Cyanoaroylation of imines bearing a thiazole ring using potassium hexacyanoferrate(II) as an eco-friendly cyanide source

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The efficient and environmentally friendly cyanoaroylation of imines bearing a thiazole ring via a one-pot three-component condensation of *N*-arylidenethiazol-2-amines, aroyl chlorides and potassium hexacyanoferrate(II) is described. This protocol has the advantages of using an eco-friendly cyanide source, a simple work-up procedure and affords high yields of products.

Keywords: green chemistry, *N*-aroylaminonitrile, multicomponent reaction, potassium hexacyanoferrate(II)

α-Aminonitriles are important intermediates for the synthesis of a wide variety of amino acids, amides, diamines, and nitrogen-containing heterocycles.[1–5] Therefore, they have considerable applications in the organic chemical industry which include, medicines, pesticides, dyestuffs, pigments, liquid crystals and polymer materials. The most general synthetic method for α-aminonitriles is the cyanation of imines via the Strecker reaction. At present, many reagents are employed for the cyanation of imines and include HCN,[6,7] KCN,[8] TMSCN,[9–12] $(EtO)_2P(O)CN$,[13] Et_2AlCN,[14] Bu_3SnCN,[15,16] MeCOCN,[17] acetone cyanohydrin,[18] and ethyl cyanoformate.[19,20] However, these cyanating agents are strongly toxic chemicals which are unsafe and environmentally unfriendly.

Potassium hexacyanoferrate(II), $K_4[Fe(CN)_6]$, is non-toxic and is even used in the food industry for metal precipitation. In addition, it has been described as an anti-agglutinating auxiliary for table salt (NaCl). $K_4[Fe(CN)_6]$ is a by-product from the coal chemical industry and commercially available on a ton scale, and is even cheaper than KCN. Recently, $K_4[Fe(CN)_6]$ has been used as a cyanide source for some substitution reactions to synthesise benzonitriles,[21–38] aroyl cyanides,[39] benzyl cyanides,[40–41] and cinnamonitriles.[42] Our current research interests have focused on the cyanation of unsaturated compounds including C=O, C=N and/or C=C functions by nucleophilic addition reactions using $K_4[Fe(CN)_6]$ as an eco-friendly cyanide source.[43–47] As an extension of this research we now report the cyanoaroylation of *N*-arylidenethiazol-2-amines via a one-pot three-component procedure using $K_4[Fe(CN)_6]$ as a nucleophilic reagent.

Results and discussion

In our previous work[48] to investigate one-pot three-component reactions of carbonyl compounds, amines, and potassium hexacyanoferrate(II), in the presence of benzoyl chloride as a promoter, the imine produced *in situ* afforded hydrocyanation products. As an extension of this work to investigate the direct cyanation of imines bearing a thiazole ring under the similar conditions, quite different cyanoaroylation products were isolated. Therefore, in order to confirm these observations the cyanation of imines, containing a thiazole moiety, with $K_4[Fe(CN)_6]$ was studied in more detail.

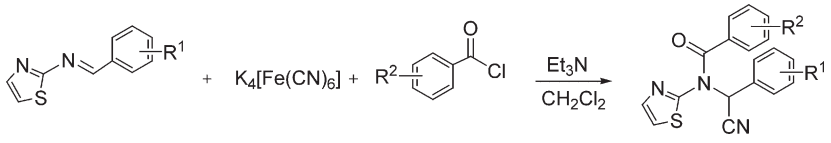
The reactions were first conducted using *N*-benzylidenethiazol-2-amine and benzoyl chloride as substrates (Scheme 1, $R^1 = R^2 = H$). It was found that the catalyst played a crucial role in the one-pot three-component reaction. No cyanobenzoylation products were observed in the absence of a catalyst. Some bases such as pyridine, hydroxylamine, sodium hydroxide, piperazine hexahydrate, 4-dimethylaminopyridine (DMAP), and 1,4-diazabicyclo[2.2.2]octane (DABCO) were employed but in each case a very low yield of product was obtained. However, it was found that the reaction gave the desired product in high yield when triethylamine was employed as a catalyst.

Solvents also had a dramatic effect on the one-pot three-component reaction of *N*-benzylidenethiazol-2-amine, benzoyl chloride and $K_4[Fe(CN)_6]$ under triethylamine catalysis. It was found that some solvents such as Et_2O, THF and PhMe suppressed the reaction. However, when *n*-hexane, MeOH, EtOH and CH_2Cl_2 were employed the desired product was obtained in moderate to high yield. In particular the reaction in CH_2Cl_2 afforded the product in highest yield (Table 1).

