Poly(*N*,*N*′-dichloro-*N*-ethylbenzene-1,3-disulfonamide) and *N*,*N*,*N*′,*N*′-Tetrachlorobenzene-1,3-disulfonamide as Novel Reagents for the Synthesis of *N*-Chloroamines, Nitriles and Aldehydes

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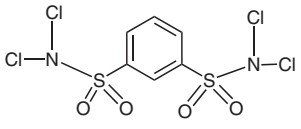
Abstract: The applications of poly(*N*,*N*′-dichloro-*N*-ethylbenzene-1,3-disulfonamide) (PCBS) and *N*,*N*,*N*′,*N*′-tetrachlorobenzene-1,3-disulfonamide (TCBDA) as novel reagents for the preparation of *N*,*N*-dichloroamines, nitriles, and aldehydes from primary amines under various conditions are described. Also, a simple and effective procedure for the direct oxidative conversion of primary alcohols into nitriles was successfully carried out with TCBDA and PCBS in aqueous ammonia.

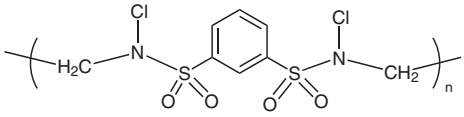
Key words: TCBDA, PCBS, dichloroamines, nitriles, alcohols

The oxidation of primary and secondary amines into the corresponding chloroamines and oxidation of primary amines into nitriles constitutes a very useful functional group transformation in organic synthesis.[1–3] Consequently, there is a great deal of interest in finding out methods for their synthesis under mild conditions. A typical method is the reaction of alkyl halides with very toxic metal cyanides via a nucleophilic pathway; the length of the carbon chain is increased by one during this process. Due to the toxicity of metal cyanides, nitriles are generally prepared by the dehydration of amides with $SOCl_2$, TsCl/pyridine, P_2O_5, $COCl_2$, $(EtO)_3P/I_2$, or Ph_3P/CCl_4.[4] It has been reported that nitriles can be prepared from carboxylic acid or its derivatives,[5] amides,[6,7] aldehydes,[8,9] oximes,[10] and nitro compounds.[11] Nitrile synthesis from amines has been one of the classical routes. Numerous metal-based oxidants, such as nickel peroxide,[12] silver reagents,[13] copper reagents,[14] OsO_4,[15] NaOCl,[16] PhIO,[17] TCCA with TEMPO[18] or Et_3N,[19] IBX/I_2,[20] I_2/NH_3,[21] 1,3-diiodo-5,5-dimethylhydantoin in aqueous NH_3,[22,23] and ruthenium reagents[24] have been used for carrying out this transformation. However, there are only a few reports on the direct oxidative conversion of primary alcohols into nitriles in a one-pot procedure, that is, using NH_4HCO_3, $(Bu_4N)_2S_2O_8$, and a catalytic amount of $Cu(HCO_2)_2{\cdot}Ni(HCO_2)_2$ in aqueous KOH and *i*-PrOH,[25] and MnO_2, NH_3, and $MgSO_4$ in THF and *i*-PrOH for benzylic and cinnamic alcohols. Recently, I_2[26] and DIH[22] in aqueous NH_3 were reported by Togo for this conversion. *N*,*N*-Dichloroamines are very useful intermediates in synthetic organic chemistry. Trichloroisocyanuric acid (TCCA)[19] and NaOCl[3] were reported for the synthesis of dichloroamines under various conditions.

In continuation of our interest in the synthesis of *N*-halosulfonamide and application of these reagents in organic synthesis,[27–32] we report here the synthesis of poly(*N*,*N*′-dichloro-*N*-ethylbenzene-1,3-disulfonamide) (PCBS) and *N*,*N*,*N*′,*N*′-tetrachlorobenzene-1,3-disulfonamide (TCBDA) as novel reagents in organic reactions (Figure 1).



