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One-pot synthesis of 2-amino-3-cyanopyridine derivatives under solvent-free conditions

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ABSTRACT

A series of 2-amino-3-cyanopyridines were obtained from aryl aldehydes, substituted acetophenones, malononitrile and ammonium acetate in good to excellent yields by proceeding through a simple, mild and efficient procedure utilizing *N,N,N′,N′*-tetrabromobenzene-1,3-disulfonamide [TBBDA] and poly(*N*-bromo-*N*-ethylbenzene-1,3-disulfonamide) [PBBS] as catalysts.

1. Introduction

Heteroaromatic rings containing nitrogen atoms often play important roles as the scaffolds of bioactive substances. [1] The pyridine ring system is one of the most popular *N*-heteroaromatics incorporated into the structure of many pharmaceuticals. Among them, 2-amino-3-cyanopyridine derivatives are known to have multiple biological activities, such as anti-microbial [2], cardiotonic, [3] anti-inflammatory [4,5], anti-parkinsonism [6] and anti-tumor properties. [7] Also, they have been identified as novel IKK-β inhibitors [4], A_{2A} adenosine receptor antagonists [6] and potent inhibitor of HIV-1 integrase (Fig. 1) [8]. Despite the existence of extensive literature for the synthesis of 2-amino-3-cyanopyridines, the most common procedures need multiple steps [9], toxic benzene or toluene as the solvent [7,10], microwave assistance [11], resulting in unsatisfactorily low yields. Due to the interesting properties of pyridines, the development of synthetic methods that enable a facile access to these heterocycles is desirable.

2. Experimental

All commercially available chemicals were obtained from Merck and Fluka companies, and used without further purification unless otherwise stated. Nuclear magnetic resonance (NMR) spectra were recorded in DMSO-d_6 on Bruker Avance 300 MHz FT and Varian 90 MHz NMR spectrometers using TMS as an internal standard. Chemical shifts were expressed in parts per million (ppm). Infrared (IR) spectroscopy was conducted on a PerkinElmer GX FT–IR spectrometer. Mass spectra were recorded on a Shimadzu QP 1100 BX Mass Spectrometer. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyzer.

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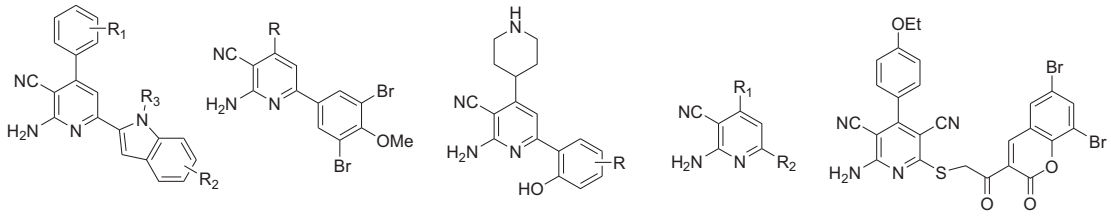


Fig. 1. a: anti-tumor; b: anti-microbial; c: novel IKK-β inhibitors; d: A_{2A} adenosine receptor antagonists; e: HIV-1 integrase.

2.1. General procedure for the synthesis of 2-amino-3-cyanopyridines

A mixture of aldehyde (2 mmol), substituted acetophenone (2 mmol), malononitrile (2 mmol), ammonium acetate (2.5 mmol) and TBBDA or PBBS (0.05 g) was heated under stirring at 100 °C for appropriate times (Table 1). The progress of the reaction was monitored by TLC (10:4, *n*-hexane/acetone). After completion of the reaction, the reaction mixture was allowed to cool to room temperature, and 95% cold EtOH (5 mL) was added. The precipitate was filtered off and washed with cold ethanol. After drying, the pure product was obtained. Removal of the solvent under reduced pressure gave back the catalysts.

