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SURFACE-MEDIATED SOLID PHASE REACTION. PART 9.¹ A CONVENIENT PROCEDURE FOR ALDOL REACTION OF KETENE SILYL ACETALS WITH ALDEHYDES ON THE SOLID SURFACE OF ALUMINA

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Abstract: The aldol reaction of ketene silyl acetals with aldehydes proceeds efficiently on the solid surface of alumina impregnated with anhydrous zinc chloride under sonication providing aldol products in high yields and with good stereoselectivity.

The stereoselective formation of carbon - carbon bonds constitutes an important goal of modern synthetic chemistry towards construction of complex molecules. The aldol reaction of silyl enolates with aldehydes , introduced by Mukaiyama,² is considered one of the most useful reactions in achieving this goal. Thus various reagents have been developed to make this reaction more useful in relation to acyclic streocontrol involving silyl enol ethers ³ as well as ketene silyl acetals.⁴ In continuation of our programme to highlight the potential of surface - mediated solid phase reaction technology ⁵ we have observed a clean aldol reaction of ketene

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silyl acetals with aldehydes on the solid surface of alumina impregnated with anhydrous zinc chloride under sonication (Scheme 1).



The experimental procedure is very simple. Zinc chloride impregnated alumina was added to a mixture of ketene silyl acetals and aldehydes and mixed uniformly. This mixture was then sonicated under nitrogen for a certain period of time as required to complete the reaction (monitored by tlc). The solid mass was then worked up as in the experimental section to give the crude product which was purified by column chromatography over silica gel. Several structurally varied ketene silyl acetals underwent smooth reaction with a wide range of aromatic and alephatic aldehydes by this procedure. The results are presented in Table 1. The reactions are reasonably fast and yields are high. The reactions on the surface of alumina under sonication without zinc chloride is rather slow and thus take a long time to be completed, but the presence of zinc chloride accelerates the reaction remarkably. On the other hand, without sonication the reaction is sluggish. Thus solid surface of alumina impregnated with zinc chloride and ultrasound are two important contributors towards easy progress of the reaction. A general trend for anti diastereoselectivity was observed in all the reactions irrespective of the nature of the silvl enol ethers and aldehydes. The products have been separated by careful column chromatography to provide pure anti and syn diastereoisomers which are of considerable synthetic potential.

To conclude, the present procedure on the solid surface of alumina provides an efficient methodology for Mukaiyama aldol reaction of ketene silyl acetals with aldehydes. The notable advantages associated with this procedure are: (a) operational simplicity; (b) relatively fast reaction; (c) high yields; (d) use of environmentally safe reagents and (e) above all, access to both anti and syn diastreoisomers. Certainly these features would make this procedure a better alternative to many existing methods^{3,4} and we believe, this will find significant application in organic synthesis.

EXPERIMENTAL

Melting points were determined on a glass disk with an electrical bath (Reichart, Austria) and are uncorrected . ¹ H NMR spectra were recorded at 60, 100, 200 and 400 MHz in CCl₄ or CDCl₃ solutions as internal standard. IR spectra were recorded on a Perkin Elmer 298 spectrometer. Elemental analyses were performed by Mr. S. Sarkar of this laboratory. Thin layer chromatography was done on precoated silica gel plates (Sigma). Alumina (Aluminium oxide, acidic, Brockmann activity grade 1 for column chromatography), supplied by SRL, India, was used in all the reactions. Silica gel (60-120 mesh) used for column chromatography was also from SRL, India. All commercial liquid chemicals were distilled before use. An Ultrasonic bath (Julabo USR-3 manufactured by Julabo Labortechnik, Germany, 50 Hz) was used for sonication.

entry	ketene silyl acetal	aldehyde	time(h)	Product	yield(%)a	syn/antib
1		PhCHO	4	MeO ₂ C Me H	^ր h ₈₇	
2	1	≻ -сно	2	MeO ₂ C Me MeO ₂ C OH	~ 86	
3	1	СНО	3	MeO ₂ C Me	<u>∧</u> 80	
4	1	∕сно	2	MeO ₂ C Me OH	70	
5	1	Ph CHO	3.5	MeO ₂ C Me	Ph 80	
6		PhCHO	5	OH CO ₂ Me	75	
7	2	≻ сно	3		77	
8	2	СНО	4	CO ₂ Me	77	
9	PhOMeOSiMe3	РЬСНО	8	Ph $\sim Ph$ CO ₂ Me	80	35:65
10		PhCHO	4		h 78	47:53

Table 1

Reaction of Ketene Silyl Acetals with Aldehydes on the surface of Alumina



Table 1 Continued

^a all yields refer to pure isolated products. ^b syn/Anti ratio was calculated from ¹H nmr analysis.