TCBDA

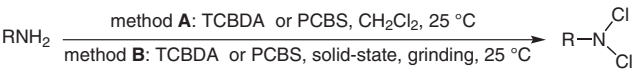


PCBS

Figure 1 Structures of TCBDA and PCBS

The advantages of TCBDA and PCBS are as follows: The preparation of TCBDA and PCBS are easy and convenient. TCBDA and PCBS are stable for two to three months under atmospheric conditions (25 °C, air). After completion of the reaction, the sulfonamides are recovered and can be reused many times without decreasing the yield.

In order to apply these novel reagents, we were interested in studying the possibility of chlorination of amines under various conditions (methods A and B, Scheme 1). Thus, benzylamine, as a model substrate, was dissolved in dichloromethane and treated with an equimolar amount of TCBDA at room temperature. After 10 minutes, TLC analysis showed the complete absence of the amine and the workup of the reaction mixture afforded *N*,*N*-dichlorobenzylamine in quantitative yield.



R = alkyl, aryl

Scheme 1

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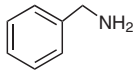
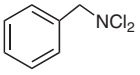
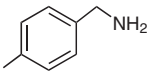
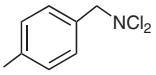
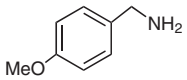
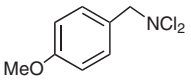
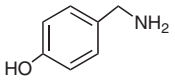
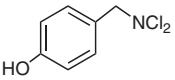
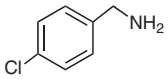
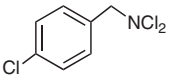
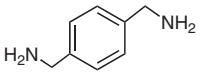
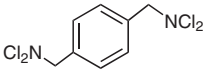
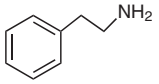
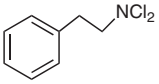
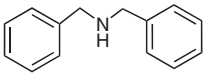
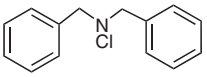
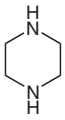
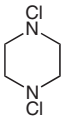
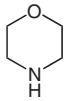
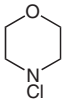
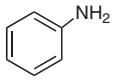
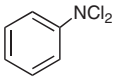
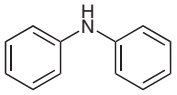
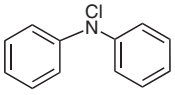
Table 1 Formation of Chloramines from Amines by Reaction with TCBDA and PCBS Under Various Conditions

Entry	Amine	Product[a]	TCBDA (Method A)		PCBS (Method A)		TCBDA (Method B)		PCBS (Method B)	
			Time (min)	Yield (%)[b]	Time (min)	Yield (%)	Time (min)	Yield (%)	Time (min)	Yield (%)
1			5	98	10	96	1	98	1	96
2			10	92	15	90	1	96	1.5	96
3			8	96	10	90	1	99	2	98
4			15	90	15	85	1	98	1.5	96
5			20	90	25	87	1.5	95	2	90
6			15	89	15	80	1.5	90	2.5	85
7			10	90	12	90	1	96	2	92
8			15	92	18	87	1.5	92	2.5	90
9			5	96	5	95	1	99	1	98
10			5	95	7	90	1	98	2	96
11			10	90	10	85	2	90	2	90
12			300	0	300	0	10	0	10	0
13			300	0	300	0	10	0	10	0

[a] The products were characterized by comparison of their spectroscopic and physical data with those of samples synthesized by reported procedures.
[b] Yields refer to pure isolated products.

The reaction reported in Scheme 1 was then applied to other primary and secondary amines. As shown in Table 1, various types of amines (aromatic and aliphatic) were cleanly and rapidly converted to the corresponding *N*,*N*-dichloroamines in CH_2Cl_2 using PCBS and TCBDA at 25 °C. The benzylic amines and aliphatic amines were more effective in this reaction. Secondary amines reacted also to give the chloramines (Table 1, entries 8–10). But, aniline and diphenylamine did not react under these conditions.