2.2. Physical and spectroscopic data

2.2.1. 2-Amino-4,6-bis(3-chlorophenyl)nicotinonitrile

Cream solid, mp: 170–173 °C. IR (KBr): 3435, 33.9, 3215, 2210, 1646, 1567, 1477, 1081, 784 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 7.11 (s, NH_2, 2H), 7.57–8.19 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 87.72, 109.89, 117.05, 126.35, 127.41, 127.69, 128.70, 129.96, 130.34, 130.99, 133.87, 134.13, 139.23, 139.95, 154.03, 157.46, 161.15. Anal. calcd for $C_{18}H_{11}Cl_2N_3$: C, 63.55; H, 3.26; N, 12.35. Found: C, 63.93; H, 2.95; N, 12.21. MS: *m/z* 339 (Table 1, **5a**).

2.2.2. 2-Amino-4-(3-chlorophenyl)-6-(4-chlorophenyl)nicotinonitrile

Yellow solid, mp: 222–224 °C. IR (KBr): 3449, 3327, 3214, 2209, 1639, 1568, 1549, 1481, 1082, 786 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 7.09 (s, NH_2, 2H), 7.35–8.23 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 87.28, 109.60, 117.18, 127.62, 128.65, 129.08, 129.50, 129.92, 130.98, 133.88, 135.50, 136.63, 139.28, 153.85, 157.87, 161.19. Anal. calcd for $C_{18}H_{11}Cl_2N_3$: C, 63.55; H, 3.26; N, 12.35. Found: C, 63.71; H, 2.97; N, 12.16. MS: *m/z* 339 (Table 1, **5b**).

2.2.3. 2-Amino-4,6-bis(4-chlorophenyl)nicotinonitrile

Yellow solid, mp: 248–250 °C. IR (KBr): 3507, 3701, 2204, 1614, 1578, 1567, 1549, 1088, 829 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 7.06 (s, NH_2, 2H), 7.35–8.24 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 87.65, 109.86, 117.09, 126.36, 127.41, 127.71, 128.72, 129.74, 129.97, 130.36, 130.99, 133.86, 134.12, 139.22, 139.93, 154.03, 157.43, 161.16. Anal. calcd for $C_{18}H_{11}Cl_2N_3$: C, 63.55; H, 3.26; N, 12.35. Found: C, 63.86; H, 2.89; N, 12.46. MS: *m/z* 339 (Table 1, **5c**).

2.2.4. 2-Amino-6-(4-fluorophenyl)-4-p-tolylnicotinonitrile

Yellow solid, mp: 198–200 °C. IR (KBr): 3495, 3394, 2207, 1610, 1583, 1549, 1510, 1223, 1161, 819 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 2.38 (s, CH_3, 3H), 6.95 (s, NH_2, 2H), 7.29–8.18 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 21.33, 86.91, 109.36, 115.84, 116.13, 117.59, 128.72, 129.73, 129.96, 130.08, 134.49, 139.85, 155.40, 157.86, 161.31, 162.19. Anal. calcd for $C_{19}H_{14}FN_3$: C, 75.23; H, 4.65; N, 13.85. Found: C, 75.23; H, 4.16; N, 13.61. MS: *m/z* 303 (Table 1, **5d**).

2.2.5. 2-Amino-6-(4-bromophenyl)-4-p-tolylnicotinonitrile

Yellow solid, mp: 235–237 °C. IR (KBr): 3496, 3391, 2207, 1608, 1583, 1547, 1009, 815 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 2.40 (s, CH_3, 3H), 7.02 (s, NH_2, 2H), 7.30–8.13 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 21.34, 87.34, 109.47, 117.53, 124.24, 128.73, 129.73, 132.05, 134.41, 137.20, 139.89, 155.49, 157.70, 161.32. Anal. calcd for $C_{19}H_{14}BrN_3$: C, 62.65; H, 3.87; N, 11.54. Found: C, 62.75; H, 3.41; N, 11.29. MS: *m/z* 363 (Table 1, **5e**).

