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Copper (I) catalyzed domino synthesis of benzonitriles from benzenecarbaldehydes

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Abstract:

The synthesis of benzonitrile from benzenecarbaldehyde and hydroxylamine hydrochloride is delineated as a two-step, one-pot methodology; due to its economic viability, environmentally sustainable reaction conditions, high atom economy, and applicability for industrial settings, numerous researchers have reported on the one-pot synthesis of benzonitrile which aids in minimizing energy consumption, solvent waste, and the overall duration of the reaction. The one-pot conversion of benzenecarbaldehyde into nitriles signifies a pivotal functional group transformation in the domain of organic synthesis. A copper-phene-β-nitrostyrene catalytic system possesses the capability to promote the synthesis of benzonitriles from benzenecarbaldehydes, utilizing tosylhydrazine as a nitrogen source in acetonitrile at a controlled temperature of 60 °C. A wide variety of aromatic nitriles were synthesized with impressive yields through a direct one-pot approach. In this investigation, we found a tandem methodology for obtaining benzonitriles from aromatic aldehydes in acetonitrile. This streamlined process was adapted from a traditional method. In contrast to those methodologies, we employed β-nitrostyrene as an enhancer. In this one-pot conversion, we successfully obtained eleven benzonitriles with ease. The direct oximation of benzenecarbaldehyde to benzaldoxime, followed by dehydration with hydroxylamine in the presence of a catalytic quantity of copper iodide has been explored and documented.

Keywords: Benzonitriles, benzenecarbadehydes, tosylhydrazine, copper iodide,phen, β-nitrostyrene and domino synthesis.

Introduction:

Nitriles represent a prevalent class of organic functional groups (also found in certain inorganic compounds such as cyanide) characterized by a carbon atom that is covalently linked to a nitrogen atom via a triple bond. The structurally analogous functionalities of nitriles[1] encompass cyanates, thiocyanates, and isonitriles. Nitriles serve as integral components in various biologically active substances, including dyes[2], herbicides[3], agrochemicals[4], pharmaceuticals[5], medicinal compounds[6], pesticides[7], liquid crystal materials[8], cysteine protease inhibitors[9], agents targeting Alzheimer's disease[10], and antibiotics[11]. These compounds are often identified within a multitude of natural products[12] derived from microorganisms, fungi, flora, and fauna, as well as in both marine and terrestrial ecosystems. They function as valuable precursors in the domain of organic chemistry, serving as solvents and synthons[13]. For example, acetonitrile, the simplest organic nitrile, is extensively utilized as an organic solvent. Furthermore, the field of polymer chemistry[14] has produced numerous significant nitrile-containing polymers, including poly(acrylonitrile-co-butadiene-co-styrene), polyacrylonitrile, poly(acrylonitrile-co-butadiene)and poly (styrene-co-acrylonitrile).

