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Short communication

Aluminum phthalocyanine: an active and simple catalyst for cyanosilylation of aldehydes

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Aluminum phthalocyanine (AlPc) in the presence of Ph_3PO acts as a highly effective catalyst for cyanosilylation of various aldehydes to the corresponding cyanohydrin trimethylsilyl ethers. The reaction proceeds smoothly with 5 mol% catalyst loading at room temperature, giving up to 96% yield.

KEYWORDS: cyanosilylation; catalysis; aldehydes; aluminum phthalocyanine; cyanohydrins; triphenylphosphine oxide

INTRODUCTION

Cyanohydrins represent a valuable starting material that can be elaborated into a variety of useful synthetic building blocks, such as α-hydroxy acids, α-hydroxy aldehydes, 1,2-diols and α-amino alcohols.[1–5] Because of their importance in organic synthesis and life science research, a large body of work has been devoted to the development of cyanohydrin synthesis. Transfer of a cyano group from TMSCN to carbonyl compounds can be catalyzed by a plethora of reagents[6–25] including Lewis acids, Lewis bases, metal alkoxides, bifunctional catalysts and inorganic salts. Many metal complexes have been successfully employed as Lewis acids for the addition of HCN or TMSCN to aldehydes and ketones, such as, magnesium, zirconium, titanium, aluminum, yttrium, lanthanum, samarium, vanadium and gadolinium complexes containing mono- or polydentate ligands.[26]

Tetramethylguanidine-catalyzed cyanosilylation of benzaldehyde proceeds smoothly with 0.1 mol% catalyst in 4 h reaction with a yield of 99%.[27] Proazaphopsphatrane, a non-ionic strong base, has been reported as a catalyst for aldehydes and ketones.[28] The reaction was carried out with about 3 mol% of catalyst at a reaction time of 1–2 h with yield 89–99%. Lithium chloride acts as an active and simple catalyst for cyanosilylation of aldehydes and ketones.[29] The reaction proceeds with a substrate–catalyst molar ratio of 100–100 000 at room temperature with yield of 80–98%. The activation of TMSCN by *N*-heterocyclic carbenes for facile cyanosilylation of carbonyl compounds has been reported recently. Cyano transfer from TMSCN to aldehydes occurs at room temperature in the presence of 0.01–0.5 mol% catalyst in 10 min with 91% yield.[30] Sato *et al*. have succeeded in the cyanosilylation of aldehydes using *N*-heterocyclic carbenes.[31] BINOL-based aluminum complexes have been employed as catalysts for cyanosilylation of aldehydes and ketones.[32–35] Recently we have reported asymmetric cyanosilylation of aldehydes[36] and ketones[37] using Al(salen)–Ph_3PO catalytic system as double activation method.

Metal phthalocyanines (Mpc) are easily accessible, stable and a cost-effective catalysts for a variety of organic reactions.[38–40] In the continuation of our work on cyanosilylation,[41–45] we are interested in the application of aluminum phthalocyanine for the cyanosilylation reactions.

RESULTS AND DISCUSSION

The optimization of the variables of the reactions of benzaldehyde with TMSCN was done by examining different types of phthalocyanines and Ph_3PO as additive. The results of these reactions are summarized in Table 1. Mn, Fe and Al phthalocyanines were used for the reactions. When manganese phthalocyanine (MnPc) and iron phthalocyanine

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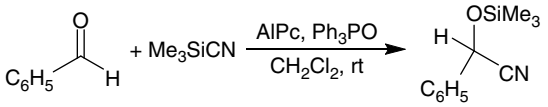
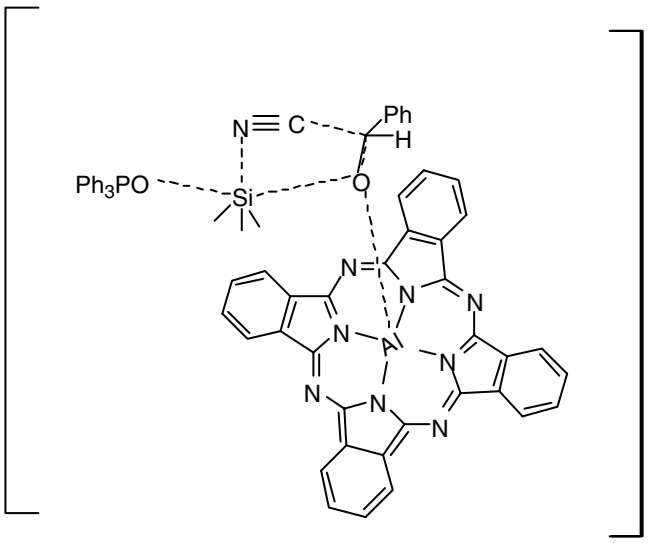
Table 1. Cyanosilylation of benzaldehyde under various conditions