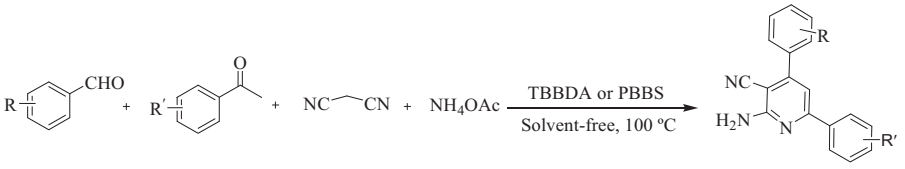
2.2.6. 2-Amino-6-(4-fluorophenyl)-4-(4-methoxyphenyl)nicotinonitrile

Brown solid, mp: 186–189 °C. IR (KBr): 3491, 3390, 2209, 1609, 1584, 1548, 1509, 1220, 826 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 3.82 (s, CH_3, 3H), 6.94–7.69 (m, CH aromatic, 9H), 8.07 (s, NH_2, 2H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 55.78, 86.81, 109.48, 114.59, 117.83, 127.68, 129.07, 129.52, 130.32, 130.47, 138.11, 154.92, 158.92, 160.86, 161.44. Anal. calcd for $C_{19}H_{14}FN_3O$: C, 71.46; H, 4.42; N, 13.16. Found: C, 71.08; H, 4.16; N, 13.25. MS: *m/z* 319 (Table 1, **5f**).

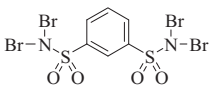
2.2.7. 2-Amino-6-(4-fluorophenyl)-4-(3-methoxyphenyl)nicotinonitrile

Brown solid, mp: 172–174 °C. IR (KBr): 3475, 3378, 2207, 1634, 1601, 1575, 1510, 1258, 834 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 3.83 (s, CH_3, 3H), 7.01 (s, NH_2, 2H), 7.21–8.27 (m, CH aromatic, 9H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 55.74, 87.09, 109.49, 114.26, 115.83, 116.12, 117.43, 120.98, 130.01, 130.12, 130.34, 134.48, 138.67, 155.27, 157.96, 159.75, 161.23. Anal. calcd for $C_{19}H_{14}FN_3O$: C, 71.46; H, 4.42; N, 13.16. Found: C, 71.28; H, 4.30; N, 13.09. MS: *m/z* 319 (Table 1 **5g**).

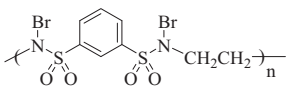
Table 1
Synthesis of 2-amino-3-cyanopyridine derivatives.



1 **2** **3** **4** **5(a-k)**



TBBDA



PBBS

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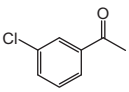
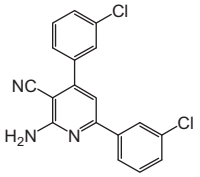
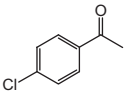
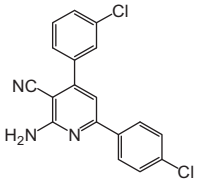
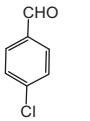
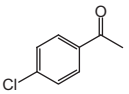
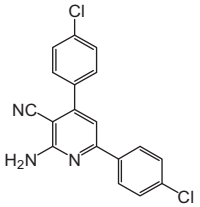
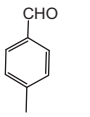
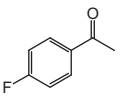
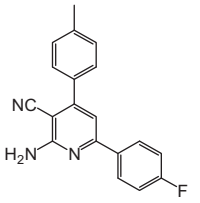
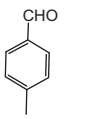
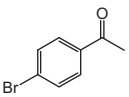
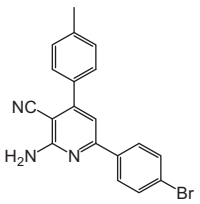
5a				10	90	10	85	170–173
5b				10	92	10	80	222–224
5c				5	94	5	90	248–250
5d				30	90	40	90	198–200
5e				30	88	40	85	235–237