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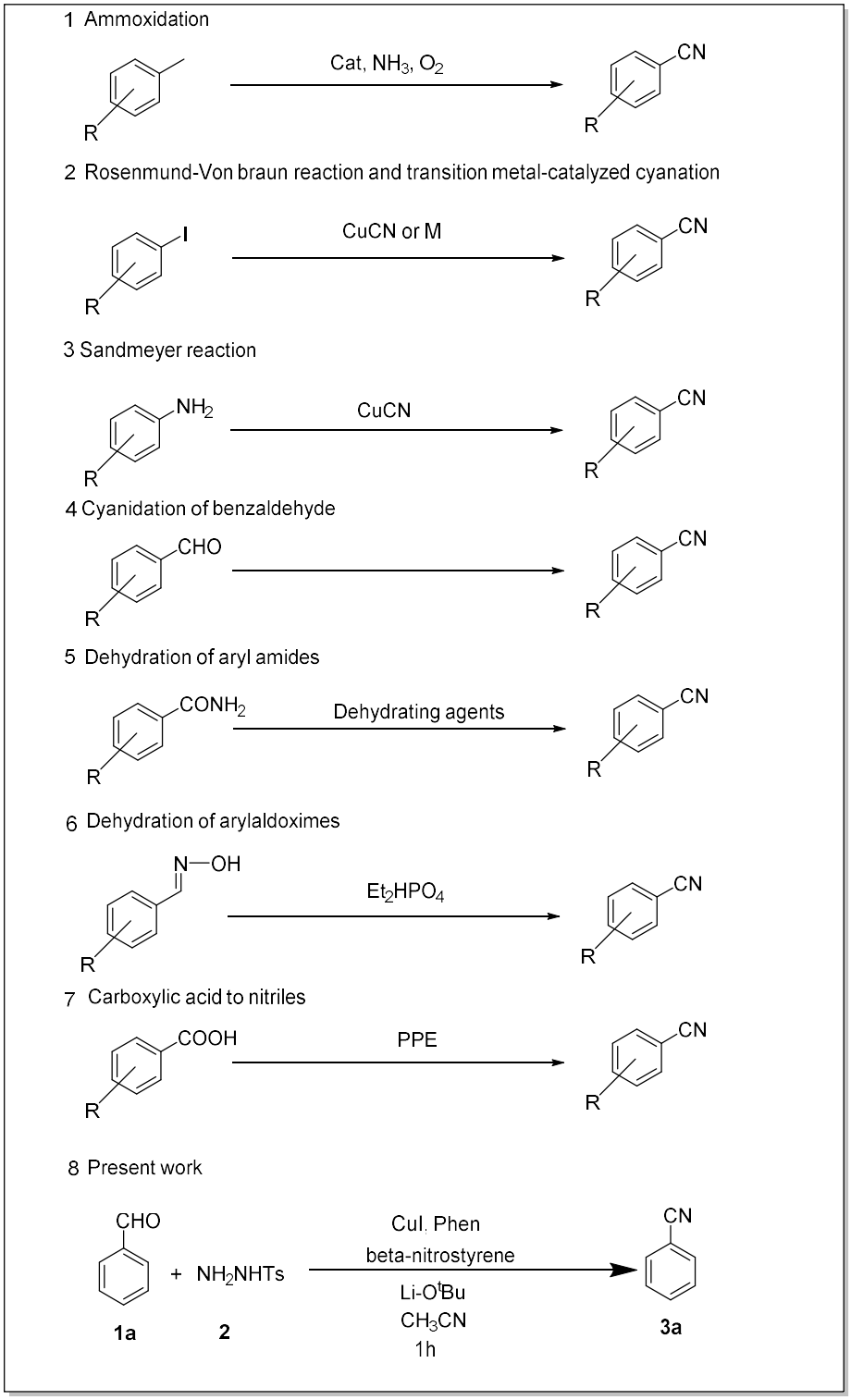
Scheme 1: Traditional methodologies utilized for the production of benzonitrile.



Classically, the synthesis of benzonitriles can be accomplished through various methodologies, including: (1) the ammoxidation of methyl-substituted aromatic compounds[15-16], (2) the cyanidation of aryl halides[17-18], (3) the Sandmeyer transformation of diazonium salts derived

from arylamines [19-20], (4) the cyanidation of benzaldehyde[21], (5) the dehydration of arylamides[22], (6) the dehydration of arylaldoximes[23-24], and (7) the reaction between benzoic acid and urea[25-36] (refer to **Scheme 1**). Furthermore, The formulation of benzonitrile from benzenecarbaldehyde and hydroxylamine hydrochloride is characterized as a two-step, one-pot approach, owing to its cost-effectiveness, environmentally benign reaction conditions, high atom economy, and suitability for industrial application, numerous scholars have documented the one-pot synthesis of benzonitrile (involving the oximation of benzenecarbaldehyde to yield benzaldoxime, followed by dehydration), which contributes to a reduction in energy consumption, solvent waste, and overall reaction duration.