Entry	Catalyst	AlPc (mol %)	Ph_3PO (mol)	Solvent	Time (h)	Yield[a] (%)
1	MnPc	5	10	CH_2Cl_2	6	N.R
2.	FePc	5	10	CH_2Cl_2	6	N.R
3.	AlPc	5	10	CH_2Cl_2	1.15	96
4.	AlPc	5	10	CH_2Cl_2	1.15	60
5.	AlPc	5	10	THF	1.15	55
6.	AlPc	5	5	CH_2Cl_2	2	90
7.	AlPc	10	10	CH_2Cl_2	1.15	95
8.	AlPc	5	—	CH_2Cl_2	6	—
9.	AlPc	—	10	CH_2Cl_2	6	—

[a] Isolated yield.



Scheme 1. Transition state involved in the cyanosilylation of aldehydes by double activation catalysis.

(FePc) were employed as catalysts, no reaction was observed (Table 1, entries 1 and 2). This might be due to the difference in the Lewis acidic properties of metals. Various solvents, including CH_3CN, THF and CH_2Cl_2, were examined with AlPc as catalyst and CH_2Cl_2 was found to be the best solvent for this reaction at room temperature (Table 1, entries 3–5). Ph_3PO at 10 mol% proved to be the optimal condition (Table 1, entries 3 and 6). Further increase in catalytic loading did not alter the reaction yield and reaction time (Table 1, entry 7).

No reaction took place either without Ph_3PO or without AlPc (Table 1, entries 8 and 9). This indicates a double activation process occurring through the catalysis of both the Lewis acid and Lewis base. The AlPc functions as a Lewis acid to activate the aldehydes while Ph_3PO acts as a Lewis base for the activation of TMSCN. Based on this study, a reaction mechanism is proposed as follows. The aluminum phthalocyanine will function as the Lewis acid to activate the carbonyl group and the triphenylphosphine oxide will function as the Lewis base to activate the TMSCN. Both the species mentioned above will attract and approach each other to form a transition state that undergoes intramolecular transfer of cyanide to aldehydes. Cyanohydrins are formed through the process along with AlPc and Ph_3PO (Scheme 1). The ^{13}C NMR spectra of both TMSCN and a mixture of TMSCN and Ph_3PO are also incorporated. Further studies are in progress to clarify the mechanism of the reaction.

Unsubstituted and substituted aromatic aldehydes undergo excellent cyanosilylation with over 93% yield (Table 2, entries 1–6). Aliphatic aldehydes were silylcyanated in good yields for a slightly longer reaction time (Table 2, entries 7–9). It should be noted that the sterically hindered pivalaldehyde was transformed into the corresponding silyl ether at 85% yield (entry 10). Furfural, a heterocyclic aldehyde (Table 2, entry 11), gives corresponding silyl ether at good yield (92%). This result indicates that this catalytic system can selectively activate the carbonyl function of furfural, keeping the furan ring intact. Acetophenone as a substrate has a slightly longer reaction time (3 h) with 90% yield. The rest of the ketones will be exposed to the similar reactions; this will be published elsewhere. Recently several improved achiral methods have been reported with low catalytic loading. *N*-heterocyclic carbene and K_2CO_3-catalyzed systems require 0.5 and 3 mol% of catalyst loading respectively.[31,48] $BiCl_3$ promoted cyanosilylation reaction occurs in 20 min time with 20 mol% catalyst loading.[49] Many of the existing methods for cyanosilylation of carbonyl compounds have disadvantages such as high catalytic loading, prolonged reaction time and poor yields of the corresponding cyanotrimethylsilyl ethers at room temperature. $LiClO_4$-catalyzed[50] cyanosilylation requires 100 mol% of catalytic loading. The $Zr(KPO_4)_2$-catalyzed[51] reaction proceeds with 35 mol% catalyst for complete conversion of benzaldehyde. Although $VO(OTf)_2$ catalysis requires less catalytic loading, it is restricted to substrate applicability.[52] As shown in Table 2, our catalytic system requires moderate catalytic loading and shorter reaction time compared with the previous studies.[9,12,46,47]

CONCLUSIONS

In summary, we have identified a new class of readily available organometallic catalyst that efficiently promotes the cyanosilylation of various aldehydes in relatively short reaction time with low catalyst loading under mild conditions. This could be the first example of phthalocyanine-based catalyst used for cyanosilylation reactions. Studies on mechanistic details and recovery of the catalyst are currently underway.