Table 1 (*Continued*)

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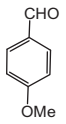
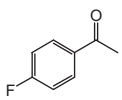
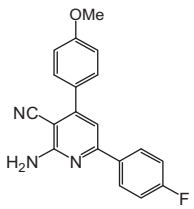
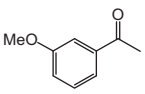
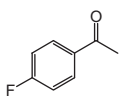
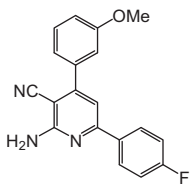
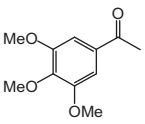
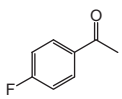
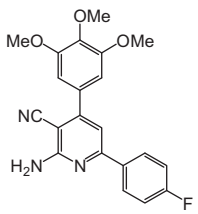
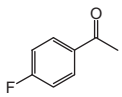
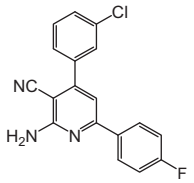
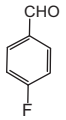
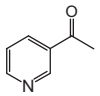
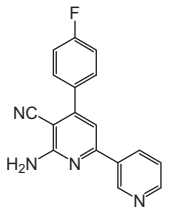
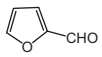
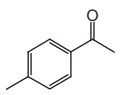
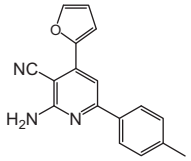
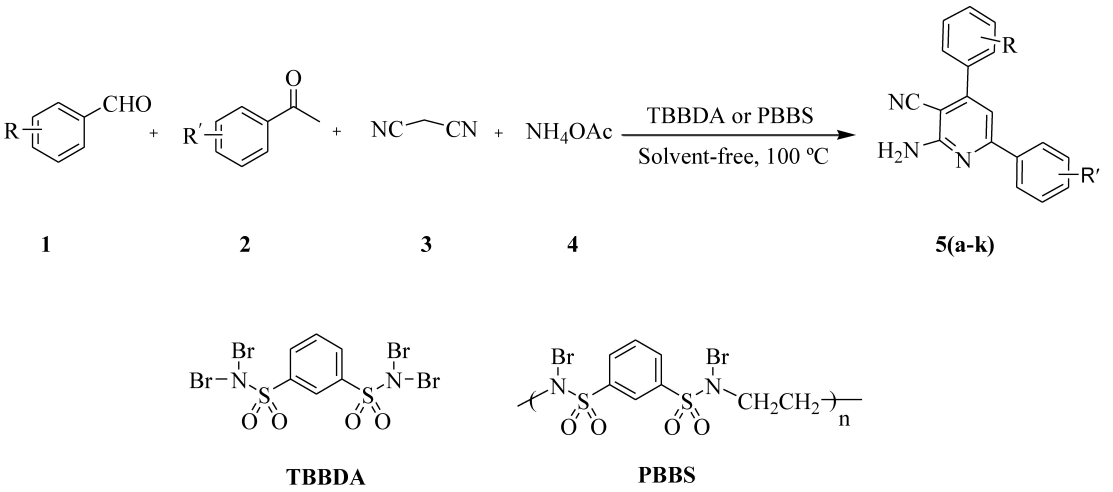
5f				25	94	25	90	186–189
5g				15	86	15	85	172–174
5h				35	85	40	85	235–237
5i				10	89	10	85	207–210
5j				15	85	15	79	269–273
5k				25	89	25	80	149–151