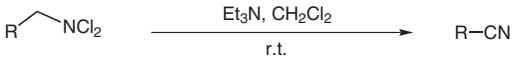
Since, in recent years, there has been an increasing interest in reactions that proceed in the absence of solvents due to their reduced pollution, low cost, simplicity in process, and handling, we decided to test the N-chlorination of benzylamine under solvent-free conditions (grinding)

with these reagents. We found that the reaction was rapid (1 min) and proceeds with an excellent yield (98%, Table 1, entry 1) using TCBDA, and in excellent yield (96%, 1 min, Table 1, entry 1) using PCBS. Thus, to test the generality and versatility of this procedure for the N-chlorination of amines, we have examined a number of aromatic and aliphatic amines using the optimized conditions (Table 1). Also, the results of Table 1 show that this method is the better than other methods using TCCA[19] for the preparation of chloramines, as summarized Table 2.

Table 2 Comparison of the Present Method with the Published Method in Terms of Reaction Times and Yields

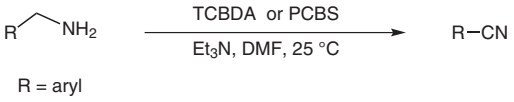
Substrate	Conditions	Time	Yield (%)	Ref.
4-chlorobenzylamine	TCCA	1 h	77	–[19]
4-chlorobenzylamine	TCBDA (Method A)	20 min	90	–
4-chlorobenzylamine	TCBDA (Method B)	1.5 min	95	–
dibenzylamine	TCCA	0.5 h	75	–[19]
dibenzylamine	TCBDA (Method A)	15 min	92	–
dibenzylamine	TCBDA (Method B)	1.5 min	92	–
benzylamine	TCCA	1 h	98	–[19]
benzylamine	TCBDA (Method A)	1 min	98	–
benzylamine	TCBDA (Method B)	1 min	98	–

Since *N*,*N*-dichloroamines may be synthetic intermediates for the preparation of nitriles and carbonyl compounds,[33] we have treated the prepared *N*,*N*-dichloroamines with 3 equivalents of triethylamine in CH_2Cl_2 (Scheme 2). The results shown in Table 3 indicate that the desired nitriles are obtained in good yields.



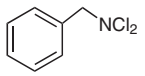
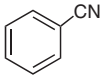
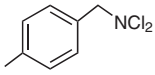
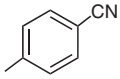
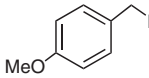
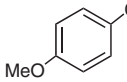
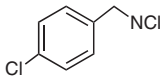
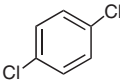
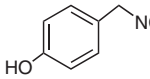
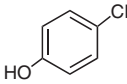
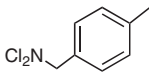
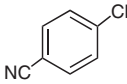
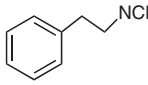
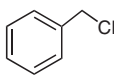
Scheme 2

Furthermore, we have checked the possibility to carry out the reaction in a one-pot procedure for preparing nitriles from amines. Best results were obtained in DMF and by introducing triethylamine directly in the reaction mixture. In this case, the reaction can be carried out in a single step: under these conditions, benzylamine afforded benzonitrile within one hour in 96% yield (Scheme 3). The results are shown in Table 4.



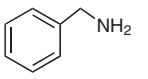
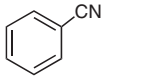
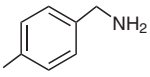
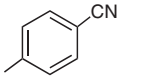
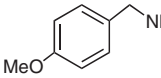
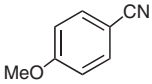
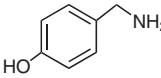
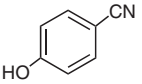
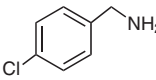
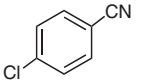
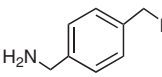
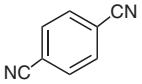
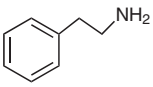
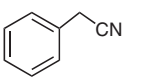
Scheme 3

