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N,N-Dimethylpyridin-4-Amine Mediated Protocol for Cyanoethoxycarbonylation of Aldehydes Under Solvent-Free Conditions

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Abstract *N,N*-Dimethylpyridin-4-amine (DAMP) (10 mol%) was successfully employed as catalyst for cyanoethoxycarbonylation of aromatic and aliphatic aldehydes at room temperature under solvent free condition to give corresponding ethylcyanocarbonates in excellent isolated yield (up to 95%) in 15–90 min. Simple experimental conditions and product isolation procedure has made this protocol quite attractive for the synthesis of ethylcyanocarbonates in an environment-friendly manner.

Keywords *N,N*-dimethylpyridin-4-amine · Cyanoethoxycarbonylation · Aldehydes · Solvent-free · Ethylcyanoformate

1 Introduction

Cyanohydrins are valuable intermediates for the synthesis of range of compounds viz., α-amino acids, α-hydroxy acids, β-amino alcohols, vicinal diols and α-hydroxy ketones [1–5] which are important building blocks for numerous pharmaceuticals, agrochemicals and insecticides [6–13]. Cyanohydrins are conventionally synthesized by the cyanation of carbonyl compounds using KCN, NaCN, trimethylsilyl cyanide (TMSCN) and HCN as sources of cyanide in the presence of various Lewis acid/base catalysts [14–30]. Kobayashi et al. for the first time have reported the use of triethylamine as an efficient catalyst for the cyanosilylation of aldehydes to give cyanosilylether in excellent yields [31]. However, this protocol has used toxic TMSCN as a cyanide source and environmentally hazardous dichloromethane as a solvent at 0 °C. Therefore, in recent days, acylcyanides and alkylcyanoformates are more preferred sources of cyanide as they are safer, effective and easily available [32–37]. These cyanide sources however, required the use of a base as catalyst to promote cyanation reaction [38–50]. In this direction, Poirier and co-workers [51, 52] effectively utilized various tertiary amines as catalysts for the methoxycarbonylation of aldehydes whereas Deng and co-workers [53, 54] have used a chiral base for the cyanocarbonylation of ketones. On the other hand Oriyama et al. [55] have reported the addition of acylcyanide to aldehydes in the absence of a catalyst but used DMSO as a solvent. All the above mentioned reactions were invariably carried out in the presence of a solvent (often toxic or hazardous) and required longer reaction time. Therefore, a solvent-free protocol is preferred for the practical applications. Recently Tian and co-workers have reported the solvent-free cyanocarbonylation of aldehydes using ethylcyanoformate as a source of cyanide in the presence of 1-methoxy-2-methyl-1-(trimethylsiloxy)propene as a catalyst (5–25 mol%) to afford the desired ethylcyanocarbonates in excellent yields in time ranging from 9 h to 72 h [42]. Nevertheless, 1-methoxy-2-methyl-1-(trimethylsiloxy)propene is highly volatile, moisture sensitive and hazardous. Earlier, we have reported solvent-free and environmentally benign synthetic protocols for the synthesis of cyanosilylethers [56], aminonitriles [57] and β-amino ketone (Mannich reaction) [58]. In this context and our ongoing interest in cyanation of aldehydes [59–64], the present study describes the addition of

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ethylcyanoformate to aldehydes using commercially available *N,N*-dimethylpyridin-4-amine (DMAP) as a catalyst to give ethylcyanocarbonates in excellent yields under solvent-free conditions at room temperature in short period of time.

2 Experimental

2.1 Materials and Methods

N,N-dimethylpyridin-4-amine, 2,6-dimethylpyridine, benzaldehyde, 3-methoxybenzaldehyde, 2-methoxybenzaldehyde, 4-methoxybenzaldehyde, 2-ethoxybenzaldehyde, 2-benzyloxybenzaldehyde, 4-chlorobenzaldehyde, 1-naphthaldehyde, 2-naphthaldehyde, thiophen-2-carboxaldehyde, hydrocinnamaldehyde, α-methyl cinnamaldehyde, hexanal, isovaraldehyde, were purchased from Sigma-Aldrich Chemicals. 2-Methylbenzaldehyde, 3-methylbenzaldehyde, 4-methylbenzaldehyde were from Merck Chemicals whereas triethylamine, pyridine, silica gel (60-200) mesh were purchased from s d Fine-Chem Limited, Mumbai (India). NMR spectra were recorded on a Bruker F113 V spectrometer (500 MHz and 125 MHz for ^{1}H and ^{13}C respectively) and were referenced internally with TMS. High-resolution mass spectra were recorded on a LC-MS (Q-TOF) LC (Waters), MS (Micromass) instruments. For the product purification, flash chromatography was performed using silica gel (60–200) mesh.