2.2.8. 2-amino-6-(4-fluorophenyl)-4-(3,4,5-trimethoxyphenyl)nicotinonitrile

Cream solid, mp: 235–237 °C. IR (KBr): 3497, 3370, 2213, 1635, 1555, 1508, 1130, 811 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 3.74 (s, CH_3, 3H), 3.86 (s, CH_3, 6H), 6.99–8.21 (m, CH aromatic and NH_2, 11H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 56.53, 60.56, 87.01, 106.47, 109.42, 115.85, 116.13, 117.71, 130.02, 130.14, 132.63, 134.52, 138.91, 153.31, 155.36, 157.80, 161.27. Anal. calcd for $C_{21}H_{18}FN_3O_3$: C, 66.48; H, 4.78; N, 11.08. Found: C, 66.18; H, 4.54; N, 10.62. MS: *m/z* 379 (Table 1 **5h**).

2.2.9. 2-amino-4-(3-chlorophenyl)-6-(4-fluorophenyl)nicotinonitrile

Cream solid, mp: 207–210 °C. IR (KBr): 3513, 3409, 2204, 1646, 1579, 1512, 1159, 834 cm^{-1}; ^1H NMR (90 MHz, DMSO-d_6): δ_H (ppm) 6.83–8.19 (m, CH aromatic and NH_2, 11H). ^{13}C NMR (75 MHz, DMSO-d_6): δ_c (ppm) 86.88, 94.50, 109.46, 115.87, 116.13, 118.97, 127.64, 128.66, 129.77, 130.06, 131.00, 133.86, 139.36, 148.61, 153.81, 154.49, 158.10, 161.19. Anal. calcd for $C_{18}H_{11}ClFN_3$: C, 66.78; H, 3.42; N, 12.98. Found: C, 67.33; H, 3.16; N, 12.20. MS: *m/z* 323 (Table 1 **5i**).



Scheme 1. One-pot synthesis of 2-amino-3-cyanopyridine derivatives.

2.2.10. 2-amino-4-(4-fluorophenyl)-6-(pyridin-3-yl)nicotinonitrile

Cream solid, mp: 269–273 °C. IR (KBr): 3476, 3362, 2215, 1650, 1578, 1514, 1193, 838 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ_H (ppm) 6.96 (s, CH, 1H), 7.03 (s, NH_2,2H), 7.42–7.47 (t, CH, 2H), 7.61–7.65 (m, CH, 1H), 7.77–7.80 (m, CH, 3H), 8.14–8.17 (m, CH, 1H), 8.76–8.77 (m, CH, 1H). ^{13}C NMR (100 MHz, DMSO-d_6): δ_c (ppm) 115.55, 115.77, 115.81, 118.58, 123.51, 130.97, 131.06, 133.28, 133.97, 146.58, 148.79, 149.01, 150.28, 153.96, 154.03, 161.59, 164.05. Anal. calcd for $C_{17}H_{11}FN_4$: C, 70.34; H, 3.82; N, 19.30. Found: C, 70.06; H, 3.99; N, 19.58. MS: *m/z* 290 (Table 1 **5j**).

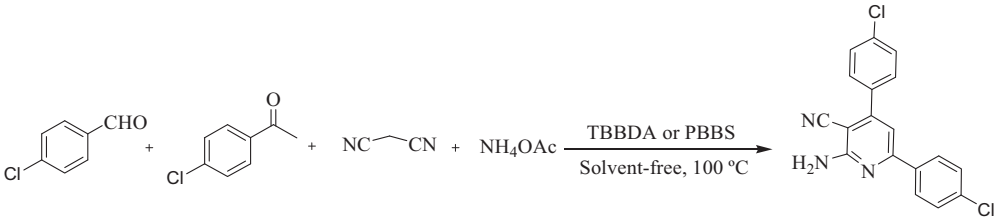
2.2.11. 2-amino-4-(furan-2-yl)-6-p-tolylnicotinonitrile