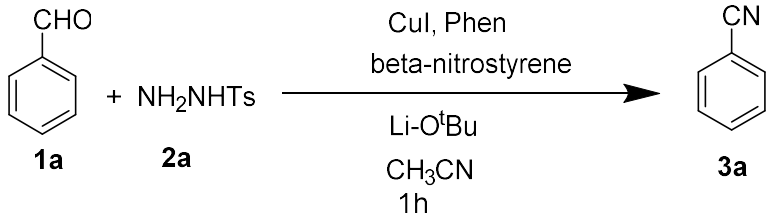
The domino transformation of aldehydes to nitriles represents a significant functional group conversion within the realm of organic synthesis. The interaction between aldehydes and azides is a well-known Schmidt reaction utilized for the generation of nitriles in a single pot[37-39]. Nonetheless, the predominant methodology employed is the oximation of benzenecarbaldehyde to benzaldoxime, followed by dehydration, which can be achieved using either hydroxylamine or ammonia in conjunction with a range of initiating substances[40-59]. A multitude of promoters has been documented for facilitating one-pot synthesis, including acids, oxone, H_2O_2, NBS, IBX, I_2, $NaICl_2$, among others. Recently, diverse catalytic systems have been developed for the oximation of benzenecarbaldehyde to benzaldoxime, followed by dehydration, encompassing palladium, [60-62], copper[63-64], iron[65], $Bi(OTf)_3$[66], zinc[67], and KF/Al_2O_3[68], among others. However, these methodologies exhibit certain limitations, which encompass stringent reaction conditions, the necessity of utilizing activated or preactivated catalysts, and are typically derived from restricted natural resources. Thus, it is imperative to devise innovative, convenient, and efficacious strategies to address these challenges. With this articulated aim, in alignment with our ongoing endeavors directed toward the advancement of new synthetic methodologies[69-71], the direct oximation of benzenecarbaldehyde to benzaldoxime, followed by dehydration with hydroxylamine in the company of a catalytic quantity of copper iodide (**Scheme 1, eq. 5**), has been investigated and documented.

Results and Reflections

At the onset of our study, we chose tosylamide (1mmol) and β-nitrostyrene(1mmol) as the model substrates to get asymmetric sulphones in the company of CuI (20 mol %), phen (20 mol %) and lithium tertiary butoxide (3 mmol) in acetonitrile at 80 ˚C for 1h, but accidentally, we got benzonitriles in excellent yield. Intrigued by the result, we performed the reaction with benzaldehyde (1mmol), tosyl hydrazine (1mmol), and β-nitrostyrene (1mmol) in the presence of phen (20 mol %), CuI (20 mol %), and lithium tertiary butoxide (3mmol) in acetonitrile at 80 ˚C for 1h. As anticipated, the desired benzonitrile formation was observed (**Table-1, entries 1**). In the absence of β-nitrostyrene, benzonitrile was not formed (**Table-1, entry 2**). In the absence of CuI and Phen, benzonitrile was not formed (**Table-1, entries 3 and 4**). This thus reveals the importance of catalysts and additives to drive the reaction. Upon elevating the temperature to

100 °C, there was a notable reduction in the yield. (**Table-1, entry 5**). Moreover, upon reducing the temperature to 60 °C, there was no observable alteration in the yield (**Table-1, entry 6**). Additionally, when the temperature was lowered to 40 °C, the yield diminished to 61 % (Table **1, entry 7**).Conversely, the application of various solvents, including DMF, DMSO, H_2O, and MeOH, resulted in the non-detection of compound 3a (**Table-1, entries 8-11**). In contrast, the utilization of toluene, DCM, and DCE yielded compound 3a in 30%, 65%, and 70% yields, respectively. (**Table-1, entries 12-14**). Thus, this reaction was most efficient when the molar ratio of aldeyhyde: tosylhydrazine: β-nitrostyrene: Phen: CuI: Lithium tertiary butoxide was 1:1:1:0.2:0.2:3 in CH_3CN at 60 ˚C for 1 h.