Alumina (1.5 g) was activated by heating at 200^oC for 4 h under vacuum followed by cooling under nitrogen and was then stirred with a solution of anhydrous zinc chloride (136 mg, 1 mmol, Fluka) in THF (2 ml) for 5 min after which the excess THF was removed under reduced pressure to furnish zinc chloride impregnated alumina as a white powder. This can also be used on the next day, being properly stored in a desiccator. Ketene silyl acetals are primarily E-isomers, obtained by the reaction of the ester in THF with LDA at -78^oC followed by treatment with trimethyl silyl chloride according to a reported procedure.⁶

General Procedure for aldol reaction. - Zinc chloride impregnated alumina (1.5 g) was added to a mixture of ketene silyl acetal (1 mmol) and aldehyde (1 mmol) and mixed uniformly at room temperature. The reaction mixture was then sonicated under nitrogen for a certain period of time as required to complete the reaction. The solid mass was then eluted with methylene chloride and evaporated to provide the crude material which was warmed with dilute (1N HCl) for 5 min. This aqueous mixture was then extracted with ether (3 \times 15 ml) and the extract was washed with sodium bicarbonate solution, brine and dried over anhydrous sodium sulfate. Evaporation of the solvent furnished the product which was purified or separated by column chromatography over silica gel to provide pure material.

The aldol products are easily characterised by their spectral and analytical data . The anti /syn ratio was estimated by ¹ H NMR analysis of the corresponding oxymethine peaks.⁴ The spectral and analytical data of the products are presented below corresponding to the entries inTable 1. 1.^{4d} mp. 69^{0} C (lit^{4d} 69.5-70.5⁰ C); IR (KBr) 3500, 1730 cm⁻¹; ¹H NMR (CDCl₃ , 200 MHz) δ 1.10 (3H, s), 1.14 (3H, s), 3.71 (3H, s), 4.89 (1H, s), 7.30 (5H, s).

2. IR (neat) 3400-3520, 1730 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 0.83 (3H, d, J= 7 Hz), 0.93 3H, d, J= 7 Hz), 1.15 (3 H, s), 1.21 (3H, s), 1.47-2.21 (1H, m), 2.69 (1H, broad), 3.33 (1H, d, J = 4 Hz), 3.66 (3H, s). (Found : C, 61.79; H, 10.53. C₁₉H₁₈O₃ requires C, 62.04; H,10.41 %).

3. IR (neat) 3400-3520, 1725 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 0.93 (3H, t, J = 6.5 Hz), 1.13 (6H,s), 1.23 -1.70 (4H, m), 2.33 (1H, broad), 3.51 (1H, t, J=5 Hz), 3.68 (3H, s) (Found : C,62.12; H, 10.32. C₁₉ H₁₈O₃ requires C, 62.04; H,10.41%).

4. IR (neat) 3400-3500, 1730 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 1.11 (6H, s),
1.68 (3H, d, J=5 Hz), 2.33 (1H, broad), 3.66 (3H, s), 3.98 (1H, d, J=6 Hz), 5.26.0 (2H, m). (Found : C,63.00; H, 9.40. C₉H₁₆O₃ requires C, 62.76; H, 9.36%).

IR (neat) 3420-3520, 1725 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 1.23 (3H,s),
 1.24 (3H,s), 2.64 (1H,broad), 3.72 (3H,s), 4.35 (1H,d,J=7 Hz), 6.18 (1H. dd,
 J=15,7 Hz), 6.63 (1H, d,J=15 Hz), 7.23-7.39 (5H, m). (Found: C, 72.02; H,7.53.
 C₁₄H₁₈O₃ requires C, 71.77, H, 7.74%).