Cream solid, mp: 149–151 °C. IR (KBr): 3477, 3311, 2204, 1642, 1542, 1508, 1264, 805 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ_H (ppm) 2.38 (s, CH_3, 3H), 6.78–6.80 (m, CH, 1H), 6.98 (s, NH_2,2H), 7.31–7.33 (d, CH, 2H), 7.52–7.53 (d, CH, 2H), 8.01–8.04 (m, CH, 3H). ^{13}C NMR (100 MHz, DMSO-d_6): δ_c (ppm) 20.90, 103.95, 112.72, 113.00, 117.30, 127.00, 129.26, 134.63, 139.97, 141.18, 145.45, 145.46, 148.72, 158.7, 161.14. Anal. calcd for $C_{17}H_{13}N_3O$: C, 74.17; H, 4.76; N, 15.26. Found: C, 74.72; H, 4.56; N, 15.58. MS: *m/z* 275 (Table 1 **5k**).

3. Results and discussion

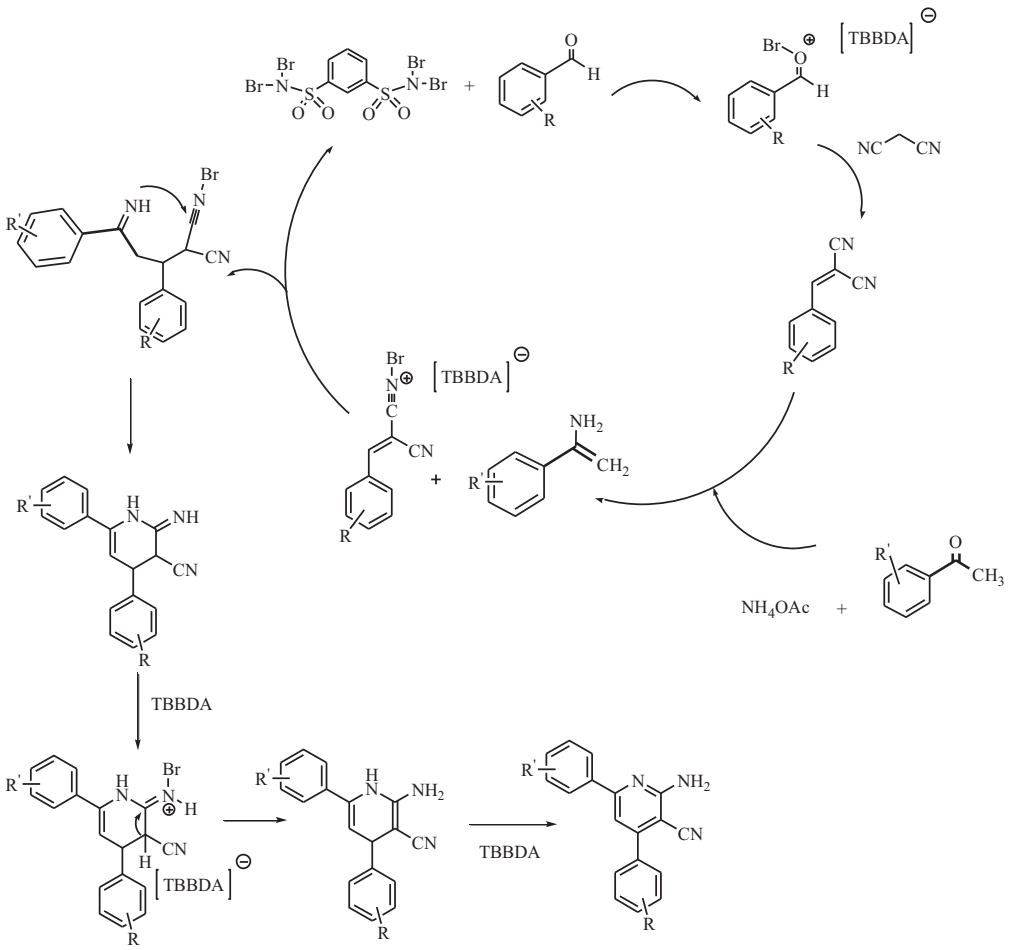
In continuation of our interest in the application of *N,N,N′,N′*-tetrabromobenzene-1,3-disulfonamide [TBBDA] and poly(*N*-bromo-*N*-ethylbenzene-1,3-disulfonamide) [PBBS] [12] in organic synthesis [13–22], we report a new and efficient method for the one-pot synthesis of 2-amino-3-cyanopyridine derivatives by the condensation of substituted acetophenone with various arylaldehydes, malononitrile and ammonium acetate in the presence of

Table 2
Reaction times and yields in various conditions.