In the light of these findings, a variety of substituted aroyl chlorides and *N*-arylidenethiazol-2-amines were examined for the cyanoaroylation reactions using $K_4[Fe(CN)_6]$ under triethylamine catalysis (Scheme 1 and Table 2). Aroyl chlorides and *N*-arylidenethiazol-2-amines bearing electron-donating or electron-withdrawing groups readily participated in the reactions to afford the desired products in high yield. The substituents on the aryl rings have no obvious effect on the yields of products.

A plausible mechanism for the cyanoaroylation reaction is shown in Scheme 2. Thus $K_4[Fe(CN)_6]$ first reacts with the aroyl chlorides to form an aroyl cyanide, which then combines with triethylamine to give intermediate **A**. The latter reacts further with *N*-arylidenethiazol-2-amines to generate intermediate **B** which finally loses triethylamine to yield the cyanoaroylation products.

In summary, an efficient and environmentally friendly method for the cyanoaroylation of imines bearing a thiazole ring through the one-pot three-component condensation of *N*-arylidenethiazol-2-amines, $K_4[Fe(CN)_6]$ and aroyl chlorides using triethylamine as a catalyst in CH_2Cl_2 has developed. The significant features of this protocol are the use of non-toxic, inexpensive $K_4[Fe(CN)_6]$ as a cyanide source to replace the traditional strongly toxic and volatile cyanating agents. The simplicity of reaction and work-up procedure, the high yield, and avoiding the use of heavy metal catalysts are noteworthy.



Scheme 1 Cyanoaroylation of imines bearing thiazole ring.

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Table 1 The effect of solvents on the yield of cyanobenzoylation product[a]

Entry	Solvent	Reaction time/h	Yield/%[b]
1	Et_2O	8	0
2	THF	8	0
3	PhMe	8	0
4	*n*-hexane	4	10
5	MeOH	4	52
6	EtOH	4	63
7	CH_2Cl_2	4	88

[a]Reaction conditions: *N*-benzylidenethiazol-2-amine (2 mmol), benzoyl chloride (2 mmol), $K_4[Fe(CN)_6]$ (0.4 mmol) and triethylamine (0.2 mmol) in 5 mL of solvent for 8 h.
[b]Isolated yield.

Table 2 Synthesis of *N*-aroylaminonitriles using $K_4[Fe(CN)_6]$ as a cyanide source[a]

Entry	R^1	R^2	Yield/%[b]	M.p./°C
1	H	H	88	133–135
2	4-CH_3	H	75	96–98
3	3,4-$(CH_3)_2$	H	81	94–96
4	4-Cl	H	70	113–115
5	H	4-CH_3	78	112–114
6	4-CH_3	4-CH_3	82	135–137
7	3,4-$(CH_3)_2$	4-CH_3	88	103–105
8	H	3-CH_3	79	96–98
9	4-CH_3	3-CH_3	84	74–76
10	3,4-$(CH_3)_2$	3-CH_3	89	103–105
11	3,4-$(CH_3)_2$	4-F	90	113–115
12	H	4-Cl	79	125–126
13	4-CH_3	4-Cl	84	152–154
14	3,4-$(CH_3)_2$	4-Cl	89	146–148

[a]Reaction condition: *N*-arylidenethiazol-2-amine (2 mmol), aroyl chloride (2 mmol), $K_4[Fe(CN)_6]$ (0.4 mmol) and triethylamine (0.2 mmol) in 5 mL of CH_2Cl_2 for 8 h.
[b]Isolated yield.

Experimental

IR spectra were recorded using KBr pellets on an Alpha Centauri FTIR spectrophotometer (Shimadzu, Japan). ^{1}H NMR and ^{13}C NMR spectra were recorded with a Mercury-400BB instrument (Varian, USA) using $CDCl_3$ as solvent and Me_4Si as internal standard. Melting points were observed using an electrothermal melting point apparatus. Potassium hexacyanoferrate(II) was dried at 80 °C under vacuum for 24 h and finely powdered prior to use.