2.2 General Procedure for Cyanoethoxycarbonylation of Aldehydes

The catalyst DMAP (10 mol%, 12 mg) was taken in ethylcyanoformate (1.5 mmol, 0.148 ml) to which an appropriate aldehyde (1 mmol) was added in small fractions over a period of 5 min while stirring the reaction mixture magnetically at room temperature (27 °C). The progress of the reaction was checked on TLC. After complete consumption of aldehyde the product was purified by silica gel flash column chromatography (eluted with hexane/ethyl acetate = 90:10). The purified products were characterized by ^{1}H and ^{13}C NMR. Characterization data of some selected products are as follows.

*2.2.1 2-Ethoxycarbonyl-2-Hydroxy-2-Phenyl-Acetonitrile (**2a**) [14, 64]*

Colorless liquid. ^{1}H NMR δ = 1.32 (t, J = 7.5, 3H), 4.25–4.30 (m, 2H), 6.26 (s, 1H), 7.43–7.54 (m, 5H) ppm. ^{13}C NMR 14.21, 65.73, 66.48, 115.93, 127.99, 129.38, 130.74, 131.37, 153.53 ppm.

*2.2.2 2-Ethoxycarbonyl-2-Hydroxy-2-(2-Methylphenyl)-Acetonitrile (**2b**) [14, 64]*

^{1}H NMR δ = 1.33 (t, J = 7.5, 3H), 2.44 (s, 3H), 4.25–4.31 (m, 2H), 6.38 (s, 1H), 7.23–7.37 (m, 3H), 7.55 (d, J = 8.0, 1H) ppm. ^{13}C NMR δ = 14.27, 19.06, 64.72, 65.75, 115.83, 126.93, 128.75, 129.55, 130.83, 131.48, 136.90, 153.61 ppm.

*2.2.3 2-Ethoxycarbonyl-2-Hydroxy-2-(4-Chlorophenyl)-Acetonitrile (**2e**) [14, 64]*

^{1}H NMR δ = 1.31 (t, J = 7.0, 3H), 4.25-4.29 (m, 2H), 6.22 (s, 1H), 7.41–7.47 (m, 4H) ppm. ^{13}C NMR δ = 14.23, 65.91, 65.72, 115.57, 129.66, 129.89, 136.97, 153.39 ppm.

*2.2.4 2-Ethoxycarbonyl-2-Hydroxy-2-(2-Methoxylphenyl)-Acetonitrile (**2f**) [64]*

^{1}H NMR δ = 1.32 (t, J = 7.0, 3H), 3.86 (s, 3H), 4.26–4.29 (m, 2H), 6.58 (s, 1H), 6.94 (d, J = 8.0, 1H), 7.00–7.03 (m, 1H), 7.40–7.4 (m, 1H), 7.55 (dd, J = 2, 6, 1H) ppm. ^{13}C NMR δ = 14.28, 55.90, 61.85, 65.57, 111.25, 116.10, 119.60, 121.09, 128.09, 132.21, 153.66, 156.89 ppm.

*2.2.5 2-Ethoxycarbonyl-2-Hydroxy-2-(3-Methoxylphenyl)-Acetonitrile (**2g**) [64]*

^{1}H NMR δ = 1.33 (t, J = 7.0, 3H), 3.82 (s, 3H), 4.26–4.29 (m, 2H), 6.23 (s, 1H), 6.97-7.11 (m, 3H), 7.33–7.36 (m, 1H) ppm. ^{13}C NMR δ = 14.12, 55.44, 65.65, 66.24, 113.12, 115.80, 116.38, 119.97, 130.39, 132.55, 153.41, 160.13 ppm.

*2.2.6 2-Ethoxycarbonyl-2-Hydroxy-2-(4-Methoxylphenyl)-Acetonitrile (**2h**) [64]*

^{1}H NMR δ = 1.25 (t, J = 7.0, 3H), 3.75 (s, 3H), 4.18–4.21 (m, 2H), 6.13 (s, 1H), 6.88 (d, J = 8.5, 2H), 7.40 (d, J = 8.5, 2H) ppm. ^{13}C NMR δ = 14.23, 55.54, 65.60, 66.26, 114.69, 116.13, 123.44, 129.85, 153.58, 161.44 ppm.