6. IR (neat) 3470-3520, 1725 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 1.0-2.2 (10H, m), 3.03 (1H, broad), 3.56 (3H, s), 4.54 (1H, s), 7.24 (5H, s). (Found: C, 72.83; H, 8.25. C₁₅H₂₀O₃ requires C, 72.55, H, 8.12%).

7. IR (neat) 3460-3540, 1725 cm⁻¹, ¹H NMR (CCl₄, 60 MHz) δ 0.71 (3H, d, J=7 Hz), 0.94 (3H, d, J=7 Hz), 1.08-2.2 (11H, m), 2.53 (1H, broad), 3.23 (1H,

broad), 3.66 (3H, s).(Found: C, 67.51; H, 10.13. C₁₂H₂₂O₃ requires C, 67.25; H, 10.35%).

8. IR (neat) 3420-3540, 1730 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 1.14-1.43 (6H, m), 1.69 (3H, d, J=6.4 Hz), 1.57-1.66 (4H, m), 2.07 (1H, broad), 3.70 (3H, s), 3.92 (1H, d, J=7.6 Hz), 5.36-5.48 (1H, m), 5.58-5.72 (1H, m). (Found: C, 67.60; H, 9.65. C₁₂H₂₀O₃ requires C, 67.89; H, 9.50%).

9. <u>syn</u>: IR (neat) 3440-3480, 1700 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 3.02-3.12 (4H, m), 3.41 (3H, s), 5.03 (1H, d, J=4.3 Hz), 7.12-7.40 (10H, m); ¹³C NMR (CDCl₃, 50 MHz) δ 29.37 (CH₂), 51.44 (CH), 55.38 (CH₃), 74.14 (CH), 126.22 (CH), 126.24 (CH), 126.54 (CH), 127.14 (CH), 127.82 (CH), 128.30 (CH), 128.35 (CH), 128.74 (CH), 130.09 (CH), 133.42 (CH), 139.13 (C), 141.38 (C), 174.43 (C). (Found: C, 75.24; H, 6.82. C₁₇H₁₈O₃ requires C, 75.53; H, 6.71%).

<u>anti:</u> mp 77.5-78^oC; IR (KBr) 3460-3540, 1735 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 2.72 (1H, dd, J=7.7,5.8 Hz), 2.87-2.92 (1H, m), 3.02-3.08 (2H,m), 3.09 (3H, s), 4.81 (1H, t, J=8 Hz), 7.09-7.38 (10H, m); ¹³C NMR (CDCl₃, 100 MHz) δ 35.73 (CH₂), 51.64 (CH), 54.95 (CH₃), 74.69 (CH), 126.26 (CH), 126.53 (CH), 128.43 (CH), 128.60 (CH), 128.77 (CH), 138.36 (C), 141.89 (C), 174.98 (C). (Found: C, 75.82; H, 6.93. C₁₇H₁₈O₃ requires C, 75.53; H, 6.71%).

10.^{4d} <u>syn</u>: IR (neat) 3450-3490, 1730 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 1.12 (3H, d, J=7 Hz), 2.73-2.87 (2H, m), 3.66 (3H, s), 5.09 (1H, d, J=4.1 Hz), 7.32 (5H, s).

anti: IR (neat) 3440-3480, 1730 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 0.97

(3H, d, J=7.1 Hz), 2.72-2.87 (2H, m), 3.70 (3H, s), 4.71 (1H, d, J=8.6 Hz), 7.31 (5H, s).

11.⁷ <u>syn</u>: IR (neat) 3410-3510, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.86 (3H, t, J=7.2 Hz), 1.58-1.82 (2H, m), 2.54-2.88 (2H, m), 3.61 (3H, s), 4.96 (1H, d, J=4.4 Hz), 7.33 (5H, s).

anti: IR (neat) 3410-3510, 1730 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 0.84 (3H, t, J=7.6 Hz), 1.3 -1.68 (3H, m), 2.64-2.75 (1H, m), 3.70 (3H, s), 4.79 (1H, d, J=8 Hz), 7.31 (5H, s).