Table 3 Reaction of *N*,*N*-Dichloroamines with Triethylamine

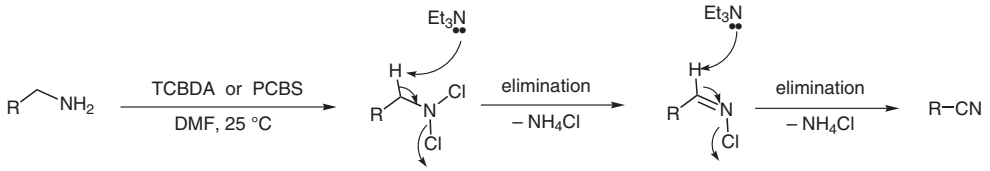
Entry	Amine	Product[a]	Time (h)	Yield (%)
1			1.5	96
2			1.2	93
3			2	96
4			1	95
5			2	90
6			3.5	82
7			2	90

[a] The products were characterized by comparison of their spectroscopic and physical data with those of samples synthesized by reported procedures.

Table 4 Oxidative Conversion of Primary Amines into Nitriles with TCBDA and PCBS

Entry	Amine	Product[a]	TCBDA		PCBS	
			Time (h)	Yield (%)	Time (h)	Yield (%)
1			1	96	1.2	92
2			1	95	1.5	90
3			1.5	90	2	90
4			1.5	90	2	88
5			1	91	2	90
6			2	90	3	80
7			2	95	2.5	87

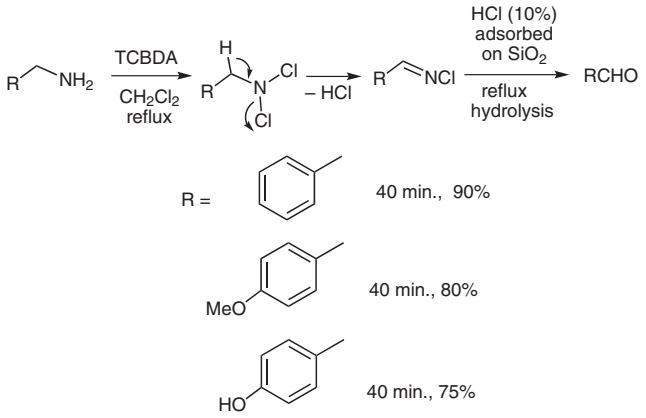
[a] The products were characterized by comparison of their spectroscopic and physical data with those of samples synthesized by reported procedures.



Scheme 4

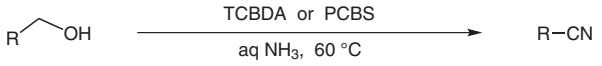
A plausible mechanism for the conversion of a primary amine into nitrile is shown in Scheme 4. According to this mechanism, the initial N-chlorination of the amine with TCBDA or PCBS occurs to form the *N*,*N*-dichloroamines. This is followed by a β-elimination of HCl by triethylamine, leading to the nitrile.[19,20]

We found that TCBDA was suitable for the conversion of benzylamines to the corresponding carbonyl compounds in good to high yields under reflux conditions in the presence of HCl (10%) adsorbed on SiO_2 (Scheme 5).[19]



Scheme 5

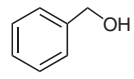
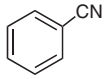
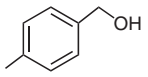
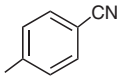
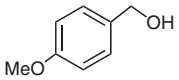
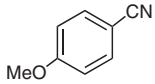
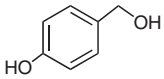
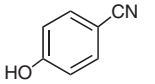
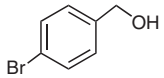
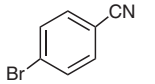
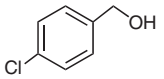
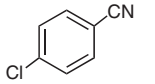
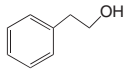
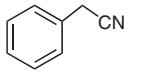
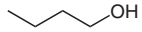
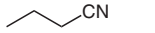
Our reagents TCBDA and PCBS were also tested for the oxidation of alcohols and found that these reagents were able to oxidize alcohols under mild conditions. Our next study was a practical and facile method for the oxidative conversion of alcohols to the corresponding nitriles directly, using PCBS and TCBDA as novel reagents in aqueous ammonia (Scheme 6).