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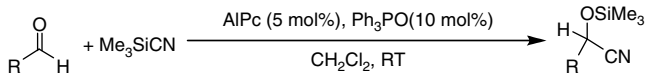
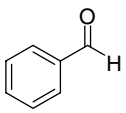
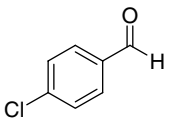
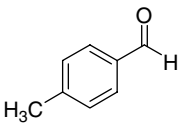
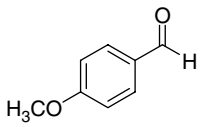
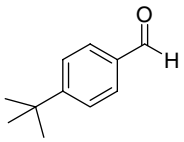
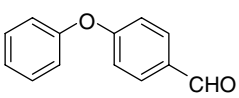
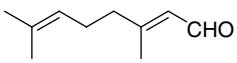
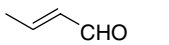
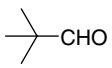
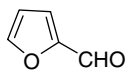
Table 2. Trimethylsilylcyanation of aldehydes with TMSCN catalyzed by AlPc/Ph_3PO[a]



Entry	Substrate	Time (h)	Yield(%)[b]	Literature values	
				Time (h)	Yield (%)
1		1.15	96	3	95[c,d,e]
				24	70[d,f]
				3	81[e]
2		1.15	95	3	75[c]
				2	80[f]
3		1.15	96	12	80[g]
4		1.30	94		
5		1.30	95		
6		1.15	93		
7		2	85	4.5	90[c]
8		1.45	90		
9	$(CH_3)_2-CH-CHO$	2	88		
10		2	85	6	88[g]
11.		1.30	92		

[a] Alpc/Ph_3PO = 5 mol%/10 mol%. [b] Isolated yield. [c] Karmi and MaMani[46] [4.5% of silica-based Sc(III)]. [d] Cordoba and Plumet[12] (10 mol% of Ph_3P^+Me,I^-). [e] Saravanan *et al.*[9] [5 mol% of $Cu(OTf)_2$]. [f] Song *et al.*[30] (1 mol% of NHC). [g] Lakshmi Kantham *et al.*[47] (10 mol% of diamino functionalized mesoporous material).

EXPERIMENTAL

In all cases the ^1H NMR (200 MHz) and ^{13}C NMR (50 MHz) spectra were recorded using a Varian Gemini 2000 spectrophotometer in $CDCl_3$ with tetramethylsilane as internal standard.

AIPc, MnPc and FePc, aldehydes and ketones were purchased from Aldrich.

Silylcyanation of benzaldehyde; 2-phenyl-2-(trimethylsilyloxy)acetonitrile (Table 2 entry 1)

In a representative protocol, benzaldehyde (106 mg, 1 mmol) was added to a stirred CH_2Cl_2 (2 ml) solution of the catalyst (5 mol% aluminum phthalocyanine and 10 mol% Ph_3PO) and the mixture stirred for 10 min at room temperature. TMSCN (1.5 equiv.) was then added with a syringe pump, the mixture

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was stirred continuously and the progress of the reaction was followed by TLC. After 1.15 h the reaction mixture was purified by silica gel flash chromatography by using an ethyl acetate–hexane (1 : 9) mixture as eluent. The desired 2-phenyl-2-(trimethylsilyloxy)acetonitrile was obtained as a colorless oil (yield 95%). The other substrates in Table 2 were also silylcyanated using the same procedure. The silyl ethers thus obtained were identified by ^{1}H and ^{13}C NMR data, which are consistent with the structure.

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.25$ (s, 9H), 5.52 (s, 1H), 7.42–7.47 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.33$, 63.59, 119.12, 126.29, 128.87, 129.27, 136.18. HRMS (E1): m/z calcd for $C_{11}H_{15}NOSi$ (M^+): 205.0923; found: 205.091.

2-(4-Cholorophenyl)-2-(trimethylsilyloxy)acetonitrile (entry 2)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.25$ (s, 9H), 5.46 (s, 1H), 7.39–7.40 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.33$, 62.93, 118.76, 127.64, 129.12, 134.80, 135.28. HRMS (EI): m/z calcd for $C_{11}H_{14}ClNOSi$ (M^+): 239.0533; found: 239.059.

2-(4-Methylphenyl)-2-(trimethylsilyloxy)acetonitrile (entry 3)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.22$ (s, 9H), 2.35 (s, 3H), 5.45 (s, 1H), 7.21 (d, 2H, J = 7.8 Hz), 7.37 (d, 2H, $J = 7.8$ Hz). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.25$, 55.78, 63.87, 114.66, 119.47, 127.88, 128.77, 160.23. HRMS (EI): m/z calcd for $C_{12}H_{17}NOSi$ (M^+): 219.1079; found: 219.107.