Table 1: Reaction Conditions Optimization:

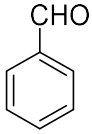
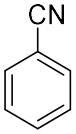
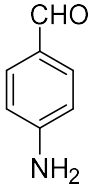
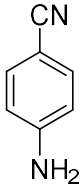
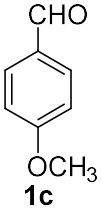
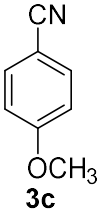
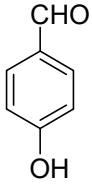
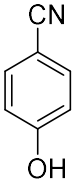


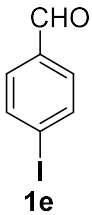
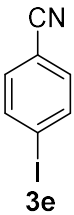
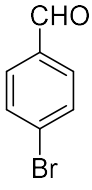
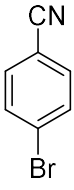
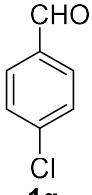
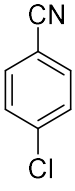
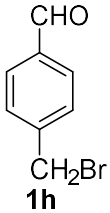
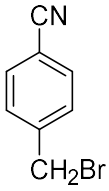
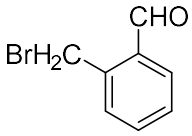
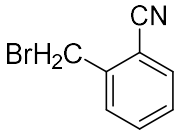
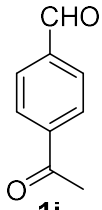
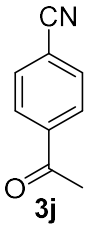
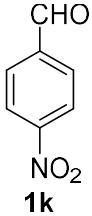
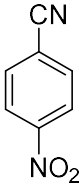
Entry[a]	Catalyst	Ligand	Additives	solvent	Yield (%)[b]
1	CuI	Phen	β-nitrostyrene	CH_3CN	95
2	CuI	Phen	------	CH_3CN	n.d
3	---	Phen	β-nitrostyrene	CH_3CN	81
4	CuI	--	β-nitrostyrene	CH_3CN	93
5	CuI	Phen	β-nitrostyrene	CH_3CN	81
6	**CuI**	**Phen**	**β-nitrostyrene**	**CH_3CN**	**93**
7	CuI	Phen	β-nitrostyrene	CH_3CN	61
8	CuI	Phen	β-nitrostyrene	DMF	n. d.
9	CuI	Phen	β-nitrostyrene	DMSO	n. d.
10	CuI	Phen	β-nitrostyrene	H_2O	n. d.
11	CuI	Phen	β-nitrostyrene	MeOH	n. d.
12	CuI	Phen	β-nitrostyrene	toluene	30
13	CuI	Phen	β-nitrostyrene	DCM	65
14	CuI	Phen	β-nitrostyrene	DCE	70

[a]Conditions of reaction: **1** (1mmol),**2** (1mmol),β-nitrostyrene(1mmol),Phen (0.2 mmol),CuI (0.2 mmol),lithium tertiary butoxide (3mmol) and solvent (2 mL) for 1 h at 80 °C. [b]Isolated yield. n. d. = not detected

Under the aforementioned optimized conditions **1a** (1mmol), **2** (1equiv), β-nitrostyrene (1mmol), Phen (0.2 mmol), CuI (0.2 mmol), lithium tertiary butoxide (3mmol) and CH_3CN (2 mL) for 1 hour at 60 °C as outlined in **(Table-1, entry 6),** to evaluate the breadth and compatibility of the method, the conversion of benzenecarbaldehyde to benzaldoxime followed by subsequent dehydration was executed utilizing various benzaldehyde substrates (**1a–1k**) in conjunction with tosylhydrazine(**2**). To our satisfaction, the transformation of benzenecarbaldehyde to benzaldoxime followed by dehydration demonstrated a commendable substrate versatility and yielded benzonitrile derivatives (**3a–3k**) in yields spanning from fair to fine as indicated in **Table-2**. The methodology proved to be compatible with a diverse array of functional groups, encompassing both simple and electron-donating substituents on the aromatic ring of the aldehyde substrates (**1a–1k**). Furthermore, the reaction exhibited compatibility with hydrazine derivatives containing simple hydrogen, phenyl, and acetyl groups. Importantly, the conversion of benzenecarbaldehyde to benzaldoxime followed by dehydration proceeded efficiently with substrate **1**, producing **3** in yields of 75 to 89% as detailed in **Table-2**.