12. <u>syn</u>: IR (neat) 3440-3540, 1735 cm⁻¹; ¹H NMR (CCL, 60 MHz), δ 0.83 (3H, t, J=7.2 Hz), 1.18-1.81 (2H, m), 2.3-2.7 (2H, m), 3.50 (3H, s), 3.71 (3H, s), 4.8 (1H, d, J=5 Hz), 6.71 (2H, d, J=8.5 Hz), 7.12 (2H, d, J=8.5 Hz). (Found: C, 65.72; H, 7.56. C₁₃H₁₈O₄ requires C, 65.53; H, 7.61%).

anti: IR (neat) 3420-3520, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.82 (3H, t, J=7.2 Hz), 1.14-1.74 (2H, m), 2.38 (1H, broad), 2.54-2.79 (1H, m), 3.74 (3H, s), 3.81 (3H, s), 4.76 (1H, d, J=8.4 Hz), 6.89 (2H, d, J=8.8 Hz), 7.28 (2H, d, J=8.8 Hz). (Found: C, 65.84; H, 7.77. C₁₃H₁₈O₄ requires C, 65.53; H, 7.61%). 13. <u>syn</u>: mp 44-45^oC; IR (KBr) 3480, 1710 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.86 (3H, t, J=7 Hz), 1.45-1.9 (2H, m), 2.58-2.80 (2H, m), 3.68 (3H, s), 5.12 (1H,

d, J=4.4 Hz), 7.56 (2H, d, J = 8.4 Hz), 8.22 (2H, d, J = 8.4 Hz). (Found: C, 56.70; H, 6.05; N, 5.58. $C_{12}H_{15}NO_5$ requires C, 56.91; H, 5.97; N, 5.53%).

anti: mp 64⁰C; IR (KBr) 3470, 1710 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.92 (3H, t, J=7 Hz)1.36-1.86 (2H, m), 2.56-2.78 (1H, m), 3.16-3.48 (1H, broad), 3.70 (3H, s), 4.9 (1H, d, J=7 Hz), 7.55 (2H, d, J=8.8), 8.23 (2H, d, J=8.8 Hz). (Found: C, 57.62; H, 6.01; N, 5.46. C₁₂H₁₅NO₅ requires C, 56.91; H, 5.97; N, 5.53%). 14.⁸ syn: IR (neat) 3410-3510, 1730 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 1.08 (3H, d, J=7 Hz), 1.20 (3H, t, J=7 Hz), 2.40-2.95 (2H, m), 4.07 (2H, q, J=7 Hz), 4.98 (1H, d, J=4 Hz), 7.24 (5H, s).

anti: IR (neat) 3410-3510, 1730 cm⁻¹; ¹H NMR (CCl₄, 60 MHz) δ 0.95 (3H, d, J=7 Hz), 1.2 (3H, t, J=7 Hz), 2.4-3.06 (2H, m), 4.07 (2H, q, J=7 Hz), 4.6 (1H, d, J=8 Hz), 7.16 (5H, s).

15. <u>syn</u>: IR (neat) 3440-3520, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 1.14 (3H, d, J=7.6 Hz), 1.19 (3H, t, J=7.6 Hz), 2.59-2.91 (2H, m), 3.80 (3H, s), 4.12 (2H, q, J=7.6 Hz), 5.02 (1H, d, J=4.4 Hz), 6.88 (2H, d, J=8.4 Hz), 7.28 (2H, d, J=8.4 Hz). (Found: C, 65.70; H, 7.68. C₁₃H₁₈O₄ requires C, 65.53; H, 7.61%)

<u>anti:</u> IR (neat) 3470, 1710 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.99 (3H, d, J=7.2 Hz), 1.27 (3H, t, J=7.2 Hz), 2.63-2.95 (2H, m), 3.81 (3H, s),4.20(2H,q,J=7.2Hz), 4.72 (1H, d, J=8.4 Hz), 6.89 (2H, d, J=8.8 Hz), 7.28 (2H,d, J=8.8 Hz). (Found: C, 65,80; H, 7.58. C₁₃H₁₈O₄ requires C, 65.53; H,7.61%).