Scheme 6

Thus, the present reaction was carried out by treating benzyl alcohol (1 mmol) with TCBDA (1.1 mmol) in aqueous ammonia (30%, 3 mL) at 60 °C to provide the corresponding nitriles in high yields (Table 5). When the same reaction was carried out in 0.5 mmol of the reagent, instead of 1.1 mmol, the yield of nitrile decreased. A similar result was obtained with PCBS under these conditions. Various primary and benzylic alcohols were treated with TCBDA and PCBS in aqueous ammonia under the same conditions, as shown in Table 5. The corresponding nitriles were obtained in good to high yields (Table 5, entries 1–8).

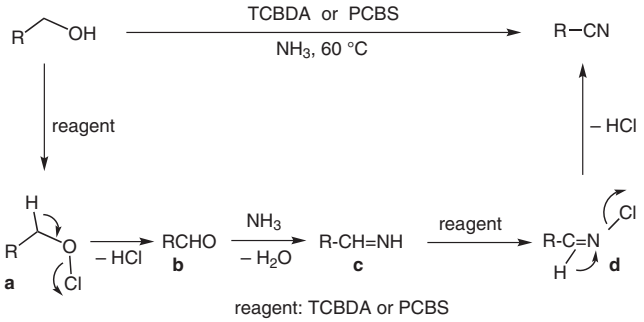
Table 5 Oxidative Conversion of Primary Alcohols into Nitriles with TCBDA and PCBS

Entry	Alcohol	Product[a]	TCBDA		PCBS	
			Time (h)	Yield (%)	Time (h)	Yield (%)
1			1	95	1.5	90
2			1.1	86	1.5	80
3			1.5	90	2	80
4			2	85	2.5	75
5			2.5	88	2.5	81
6			1	95	1.5	90
7			3.5	90	4	90
8			5	85	5	82

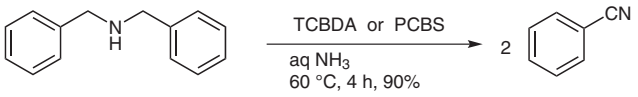
[a] The products were characterized by comparison of their spectroscopic and physical data with those of samples synthesized by reported procedures.

A plausible reaction pathway for the conversion of primary alcohols into nitriles is shown in Scheme 7. According to this pathway, the initial O-chlorination of the alcohol with TCBDA or PCBS occurs to form the *O*-chloro compounds **a**, followed by β-elimination of HCl by ammonia to form the aldehyde **b**. This aldehyde reacts with ammonia to form an aldimine **c**. Then, the aldimine **c** reacts with TCBDA or PCBS in the presence of ammonia to form an *N*-chloroaldimine **d**, followed by β-elimination of HCl by ammonia to generate the corresponding nitrile.[26]

We found that under these conditions, dibenzylamine reacted with TCBDA or PCBS in aqueous NH_3 to provide benzonitrile in good yield (Scheme 8).

