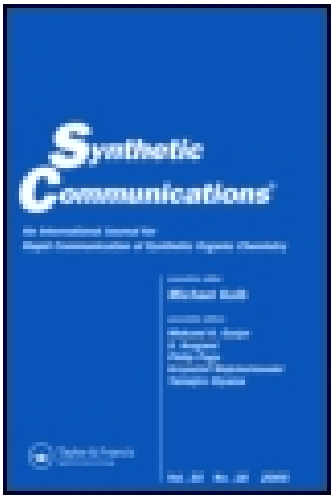

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Knoevenagel Condensation Catalysed by New Montmorillonitesilylpropylethylene

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KNOEVENAGEL CONDENSATION CATALYSED BY NEW MONTMORILLONITESILYLPROPYLETHYLENEDIAMINE

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Abstract : Facile knoevenagel condensations catalysed by new montmorillonitesilylpropylethylenediamine is reported.

Trisubstituted alkenes are easily obtained by employing the widely known base catalysed condensation of carbonyl compounds with compounds containing active methylenic group devised by knoevenagel condensation[1]. The reaction is generally catalysed by weak bases under homogeneous conditions[1] and solids[2] and solid supported basic catalysts[3,4]. We recently reported the phase transfer nucleophilic substitution reactions catalysis by quaternary ammonium salts covalently anchored on montmorillonite[5]. Herein this communication we describe a facile knoevenagel condensation catalysed by a new montmorillonitesilylpropylethylenediamine (cat-I).

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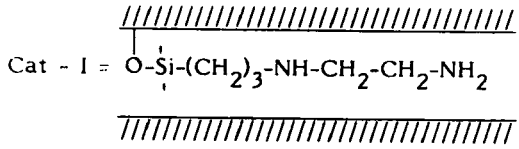


Cat - I = O-Si-$(CH_2)_3$-NH-CH_2-CH_2-NH_2

The reaction (Scheme 1) proceeds very rapidly to completion within 5 minutes afforded the trisubstituted olefins in near quantitative yields. The reaction described here is very convenient since the catalyst can be easily filtered off and reused for several recycles without any regeneration. In fact the same catalyst reused in all the reactions described in the Table 1 by simple filtration and washing gave identical yields with consistant rate compared with the results obtained by the use of fresh catalyst. The catalyst was prepared by condensation of 3-triethoxysilylpropylethylenediamine with montmorillonite. Thus the abundantly available acidic montmorillonite was converted into basic one. The use of present catalyst described here becomes practical alternative to the best catalysts reported in literature.

Experimental

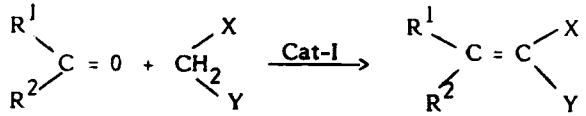
The catalyst was prepared by the condensation of 3-triethyoxysilylpropylethylenediamine (1.2 g, 5.37 mmol) with montmorillonite K10 (4.0 g) in dry toluene under reflux for 24 hours. Then it was filtered and washed gently with toluene and ether and dried[6].

in a typical experiment the reaction flask was charged with catalyst (0.5 g), toluene 5-6 ml and substrates in equal molar ratio (50 mmol) and stirred at ambient temperature. The reaction was followed by TLC. After completion of the reaction, it was filtered and washed with toluene, and the catalyst was kept aside for another recycle. The filtrate was concentrated and analysed by IR, NMR and Mass.

Table 1 : Knoevenagel Condensations with Catalyst-I

S.No.	Carbonyl Comp. (A)	Active Methylene Comp. (B)	Yield (%)[a] (C)
1	Benzaldehyde	Malononitrile	99
2	Benzaldehyde	Ethylcyanoacetate	98
3	Anisaldehyde	Malononitrile	94
4	Anisaldehyde	Ethylcyanoacetate	94
5	Cinnamaldehyde	Malononitrile	90
6	Cinnamaldehyde	Ethylcyanoacetate	91
7	Furfuraldehyde	Malononitrile	84
8	Furfuraldehyde	Ethylcyanoacetate	87
9	3,4,5 trimethoxy benzaldehyde	Malononitrile	97
10	3,4,5 trimethoxy benzaldehyde	Ethylcyanoacetate	96
11	Benzaldehyde[b]	Malononitrile	0

a : Isolated yields, b : With montmorillonite K10



A B C

Scheme - 1

Acknowledgements

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