*2.2.7 2-Ethoxycarbonyl-2-Hydroxy-2-(2-Ethoxylphenyl)-Acetonitrile (**2i**) [64]*

^{1}H NMR δ = 1.33 (t, J = 6.5, 3H), 1.42 (t, J = 6.5, 3H), 4.10 (brd, 2H), 4.28 (brs, 2H), 6.62 (s, 1H), 6.92 (d, J = 8.0, 1H), 7.00 (t, J = 7, 1H), 7.39 (t, J = 7.5, 1H), 7.57 (d, J = 7.0, 1H) ppm. ^{13}C NMR 14.70, 14.26, 61.98, 64.29, 65.48, 111.94, 116.08, 119.60, 120.83, 128.77, 132.07, 153.65, 156.27 ppm.

2.2.8 2-Ethoxycarbonyl-2-Hydroxy-2-(2-Benzyloxyphenyl)-Acetonitrile (**2j**) [64]

^{1}H NMR $\delta = 1.27$ (t, $J = 7.0$, 3H), 4.18–4.22 (m, 2H), 5.11 (s, 2H), 6.64 (s, 1H), 6.95 (d, $J = 8.5$, 1H), 7.01 (t, $J = 7.5$, 1H), 7.29–7.41 (m, 6H), 7.60 (d, $J = 7.5$, 1H) ppm. ^{13}C NMR $\delta = 14.18$, 62.00, 65.45, 70.46, 112.43, 115.98, 119.86, 121.26, 127.31, 128.23, 128.71, 128.85, 132.06, 136.09, 153.53, 155.89 ppm.

2.2.9 2-Ethoxycarbonyl-2-Hydroxy-2-(2-Naphthyl)-Acetonitrile (**2l**) [64]

^{1}H NMR $\delta = 1.34$ (t, $J = 7.5$, 3H), 4.28–4.33(m, 2H), 6.43 (s, 1H), 7.25 (s, 1H), 7.54–7.59 (m, 3H), 7.86–8.04 (m, 3H) ppm. ^{13}C NMR $\delta = 14.32$, 65.87, 66.77, 116.00, 124.37, 127.29, 127.86, 128.03, 128.29, 128.63, 129.69, 133.01, 134.18, 153.68 ppm.

2.2.10 2-Ethoxycarbonyl-2-Hydroxy Heptanenitrile (**2o**)

^{1}H NMR $\delta = 0.87$–0.90 (m, 3H), 1.31–1.32 (m, 7H), 1.46–1.54 (m, 2H), 1.89–1.94 (m, 2H), 4.23–4.37 (m, 2H), 5.18 (t, $J = 6.7$, 1H) ppm.

2.2.11 2-Ethoxycarbonyl-2-Hydroxy-4-Phenyl-Butanonitrile (**2q**)

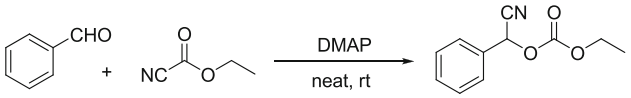
^{1}H NMR $\delta = 1.34$ (t, $J = 7.0$, 3H), 2.23–2.29 (m, 2H), 2.85 (t, $J = 7$, 2H), 4.26–4.28 (m, 2H), 5.12 (t, $J = 6.0$, 1H), 7.18–7.32 (m, 5H) ppm.

3 Results and Discussion

To achieve high yield of ethylcyanocarbonates under solvent-free condition using DMAP as a catalyst, we have started our systemic study by varying the catalyst loading (2.5–20 mol%) with benzaldehyde as a representative substrate and ethylcyanoformate as a source of cyanide at RT (Table 1). On increasing the catalyst loading from 2.5 to 10 mol% there was a steady increase in the yield of the product with a decrease in the reaction time (entries, 1–3). A further increase in the catalyst loading (20 mol%) did not show any significant improvement in the performance of the reaction (entry 4). Hence, 10 mol% catalyst loading was considered to be optimum to achieved high yield of ethylcyanocarbonates under solvent-free condition at RT.