Table 2: Domino synthesis of benzonitriles.

Entry[a]	*Benzaldehyde*(**1**)	*hydrazine* (**2**)	*Product* (**3**)	*Yields*[b]
1	 **1a**	NH_2NHTs **2a**	 **3a**	85
2	 **1b**	NH_2NHTs **2a**	 **3b**	87
3	 **1c**	NH_2NHTs **2a**	 **3c**	89
4	 **1d**	NH_2NHTs **2a**	 **3d**	90

5	 **1e**	NH_2NHTs **2a**	 **3e**	86
6	 **1f**	NH_2NHTs **2a**	 **3f**	85
7	 **1g**	NH_2NHTs **2a**	 **3g**	82
8	 **1h**	NH_2NHTs **2a**	 **3h**	87
9	 **1i**	NH_2NHTs **2a**	 **3i**	85
10	 **1j**	NH_2NHTs **2a**	 **3j**	81
11	 **1k**	NH_2NHTs **2a**	 **3k**	75

[a]Conditions of reaction: **1** (1mmol),**2** (1 mmol),β-nitrostyrene(3mmol),Phen (0.2 mmol),CuI (0.2 mmol),lithium tertiary butoxide (3mmol) and solvent (2 mL) for 1 h at 60 °C. [b]Isolated yield.

Conclusions:

In conclusion, we have successfully devised a straightforward, pragmatic, and efficient domino synthesis of benzonitriles. Notably, this approach demonstrated efficacy with the utilization of β-nitrostyrene in conjunction with phen (20 mol %) and CuI (20 mol %).

Experimental Section:-

General:

Infrared (IR) spectrum was obtained through the utilization of a Bruker Tensor 37 Fourier Transform Infrared (FTIR) spectrophotometer. (^{1}H NMR) spectra were recorded using a 'Bruker Avance 400 spectrometer' functioning at a frequency of 400MHz and a temperature of 295K in deuterated chloroform ($CDCl_3$), the coupling constants [Hz] and chemical shifts (δ, ppm) are provided in accordance with established conventions, utilizing the internal standard tetramethylsilane for reference.

Materials and methods.

The solvents were dehydrated utilizing established methodologies as deemed appropriate. ^{1}H NMR and ^{13}C NMR spectra [^{1}H, 400MHz,^{13}C, 100MHz] were obtained employing a 400MHz spectrometer using $CDCl_3$, with chemical shifts calibrated against $SiMe_4$ (δ = 0 ppm). Infrared spectra were acquired utilizing an FT-IR spectrophotometer. ESI–MS (Micromass VG Autospec) and high-resolution mass spectrometry (HRMS) with an ESI-TOF analyzer were used for mass spectrometric analyses. The organic extracts were desiccated over anhydrous sodium sulfate (Na_2SO_4). Elemental analysis of CHN was executed with an Elementar Vario Microcure Analyzer, yielding results that correlated favorably with theoretical values. Column chromatography was conducted on (100-200 mesh)silica gel employing a mixture of hexaneand ethyl acetate (EtOAc).

