4.2.9. (Z)-2-Styryl-furan and (E)-2-styryl-furan
See Refs. 23 and 16, respectively.

4.2.10. (Z)-3-Styryl-thiophene and (E)-3-styryl-thiophene
See Ref. 24.

4.2.11. (Z/E)-2-Styryl-pyridine
See Refs. 25 and 26.

4.2.12. (Z)-Non-1-enyl-benzene and (E)-non-1-enyl-benzene
See Refs. 27 and 28, respectively.

4.2.13. (Z)-(2-Cyclohexyl-vinyl)-benzene and (E)-(2-cyclohexyl-vinyl)-benzene
See Ref. 29.

4.2.14. (Z)-3-Methyl-1-phenyl-1-butene and (E)-3-methyl-1-phenyl-1-butene
See Refs. 30 and 31, respectively.

4.2.15. (Z)-3,3-Dimethyl-1-phenyl-1-butene and (E)-3,3-dimethyl-1-phenyl-1-butene
See Refs. 24 and 32, respectively.

4.2.16. (Z)-1,3-Diphenyl-propene and (E)-1,3-diphenyl-propene
See Refs. 33 and 34, respectively.

4.2.17. (1Z,3E)-1-Phenyl-1,3-pentadiene and (1E,3E)-1-phenyl-1,3-pentadiene
See Refs. 35 and 36, respectively.

4.2.18. (Z)-4-Methyl-1-phenyl-1,3-pentadiene and (E)-4-methyl-1-phenyl-1,3-pentadiene
See Ref. 37.

4.2.19. (1Z,3E)-1,4-Diphenyl-1,3-butadiene and (1E,3E)-1,4-diphenyl-1,3-butadiene
See Ref. 38.

4.2.20. (Z)-4-Methyl-cinnamonitrile and (E)-4-methyl-cinnamonitrile
See Ref. 39.

4.2.21. (Z/E)-4-(p-Tolyl)-3-buten-2-one
See Ref. 40.

4.2.22. (Z)-4-Methoxy-4′-methyl-stilbene
^{1}H NMR ($CDCl_3$), δ (ppm): 7.24 (2H, d, J=8.8 Hz), 7.2 (2H, d, J=8.1 Hz), 7.17 (2H, d, J=8.06 Hz), 6.7 (2H, d, J=8.80 Hz), 6.5 (2H, s), 3.78 (3H, s), 2.32 (3H, s). ^{13}C NMR, δ (ppm): 158.8, 130.2, 130, 129.3, 129.4, 128.9, 127.7, 127.5, 126.3, 113.8, 55.13, 21.4. IR, ν (cm^{-1}): 3047, 1572. HRMS calcd for $C_{16}H_{16}O$ (M^+), 224.3024. Found: 224.3038.

4.2.23. (E)-4-Methoxy-4′-methyl-stilbene
See Ref. 41.

4.2.24. (Z)-4-Methyl-4′-nitro-stilbene and (E)-4-methyl-4′-nitro-stilbene
See Ref. 42.

4.2.25. (Z)-1-(p-Tolyl)-1-propene and (E)-1-(4-tolyl)-1-propene
See Refs. 43 and 44, respectively.

4.2.26. (Z)-1-(p-Tolyl)-3-methyl-1-butene
^{1}H NMR ($CDCl_3$), δ (ppm): 7.3 (2H, d, J=8.1 Hz), 7.1 (2H, d, J=8.1 Hz), 6.4 (1H, br d, J=12.2 Hz), 6.2 (1H, dd, J=12.2 Hz, J=6.5 Hz), 2.6–2.8 (1H, m), 2.3 (3H, s), 1.9 (6H, dd, J=1.5 Hz, J=6.5 Hz). ^{13}C NMR, δ (ppm): 137.6, 132.3, 132.3, 129, 126.3, 125.8, 25.3, 24.3, 24. HRMS calcd for $C_{12}H_{16}$ (M^+), 160.2590. Found: 160.2612.

4.2.27. (E)-1-(p-Tolyl)-3-methyl-1-butene
^{1}H NMR ($CDCl_3$), δ (ppm): 7.3 (2H, d, J=8.1 Hz), 7.1 (2H, d, J=8.1 Hz), 6.3 (1H, d, J=16.2 Hz), 6.12 (1H, dd, J=16.2 Hz, J=6.5 Hz), 2.4–2.5 (1H, m), 2.31 (3H, s), 1.1 (6H, d, J=6.5 Hz). ^{13}C NMR, δ (ppm): 137.6, 132.3, 129, 126.3, 125.9, 31.3, 24.3, 24. HRMS calcd for $C_{12}H_{16}$ (M^+), 160.2590. Found: 160.2698.

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11. ^{1}H and ^{13}C NMR spectra were performed in $CDCl_3$ using a Varian XL-300 spectrometer. MS spectra were recorded with an HP5971A/MS detector coupled with an HP5890 GC. **(Z)-5-Phenyl-2,3-epoxy-4-penten-1-ol benzyl ether**: colourless oil. ^{1}H NMR ($CDCl_3$), δ (ppm): 7.5–7.3 (10H, m), 6.80 (1H, d, J=11.7 Hz), 5.52 (1H, dd, J=7.3, 11.7 Hz), 4.65 (2H, s), 3.9–3.6 (4H, m). ^{13}C NMR, δ (ppm): 137.9,

135.7, 129.9, 128.5, 127.8, 125.6, 68.7, 57.6, 53.0. MS, *m/z*: 266 (100), 248 (10.3), 218 (16.9), 199 (17.3), 189 (33.7), 183 (16.9), 145 (44.6), 91 (52.4), 77 (23.3).

12. Some doubts were raised about the role of LiOH in the reaction mechanism. In fact, it was proposed that *i*-PrOLi could behave as a base in place of LiOH. In order to verify this hypothesis, the following two experiments were carried out. In the first experiment, 1.6 mL of butyl lithium (2.5 M solution in hexane, 4.0 mmol) was added dropwise to 10 mL of isopropyl alcohol at 0 °C to obtain a precipitate. After few minutes the solvent was removed in vacuo and the residue dissolved in 10 mL of anhydrous DMF. Then, 4 mmol of benzyl bromide was added and the solution was stirred at room temperature for 15 min. The reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4 and the solvent removed in vacuo to obtain the expected isopropyl benzyl ether as a single product that was characterised by ^1H and ^{13}C NMR. In the second experiment 4.0 mmol of LiOH were dissolved in 10 mL of isopropyl alcohol and the solution was refluxed for 1 h. The solvent was removed in vacuo and the residue dissolved in 10 mL of anhydrous DMF. Then 4 mmol of benzyl bromide were added and the solution refluxed for 3 h. The reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na_2SO_4 and the solvent removed in vacuo to obtain benzyl alcohol as a single product that was characterised by ^1H and ^{13}C NMR.
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