Based on the above optimum reaction conditions, we have extended this catalytic protocol (DMAP; 10 mol%) for the cyanation of a variety of aromatic and aliphatic aldehydes (Table 2). It is evident from the results that this protocol is highly sensitive towards the type of aldehydes used as substrate. Using this protocol ethylcyanocarbonates from benzaldehyde and methyl substituted benzaldehyde were obtained in excellent isolated yield (~90%) in 90 min (entries 1–4). Whereas chlorobenzaldehyde reacted very slowly (480 min) under this condition to give the desired product in 62% isolated yield (entry 5). Evidently, electron rich benzaldehydes viz. methoxy, ethoxy and benzyloxy substituents readily formed corresponding products in excellent yield (~93%) in 40 min (entries 6–10). Remarkably, 1 and 2-naphthaldehydes were turned out to be most reactive substrates under the present reaction conditions (entries 11, 12). The heterocyclic aromatic aldehyde viz., thiophene 2-caboxylaldehyde also reacted readily (35 min) to give corresponding ethylcyanocarbonate in 91% yield (entry 13). Furthermore, this catalytic system also worked well with conjugated aldehydes viz., α-methyl cinnamaldehyde (entry 14). We also checked the utility of the present protocol for the cyanoethoxycarbonylation of aliphatic aldehydes. The straight chain and branched aldehydes viz., hexanal and isovaraldehyde gave the corresponding ethylcyanocarbonates in 72 and 86% yield respectively in 60 min. However, aliphatic aldehyde with phenyl substituent (hydrocinnamaldehyde) reacted sluggishly (180 min) to give the desired product in 80% yield (entry 17). It can therefore be concluded that the present cyanation protocol is able to accommodate a variety of aldehydes. We also tried this protocol for cyanoethoxycarbonylation of ketones (acetophenone and cyclohexanone), however, the system failed to give the desired products.

Table 1 Optimization of cyanoethoxycarbonylation of benzaldehyde under solvent free condition[a]

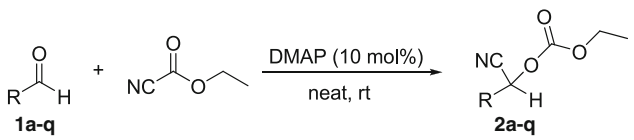


Entry	Catalyst	Catalyst mol (%)	Time (min)	Yield (%)[b]
1	DMAP	2.5	180	68
2	DMAP	5	150	73
3	DMAP	10	90	89
4	DMAP	20	90	91
5	Pyridine	10	12(h)	ND
6	2,6-DMP	10	12(h)	ND
7	TEA	10	120	92

[a] All reactions were carried out using aldehyde 1 mmol, ethylcyanoformate 1.5 mmol, in presence of base as catalyst at room temperature

[b] Isolated yield

Table 2 Cyanoethoxycarbonylation of various aldehydes under solvent-free conditions in the presence of DMAP as catalyst[a]



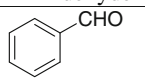
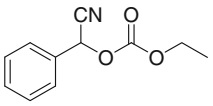
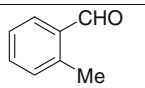
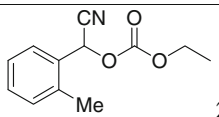
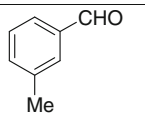
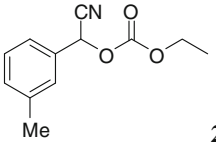
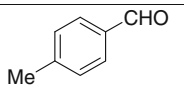
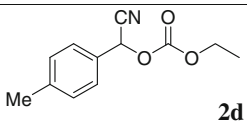
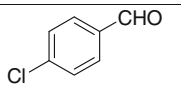
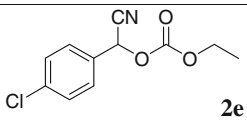
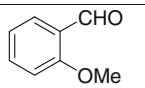
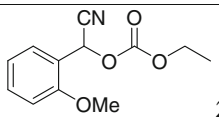
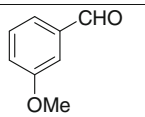
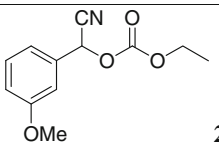
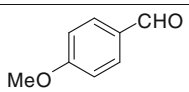
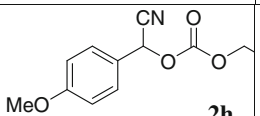
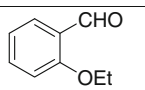
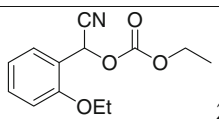
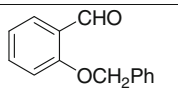
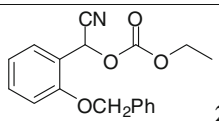
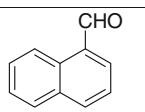
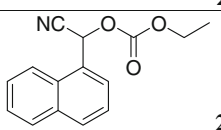
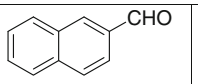
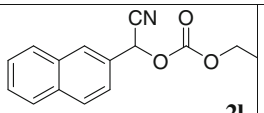
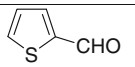
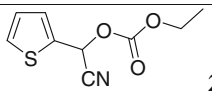
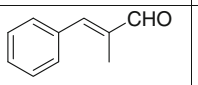
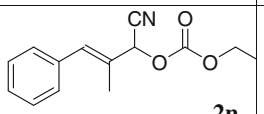
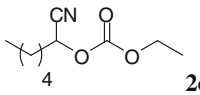
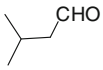
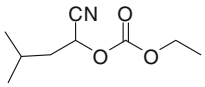
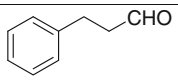
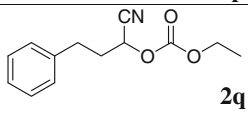
Entry	Aldehyde	Product	Time (min)	Yield (%)[b]
1		 **2a**	90	89
2		 **2b**	90	90
3		 **2c**	90	91
4		 **2d**	90	87
5		 **2e**	480	62
6		 **2f**	40	94
7		 **2g**	40	93
8		 **2h**	40	92
9		 **2i**	40	94
10		 **2j**	40	95
11		 **2k**	15	93