General procedures for benzonitrilessynthesis (3a)

The reaction commenced upon the addition of benzaldehyde (0.106g, 1mmol), tosyl hydrazine (0.186g, 1 mmol), β-nitrostyrene (0.447g, 3mmol), copper iodide (0.038g, 0.2 mmol), lithium tert-butoxide (0.241g, 3mmol) and 1,10-phenanthroline (0.036g, 0.2 mmol) into acetonitrile (2 mL) within a 25 mL RB flask. The resultant amalgamation was agitated at a temperature of 60 °C for an interval of one hour and subsequently allowed to attain room temperature conditions. Ethyl acetate (3x25 mL) was employed for the extraction of the composite mixture. The consolidated ethyl acetate extract was subsequently washed with brine (75 mL), followed by drying over anhydrous sodium sulfate and subsequent filtration. The solvent was eliminated utilizing diminished pressure conditions, and the crude product obtained was subjected to purification via silica gel chromatography utilizing a solvent system composed of hexane and ethyl acetate in a ratio of 80:20 to obtain compound **3**. Detailed information regarding the yields of all synthesized compounds is provided in **Table-2**.

4-Amino benzonitrile (**3b**)

IR(KBr, cm^{-1}): 3283, 2229, 1602, 833; ^{1}H-NMR; (400MHz $CDCl_3$):δ 7.37 (dd, *J* = 8.2 Hz and 2.1 Hz 2H), 6.73 (d, *J* = 8.7 Hz, 2H), 4.44 (s,2H);^{13}C-NMR (100MHz in $CDCl_3$): δ 149.70, 134.05, 132.37,

117.98, 117.71, 115.21, 95.96;ESI MS: m/z 119 [$(M+H)^+$];Analytical calculated for $C_7H_6N_2$: C, 71.16; H, 5.13. N; 23.72, Found: C, 71.05; H, 5. 18, N; 23.70 .

4-Methoxy benzonitrile (**3c**)

IR(KBr, cm^{-1}): 3351, 2227, 1683, 829; ^{1}H-NMR(400MHz, $CDCl_3$):δ 7.60 (d,*J* = 8.2 Hz, 2H), 6.94 (d, *J* = 8.1 Hz, 2H), 3.85 (s, 3H);^{13}C-NMR(100MHz, $CDCl_3$): δ 162.86, 134.01, 119.24, 114.76, 103.98, 55.55;ESI MS: m/z 134 [$(M+H)^+$];Analytical calculated for C_8H_7NO: C, 72.18; H, 5.31. N; 10.51, Found: C, 72.04; H, 5. 36, N; 10.52 .

4-Hydroxy benzonitrile (**3d**)

IR(KBr, cm^{-1}): 3311, 3210, 2211, 1620, 825; ^{1}H-NMR(400 MHz, Chloroform):δ 7.48 – 7.47 (m, 2H), 6.95 – 6.94 (m, 1H), 6.93 – 6.92 (m, 2H);^{13}C-NMR (100MHz, $CDCl_3$): δ 161.05, 133.02, 133.06, 119.15, 116.79, 116.68, 103.74; ESI MS: m/z 119.17 [$(M+H)^+$]; Analytical calculated for C_7H_5NO: C, 70.58; H, 4.23. N; 11.76, Found: C, 70.46; H, 4. 29, N; 11.75 .

4-Iodo benzonitrile (**3e**)

IR(KBr, cm^{-1}): 3351, 2227, 1683, 829 cm^{-1}**;**^{1}H-NMR(400MHz, $CDCl_3$):δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.2 Hz, 2H); ^{13}C-NMR(100MHz, $CDCl_3$): δ 138.52, 133.16, 118.19, 111.76, 100.27; ESI MS: m/z 229.02 [$(M)^+$]; Analytical calculated for C_7H_4IN: C, 36.73; H, 1.75. N; 6.14, Found: C, 36.58; H, 1. 82, N; 6.10 .