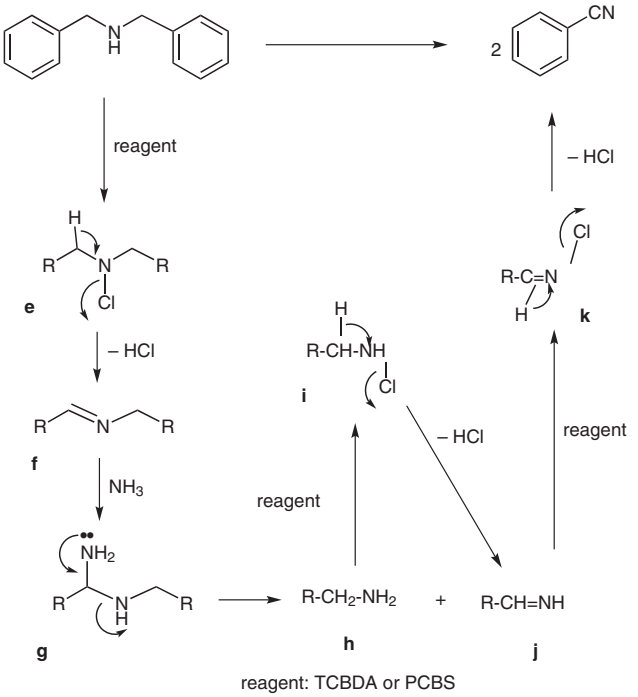


Scheme 7



Scheme 8

Based on these results, a plausible reaction pathway for the conversion of dibenzylamine into nitrile is shown in Scheme 9. Thus, initial N-chlorination of the amine occurs to give the N-chloro secondary amine **e**, followed by a β-elimination of HCl to form the imino compound **f**. Once imino compound **f** is formed, it reacts with aqueous NH_3 to form the 1,1-diamino compound **g**, which smoothly decomposes to the primary amine **h** and imine **j**. The primary amine **h** react with TCBDA or PCBS to form the *N*-chloroamine **i**, followed by a β-elimination of HCl to generate imine **j**. Imine **j** reacts with TCBDA or PCBS to form the *N*-chloroimine **k**, and finally elimination of HCl by NH_3 occurs to provide the corresponding nitrile.[21]



Scheme 9

In conclusion, PCBS and TCBDA are novel and efficient reagents for the formation of *N*,*N*-dichloroamines, nitriles, and aldehydes from primary amines, and good reagents for oxidative conversion of primary alcohols into the corresponding nitriles in good to high yields in aqueous ammonia. Prominent advantages of these new methods are their mild reaction conditions, operational simplicity, easy reaction workup, and generality of the reactions.

All commercially available chemicals were obtained from Merck and Fluka companies, and used without further purifications unless otherwise stated. Nuclear magnetic resonance (NMR) spectra were recorded on Bruker Avance 300 MHz and Jeol 90 MHz FT NMR spectrometers. Infrared (IR) spectroscopy was conducted on a Perkin Elmer GX FT-IR spectrometer. All yields refer to isolated products.

Poly(*N*,*N*′-dichloro-*N*-ethylbenzene-1,3-disulfonamide) (PCBS) and *N*,*N*,*N*′,*N*′-Tetrachlorobenzene-1,3-disulfonamide (TCBDA)

A sample of white, finely powdered poly(*N*-ethylbenzene-1,3-disulfonamide) (1 g) or benzene-1,3-disulfonamide (1 g, 4.9 mmol) was dissolved in a solution of NaOCl (50 mL, 14%) at 25 °C for 30 min. The color of the solution did not change. After this time, AcOH (20 mL, 50%) was added to the solution. The insoluble chlorinated reagent was removed by filtration and washed with H_2O (5 mL) to give PCBS or TCBDA, respectively.

***N*,*N*,*N*′,*N*′-Tetrachlorobenzene-1,3-disulfonamide**

Yield: 1.28 g (80%); white solid; mp 145–147 °C.

IR (KBr): 3050, 2950, 2900, 1570, 1462, 1417, 1377, 1304, 1167, 1082, 807, 776, 675 cm^{-1}.

^1H NMR (FT-250 MHz, $CDCl_3$): δ = 7.95–8.09 (m, CH_{arom}, 1 H), 8.11–8.58 (m, CH_{arom}, 2 H), 8.79 (s, CH_{arom}, 1 H).

MS: *m*/*z* = 374 ($[M + H]^+$), 339, 337, 321, 319, 305, 303, 272, 269, 267, 156, 154, 139, 125, 120, 104, 91, 77, 63.

Anal. Calcd for $C_6H_4Cl_4N_2O_4S_2$: C, 19.25; H, 1.06; N, 7.48; S, 17.11. Found: C, 19.05; H, 1.16; N, 6.94; S, 16.28.

Poly(*N*,*N*′-dichloro-*N*-ethylbenzene-1,3-disulfonamide)

Yield: 0.9 g (78%); white solid; mp 175–178 °C.

IR (KBr): 3050, 2950, 2900, 1578, 1462, 1418, 1377, 1303, 1168, 1081, 809, 779, 674, 603, 570 cm^{-1}.

^1H NMR (FT-250 MHz, $CDCl_3$): δ = 7.56–8.48 (br, CH_{arom}).