Table 2 continued

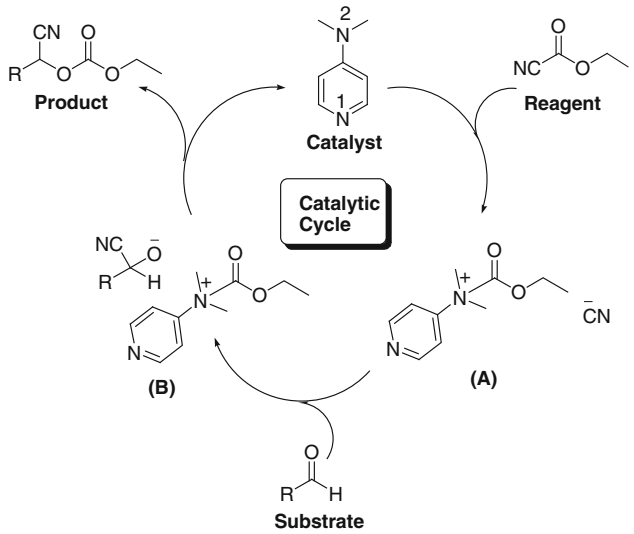
12		 **2l**	15	95
13		 **2m**	35	91
14		 **2n**	60	92
15		 **2o**	60	72
16		 **2p**	60	86
17		 **2q**	180	80

[a] All reactions were carried out using aldehyde 1 mmol, ethylcyanoformate 1.5 mmol, in presence of 10 mol% DMAP at room temperature

[b] Isolated yield

3.1 Mechanism of the Reaction

Deng et al. in a probable mechanism have proposed the distinct role of basic center in various tertiary amines for cyanoethoxycarbonylation of aldehydes [47, 48, 53, 54]. However, in our case the DMAP has got two basic centers. In order to understand which basic center is essentially responsible for the catalytic activity we have conducted a series of experiments for cyanoethoxycarbonylation of benzaldehyde with triethylamine (TEA), pyridine and 2,6-dimethylpyridine as catalysts. TEA catalyzed the reaction effectively to give the desired product in 92% isolated yield in 120 min (Table 1, entry 7). On the other hand both pyridine and 2,6-dimethyl pyridine failed to catalyze the cyanoethoxycarbonylation of benzaldehyde (Table 1, entries 5,6). From these results it is evident that tertiary nitrogen (N2) is largely responsible for the catalytic activity. Based on the above results and from the understandings of previous reports [35, 41, 47, 53], we have proposed a probable mechanism as depicted in Scheme 1. Accordingly, ethylcyanoformate reacts with DMAP to form an intermediate (A). Thus activated cyanide ion from intermediate-A reacts with the aldehyde to give the cyanoalkoxide intermediate (B). In a subsequent step intramolecular nucleophilic attack of the cyanoalkoxide on the carbonyl carbon in intermediate B takes place to give the desire product ethylcyanocarbonate.



Scheme 1 Proposed mechanism for cyanoethoxycarbonylation of aldehydes

4 Conclusion

In conclusion we have developed a simple and efficient protocol for the synthesis of ethylcyanocarbonate of various aldehydes using commercially available DMAP as a catalyst under solvent-free condition. Using the present catalytic protocol, 2-naphthaldehyde as a substrate reacted very rapidly (15 min) to form corresponding ethylcyanocarbonate in excellent yield (95%).

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