2-(4-Methoxyphenyl)-2-(trimethylsilyloxy) acetonitrile (entry 4)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.21$ (s, 9H), 3.82 (s, 3H), 5.43 (s, 1H), 6.94 (d, 2H, $J = 8.8$ Hz), 7.38 (d, 2H, $J = 8.8$ Hz). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.23$, 55.34, 63.34, 114.25, 119.32, 127.93, 128.46, 160.33. HRMS (EI): m/z calcd for $C_{12}H_{17}NO_2Si$ (M^+): 235.1029; found: 235.103

2-(4-tert-butylphenyl)-2-(trimethylsilyloxy) acetonitrile (entry 5)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.24$ (s, 9H), 1.32 (s, 9H), 5.46 (s, 1H), 7.29–7.42 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.39$, 31.10, 34.52, 63.32, 119.18, 125.73, 126.04, 133.19, 152.37. HRMS (EI): m/z calcd for $C_{15}H_{23}NOSi$ (M^+): 261.1552; found: 235.158.

2-(4-Phenoxyphenyl)-2-(trimethylsilyloxy)acetonitrile (entry 6)

^{1}H NMR ($CDCl_3$, 200 MHz): 0.22 (s, 9H), 5.46 (s, 1H), 6.99–7.13 (m, 5H), 7.31–7.44 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.26$, 63.18, 117.50, 118.75, 119.48, 120.42, 124.04, 124.94, 128.02, 128.41, 129.92, 130.73, 131.98, 156.38, 158.84. HRMS (EI): m/z calcd for $C_{17}H_{19}NO_2Si$ (M^+): 297.1185; found: 297.118.

4, 8-Dimethyl-2-(trimethylsilyloxy)nona-3, 7-dienenitrile (entry 7)

^{1}H NMR ($CDCl_3$, 200 MHz): 0.22 (s, 9H), 1.62 (s, 3H), 1.70–1.80 (m, 6H), 2.10–2.12 (m, 4H), 5.09–5.13 (m, 2H), 5.31–5.33 (m, 1 H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.12$, 16.76, 17.65, 23.18, 25.83, 39.13, 58.42, 119.50, 120.59, 123.14, 133.04, 142.82. HRMS (EI): m/z calcd for $C_{14}H_{25}NOSi$ (M^+): 251.1705; found: 251.171.

2-(Trimethylsilyloxy)pent-3-enenitrile (entry 8)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.20$ (s, 9H), 1.79 (dd, 3H, 2.4 Hz, 2.4 Hz), 4.90 (d, 1H, 8.4 Hz), 5.51–5.62 (m, 1H), 5.93–6.04 (m, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.39$, 17.16, 61.91, 118.55, 126.06, 130.98. HRMS (EI): m/z calcd for $C_8H_{15}NOSi$ (M^+): 169.0922; found: 169.092.

3-Methyl-2-trimethylsilyloxybutanenitrile (entry 9)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.25$ (s, 9H), 1.09 (m, 1H), 1.97 (m, 1H), 4.17 (1H). ^{13}C ($CDCl_3$, 50 MHz): −0.31, 18.15, 32.26, 68.67, 118.42. HRMS (EI): m/z calcd for $C_8H_{17}NOSi$ (M^+): 171.1079; found: 171.124.

3,3-Dimethyl-2-trimethylsilyloxybutyronitrile (entry 10)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.22$ (s, 9H), 1.03 (s, 9H), 4.00 (s, 1H). ^{13}C ($CDCl_3$, 50 MHz): −0.32, 25.45, 33.42, 76.58, 118.49. HRMS (EI): m/z calcd for $C_9H_{19}NOSi$ (M^+): 185.1236; found: 185.124.

2-Furanyl (trimethylsilyloxy)acetonitrile (entry 11)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.22$ (s, 9H), 5.55 (s, 1H), 6.41–6.43 (m, 1H), 6.55–6.57 (m, 1H), 7.28–7.48 (m, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.43$, 57.41, 109.70, 110.78, 117.11, 143.84, 148.20. HRMS (EI): m/z calcd for $C_9H_{13}NO_2Si$ (M^+): 195.0715; found: 195.071.

2-(trimethylsilyloxy)-2-phenylpropanenitrile (entry 12)

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 0.17$ (s, 9H), 1.86 (s, 3H) 7.36–7.53 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = -0.89$, 33.40, 71.45, 121.43, 124.45, 128.49, 141.86. HRMS (EI): m/z calcd for $C_{12}H_{17}NOSi$ (M^+): 219.1079; found: 219.109.

Trimethylsilane carbonitrile (TMSCN)

^{13}C NMR (400 MHz): −1.73, 1.99, 127.00.

A Mixture of trimethylsilane carbonitrile and triphenylphospine oxide (TMSCN + Ph_3PO)

^{13}C NMR (400 MHz): −1.73, 1.38, 1.98, 127.00, 128.44, 131.99.

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