Cyanoaroylation of imines bearing a thiazole ring using $K_4[Fe(CN)_6]$ as a cyanide source; general procedure

A mixture of $K_4[Fe(CN)_6]$ (0.4 mmol) and aroyl chloride (2 mmol) was heated at 160 °C for 4 h. Then the reaction system was cooled to room temperature and triethylamine (0.2 mmol) and the *N*-arylidene-thiazol-2-amine (2 mmol) in CH_2Cl_2 (5 mL) were added. The resulting mixture was stirred at room temperature for 4 h. After completion of the reaction (TLC), the mixture was filtered to remove the solids, and the filtrate was concentrated and isolated by column chromatography using ethyl acetate and petroleum ether (b.p. 60–90 °C) (1:10) as eluent to give pure products. Analytical data for the products (Table 2) are given below.

N-*[Cyano(phenyl)methyl]*-N-*(thiazol-2-yl)benzamide* (entry 1): White solid; m.p. 133–135 °C; IR (KBr, ν_{max}/cm^{-1}): 2244 (CN), 1670 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.48–6.99 (m, 12H, ArH and ThH), 6.98 (s, 1H, CH); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.6, 159.0, 139.1, 133.1, 131.7, 131.6, 129.3, 128.9, 128.7, 128.2, 127.3, 118.9, 115.9, 52.6. Anal. Calcd for $C_{18}H_{13}N_3OS$: C, 67.69; H, 4.10; N, 13.16. Found: C, 67.55; H, 4.09; N, 13.11%.

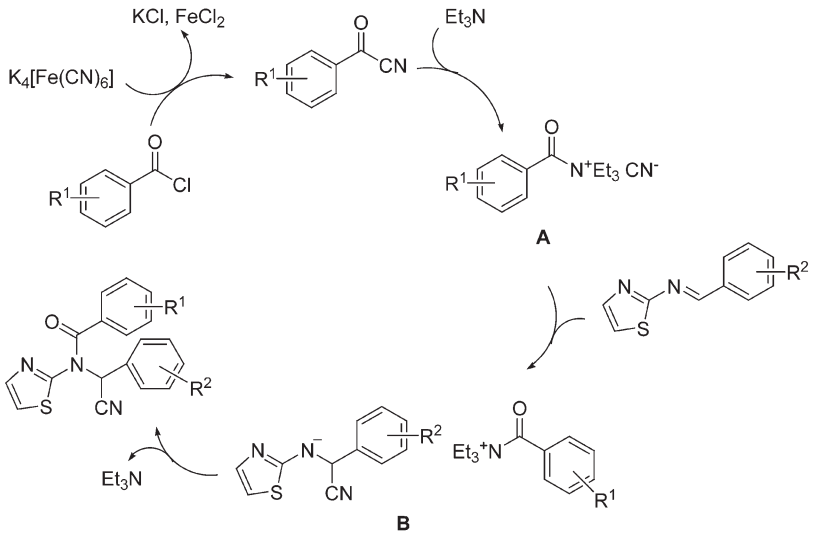
N-*[Cyano(4-tolyl)methyl]*-N-*(thiazol-2-yl)benzamide* (entry 2): White solid; m.p. 96–98 °C; IR (KBr, ν_{max}/cm^{-1}): 2247 (CN), 1671 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46–6.97 (m, 11H, ArH and ThH), 6.94 (s, 1H, CH), 2.25 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.6, 159.0, 139.3, 139.2, 133.2, 131.6, 129.6, 128.6, 128.2, 127.3, 119.0, 116.0, 52.3, 21.1. Anal. Calcd for $C_{19}H_{15}N_3OS$: C, 68.45; H, 4.53; N, 12.60. Found: C, 68.31; H, 4.55; N, 12.56%.

N-*[Cyano(3,4-dimethylphenyl)methyl]*-N-*(thiazol-2-yl)benzamide* (entry 3): White solid; m.p. 94–96 °C; IR (KBr, ν_{max}/cm^{-1}): 2249 (CN), 1671 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46–6.98 (m, 10H, ArH and ThH), 6.92 (s, 1H, CH), 2.15 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.6, 159.1, 139.1, 138.0, 137.4, 133.3, 131.5, 130.1, 128.9, 128.6, 128.4, 128.2, 124.6, 118.9, 116.1, 52.3, 19.7, 19.5. Anal. Calcd for $C_{20}H_{17}N_3OS$: C, 69.14; H, 4.93; N, 12.09. Found: C, 69.23; H, 4.95; N, 12.12%.