4-Bromo benzonitrile (**3f**)

IR(KBr, cm^{-1}):3351, 2222, 1633, 829;^{1}H-NMR**;**(400MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.5 Hz, 2H), 7.78 (d, *J* = 8.6 Hz, 2H);^{13}C-NMR(100MHz, $CDCl_3$): δ 133.41, 132.65, 128.01, 124.29, 118.04, 111.25; ESI MS: m/z 182.02 [$(M)^+$]; Analytical calculated for C_7H_4BrN: C, 46.20; H, 2.21. N; 7.60, Found: C, 46.08; H, 2. 29, N; 7.68

4-Chloro benzonitrile (**3g**)

IR(KBr, cm^{-1}): 3350, 2222, 1680, 820;^{1}H-NMR(400MHz, $CDCl_3$):δ 7.62 (d, *J* = 7.5 Hz, 2H), 7.47 (d, *J* = 7.6 Hz, 2H);^{13}C-NMR(100MHz, $CDCl_3$): δ 139.55, 133.42, 129.61, 117.98, 110.81; ESI MS: m/z 137.57 [$(M)^+$]; Analytical calculated for C_7H_4ClN: C, 61.13; H, 2.92. N; 10.17, Found: C, 61.00; H, 2. 99, N; 10.17 .

4-Bromomethyl benzonitrile (**3h**)

IR(KBr, cm^{-1}): 3263, 2225, 1610, 820; ^{1}H-NMR(400MHz, $CDCl_3$):δ 7.64 (d, *J* = 7.7 Hz, 2H), 7.50 (d, *J* = 7.9 Hz, 2H), 4.48 (s, 2H);^{13}C-NMR(100MHz, $CDCl_3$); δ 142.8, 132.61, 129.75, 118.30, 112.2, 31.56; ESI MS: m/z 194.96 [$(M)^+$]; Analytical calculated for C_8H_6BrN: C, 49.01; H, 3.08. N; 7.14, Found: C, 48.89; H, 3. 14, N; 7.13 .

2-Bromomethyl benzonitrile (**3i**)

IR(KBr, cm^{-1}): 3351, 2227, 1633, 83;^{1}H-NMR(400MHz, $CDCl_3$):δ 7.67 (d, *J* = 7.7 Hz, 1H), 7.63 – 7.53 (m, 2H), 7.42 (t, *J* = 7.5 Hz, 1H), 4.64 (s, 3H);^{13}C-NMR(100MHz, $CDCl_3$): δ 141.15, 133.31, 133.22, 130.51, 128.99, 116.79, 112.47, 29.37; ESI MS: m/z 194.96 [$(M)^+$]; Analytical calculated for C_8H_6BrN: C, 49.01; H, 3.08. N; 7.14, Found: C, 48.89; H, 3. 14, N; 7.13 .

4-Acetyl benzonitrile (**3j**)

IR(KBr, cm^{-1}): 3253, 2229, 1683, 1602, 829;^{1}H-NMR(400MHz, $CDCl_3$):δ 8.06 (d, *J* = 8.50 Hz, 2H), 7.78 (d, *J* = 8.5 Hz, 2H), 2.67 (s, 3H);^{13}C-NMR(100MHz, $CDCl_3$): δ 196.54, 139.94, 132.53, 128.71, 117.93, 116.50, 26.77; ESI MS: m/z 145.07 [$(M)^+$]; Analytical calculated for C_9H_7NO: C, 74.47; H, 4.86. N; 9.66, Found: C, 74.35; H, 4. 92, N; 9.65 .

2-Nitro benzonitrile (**3k**)

IR(KBr, cm^{-1}): 3084, 2228, 1654, 831;^{1}H-NMR(400MHz, $CDCl_3$):δ 7.26 (s, 2H), 7.26 (s, 2H).^{13}C-NMR(101MHz, $CDCl_3$): δ 169.61, 140.83; ESI MS: m/z 148.12 [$(M)^+$]; Analytical calculated for $C_7H_4N_2O_2$: C, 56.76; H, 2.72. N; 18.91, Found: C, 56.64; H, 2. 78, N; 18.88

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