Entry	Catalyst	Solvent	Amount of catalyst (mol%)	Temperature	Time (min)	Yield (%)[a]
1	None	Ethanol	–	Reflux	720	38
2	$ZnCl_2$	Ethanol	12.83	Reflux	720	36
3	$AlCl_3$	Ethanol	13.12	Reflux	720	18
4	$SnCl_2$	Ethanol	9.22	Reflux	720	34
5	TBBDA	Ethanol	4.53	Reflux	90	80
6	TBBDA	Solvent-free	4.53	100 °C	10	92 (92, 90, 89)[b]
7	TBBDA	Solvent-free	3.62	100 °C	10	88
8	TBBDA	Solvent-free	6.34	100 °C	20	90

[a] Isolated yield.
[b] The catalyst was reused for three times.



Scheme 2. Suggested mechanism for the synthesis of 2-amino-3-cyanopyridine derivatives.

TBBDA and PBBS as the efficient catalysts at 100 °C in good yields, as shown in Scheme 1.

Initially, we decided to examine various catalysts for the synthesis of 2-amino-4,6-*bis*(4-chlorophenyl)nicotinonitrile as a model compound (Table 2). We investigated the effects of various catalysts, including $ZnCl_2$, $SnCl_2$, $AlCl_3$ and *N,N,N′,N′*-tetrabromobenzene-1,3-disulfonamide [TBBDA] under various conditions. The results are summarized in Table 2. The reaction without catalyst provided little amounts of product (Table 2, Entry 1). The best result in ethanol was achieved using *N,N,N′,N′*-tetrabromobenzene-1,3-disulfonamide (Table 2, entry 5). In recent years, the synthesis of compounds under solvent-free conditions is an important challenge in heterocyclic synthesis. Therefore, we decided to test this solvent-free reaction with various ratios of TBBDA. We found that the reaction was rapid and gave excellent yield of the product when using *N,N,N′,N′*-tetrabromobenzene-1,3-disulfonamide [TBBDA] (10 min, 92%, Entry 6). In the light of this, subsequent studies were carried out under the following optimized conditions, that is, with 4.53 mol% TBBDA at 100 °C.

These results encouraged us to investigate the scope and generality of this new protocol for various aromatic aldehydes and ketones under optimized conditions. Surprisingly, while *para*-substituted, the *meta*-substituted aromatic aldehydes, the heteroaromatic aldehyde and the heteroaromatic ketone gave 2-amino-3-cyanopyridines in good to excellent yields, the *ortho*-substituted aromatic aldehydes, the aliphatic aldehyde (hexanale) and the aliphatic ketone (acetone) gave no products. Obviously, the reactivity of aldehydes is the key factor for this one-pot transformation.

Mechanistically, it is likely that these catalysts release Br^+ in situ, which can act as an electrophilic species and the mechanism shown in Scheme 2 is proposed for the synthesis of 2-amino-3-cyanopyridine derivatives [17,20].

4. Conclusion

In conclusion, we have developed a simple procedure for the synthesis of novel 2-amino-3-cyanopyridine derivatives from the reaction of various aryl aldehydes, substituted acetophenones, malononitrile and ammonium acetate in the presence of TBBDA and PBBS as the catalysts under solvent-free conditions. Moreover, the method has advantages in terms of product yields, recyclable catalyst, operational simplicity (easy work-up of reactions),environmental

friendliness (non-corrosive catalyst) and short reaction times.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.crci.2013.06.006.

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