16. <u>syn</u>: IR (neat) 3460-3510, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 1.08 (3H, d, J=7.2 Hz), 1.26 (3H,t, J=7.6 Hz), 2.66-2.92 (1H, m), 3.34 (1H, broad), 4.18 (2H, q, J=7.6 Hz), 5.25 (1H, d, J=4 Hz), 7.55 (2H, d, J=8.8 Hz), 8.22 (2H, d, J=8.8 Hz). (Found: C, 56.59; H, 5.87; N, 5.65. C₁₂H₁₅NO₅ requires C, 56.91; H, 5.97; N, 5.53%).

anti: IR (neat) 3450-3510, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz), δ 1.13 (3H, d, J=7.4 Hz),1.25 (3H, t, J=7 Hz), 2.67-2.97 (1H, m), 3.51 (1H, broad d), 4.17 (2H, q, J=7 Hz), 4.87 (1H,dd, J=8, 4 Hz), 7.55 (2H, d, J=8.8 Hz), 8.24 (2H, d, J=8.8 Hz). (Found: C, 57.65; H, 5.90; N, 5.70. C₁₂H₁₅NO₅ requires C, 56.91; H, 5.97; N, 5.53%).

17.⁹ syn: IR (neat) 3420-3520, 1720 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.88

(3H, t, J=7.2 Hz), 1.12 (3H, t, J=7 Hz), 1.53-1.85 (2H, m), 2.55-2.76 (1H, m),

2.83 (1H, broad), 4.07 (2H, q, J=7.2 Hz), 4.95 (1H, d, J=4.4 Hz), 7.36 (5H, m).

anti: IR (neat) 3420-3520, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.88 (3H, t, J=7 Hz), 1.24 (3H, t, J=7 Hz), 1.32-1.72 (2H, m), 2.57-2.80 (1H, m), 2.88 (1H, d, J=4 Hz), 4.17 (2H, q, J=7 Hz), 4.83 (1H, dd, J= 8, 4 Hz), 7.34 (5H, s).

18. <u>syn</u>: IR (neat) 3440-3520, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.88 (3H, t, J=7.2 Hz), 1.13 (3H, t, J=7.2 Hz), 1.53-1.90 (2H, m), 2.81 (2H, m), 3.80 (3H, s), 4.05 (2H, q, J=7.2 Hz), 4.87 (1H, d, J= 4.5 Hz), 6.87 (2H, d, J=8.8 Hz), 7.29 (2H, d, J=8.8 Hz). (Found: C,66.34; H, 8.06. C₁₄H₂₀O₄ requires C, 66.64; H, 7.99%).

anti: IR (neat) 3440-3520, 1730 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.85 (3H, t, J=7.2 Hz), 1.24 (3H, t, J=7.6 Hz), 1.23-1.82 (2H, m), 2.54-2.79 (2H, m), 3.80 (3H, s), 4.19 (2H, q, J=7.6 Hz), 4.76 (1H, d, J=8 Hz), 6.88 (2H, d, J=9 Hz), 7.27 (2H, d, J=9 Hz). (Found: C, 67.02; H, 7.93. C₁₄H₂₀O₄ requires C, 66.64; H, 7.99%).

19. <u>syn</u>: IR (neat) 3470, 1725 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.87 (3H, t, J=7.2 Hz), 1.23 (3H, t, J=7 Hz), 1.4-1.93 (2H, m), 2.55-2.74 (1H, m), 3.29 (1H, broad), 4.15 (2H, q, J=7 Hz), 5.12 (1H, d, J=4 Hz), 7.56 (2H, d, J=8.8 Hz), 8.2

(2H, d, J=8.8 Hz). (Found: C, 58.14; H, 6.35; N, 5.14. C₁₃H₁₇NO₅ requires 58.42; H, 6.41; N, 5.24%).

anti: mp 77.5°C; IR (KBr) 3470, 1710 cm⁻¹; ¹H NMR (CDCl₃, 100 MHz) δ 0.94 (3H, t, J=7.2 Hz), 1.19 (3H, t, J=7.2 Hz), 1.36-1.88 (2H, m), 2.56-2.80 (1H, m), 3.46 (1H, d, J=6.4 Hz), 4.13 (2H, q, J=7.2 Hz), 4.9 (1H, t, J=6.5 Hz), 7.53 (2H, d, J=9 Hz), 8.23 (2H, d, J=9 Hz). (Found: C,58.75; H, 6.50; N, 5.30. C₁₃ H₁₇NO₅ requires C, 58.42; H, 6.41; N, 5.24%).

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