Oxidation of Benzylamines to *N*,*N*-Dichlorobenzylamine in Solvent; Typical Procedure; Method A

Benzylamine (214 mg, 2 mmol) was dissolved in CH_2Cl_2 (3 mL) and treated with TCBDA (0.6 mmol) or PCBS (0.5 g) at 25 °C. After the addition, the mixture was stirred for the required time until completion. After 5–10 min, TLC analysis showed the complete absence of the amine, the mixture was filtered on Celite and the solvent was evaporated to yield *N*,*N*-dichlorobenzylamine, which was isolated without further purification (oil, 98%).

***N*,*N*-Dichlorobenzylamine under Solvent-Free Conditions (Grinding); Typical Procedure; Method B**

Benzylamine (214 mg, 2 mmol) and the reagent PCBS (0. 5 g) or TCBDA (0.6 mmol) were added to a mortar and the mixture was pulverized with a pestle. A spontaneous reaction took place [1–2.5 min, Table 1, monitored by TLC (4:1, hexane–acetone)]. After completion of the reaction, CH_2Cl_2 (5 mL) was added, and the in-

soluble reagent was removed by filtration. The filtrate was evaporated under reduced pressure to give the product in good yield.

Nitriles from *N*,*N*-Dichloroamines

The *N*,*N*-dichloroamine (1 mmol) was dissolved in CH_2Cl_2 (3 mL) and treated with Et_3N (3 mL) at 25 °C. After completion of the reaction [1–3.5 h, Table 3, monitored by TLC (3:1, hexane–acetone)], the mixture was washed with H_2O (10 mL) and HCl (10%, 5 mL). The organic layer was dried ($MgSO_4$), filtered, and concentrated in vacuo to obtained the desired product in high yield.

Nitriles from Amines; General One-Pot Procedure

Amine (1 mmol) was dissolved in DMF (3 mL) and treated with Et_3N (2.56 mL, 3 mmol) and with PCBS (0. 5 g) or TCBDA (0.6 mmol) at 25 °C. After completion of the reaction [1–2 h, Table 4, monitored by TLC (3:1, hexane–acetone)], the mixture was quenched with H_2O (10 mL), and extracted with CH_2Cl_2 (2 × 15 mL). The combined organic layers were washed with H_2O (5 mL), HCl (10%, 5 mL), dried ($MgSO_4$), and the solvent evaporated to give the crude product. The crude product was purified by TLC using *n*-hexane–acetone (80:20) as eluent to afford the pure products.

Aldehydes from Amines; General One-Pot Procedure (Scheme 5)

Amine (1 mmol) was dissolved in CH_2Cl_2 (5 mL) and treated with TCBDA (0.5 mmol) at 25 °C. After the addition, the mixture was stirred for 10 min under reflux. Then, silica gel (1 g) treated with aq 10% HCl was added and the mixture was refluxed for 30 min. After completion of the reaction, CH_2Cl_2 (5 mL) was added, and the insoluble SiO_2 was removed by filtration. The filtrate was quenched with H_2O (10 mL) and extracted with CH_2Cl_2 (2 × 15 mL). The organic layers were dried ($MgSO_4$), and the solvent was evaporated. The product benzaldehyde was purified by column chromatography on silica gel (hexane–acetone, 9:1).

Oxidative Conversion of Primary Alcohols into Nitriles; General Procedure

To a mixture of benzyl alcohol (108 mg, 1 mmol) and aq ammonia (3 mL) was added PCBS (0.8 g) or TCBDA (1.1 mmol) at 25 °C. The mixture obtained was stirred at 60 °C. After completion of the reaction [1–2.5 h, Table 5, monitored by TLC (6:1, hexane–acetone)], the mixture was quenched with aq HCl (10%, 10 mL), and extracted with CH_2Cl_2 (2 × 15 mL). The organic layer was dried ($MgSO_4$) and evaporated to provide the corresponding nitrile in good yield. If necessary, the product was purified by column chromatography on silica gel (hexane–EtOAc, 4:1).

Acknowledgment

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