N-*[Cyano(4-chlorophenyl)methyl]*-N-*(thiazol-2-yl)benzamide* (entry 4): White solid; m.p. 113–115 °C; IR (KBr, ν_{max}/cm^{-1}): 2245 (CN), 1666 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.54–7.06 (m, 11H, ArH and ThH), 6.99 (s, 1H, CH); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.5, 158.9, 139.2, 135.4, 132.8, 131.8, 130.3, 129.1, 128.8, 128.7, 128.2, 119.1, 115.5, 52.0. Anal. Calcd for $C_{18}H_{12}ClN_3OS$: C, 61.10; H, 3.42; N, 11.88. Found: C, 61.18; H, 3.43; N, 11.92%.

N-*[Cyano(phenyl)methyl]*-N-*(thiazol-2-yl)-4-methylbenzamide* (entry 5): White solid; m.p. 112–114 °C; IR (KBr, ν_{max}/cm^{-1}): 2246 (CN), 1667 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50–7.04 (m, 11H, PhH and ThH), 7.03 (s, 1H, CH), 2.35 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.7, 159.2, 142.4, 139.0, 131.7, 130.1, 129.3, 129.2, 128.9, 128.4, 127.2, 118.7, 115.9, 52.6, 21.5. Anal. Calcd for $C_{19}H_{15}N_3OS$: C, 68.45; H, 4.53; N, 12.60. Found: C, 68.31; H, 4.54; N, 12.64%.

N-*[Cyano(4-tolyl)methyl]*-N-*(thiazol-2-yl)-4-methylbenzamide* (entry 6): White solid; m.p. 135–137 °C; IR (KBr, ν_{max}/cm^{-1}): 2245



Scheme 2 The proposed mechanism for the cyanoaroylation of imines bearing a thiazole ring using $K_4[Fe(CN)_6]$ as a cyanide source.

(CN), 1674 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50–7.03 (m, 10H, PhH and ThH), 7.00 (s, 1H, CH), 2.35 (s, 3H, CH_3), 2.32 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.7, 159.2, 142.3, 139.2, 139.1, 130.2, 129.5, 129.3, 128.7, 128.4, 127.2, 118.7, 116.1, 52.4, 21.5, 21.1. Anal. Calcd for $C_{20}H_{17}N_3OS$: C, 69.14; H, 4.93; N, 12.09. Found: C, 69.01; H, 4.91; N, 12.05%.

N-*[Cyano(3,4-dimethyl)methyl]*-N-*(thiazol-2-yl)-4-methylbenzamide* (entry 7): White solid; m.p. 103–105 °C; IR (KBr, ν_{max}/cm^{-1}): 2245 (CN), 1670 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.51–7.04 (m, 9H, PhH and ThH), 6.98 (s, 1H, CH), 2.35 (s, 3H, CH_3), 2.22 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.7, 159.3, 142.3, 139.1, 137.9, 137.3, 130.3, 130.0, 129.3, 129.0, 128.4, 128.3, 124.6, 118.7, 116.2, 52.4, 21.5, 19.7, 19.5. Anal. Calcd for $C_{21}H_{19}N_3OS$: C, 69.78; H, 5.30; N, 11.63. Found: C, 69.89; H, 5.31; N, 11.68%.

N-*[Cyano(phenyl)methyl]*-N-*(thiazol-2-yl)-3-methylbenzamide* (entry 8): White solid; m.p. 96–98 °C; IR (KBr, ν_{max}/cm^{-1}): 2250 (CN), 1671 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42–6.97 (m, 11H, PhH and ThH) 6.96 (s, 1H, CH), 2.25 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.8, 158.9, 138.9, 138.8, 133.0, 132.5, 131.7, 129.2, 128.9, 128.7, 128.5, 127.2, 125.1, 118.5, 115.9, 52.6, 21.2. Anal. Calcd for $C_{19}H_{15}N_3OS$: C, 68.45; H, 4.53; N, 12.60. Found: C, 68.49; H, 4.52; N, 12.57%.

N-*[Cyano(4-tolyl)methyl]*-N-*(thiazol-2-yl)-3-methylbenzamide* (entry 9): White solid; m.p. 74–76 °C; IR (KBr, ν_{max}/cm^{-1}): 2249 (CN), 1671 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42–6.96 (m, 10H, PhH and ThH), 6.93 (s, 1H, CH), 2.24 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.8, 158.9, 139.2, 138.9, 138.7, 133.1, 132.4, 129.5, 128.7, 128.6, 128.4, 127.1, 125.0, 118.6, 116.0, 52.3, 21.2, 21.1. Anal. Calcd for $C_{20}H_{17}N_3OS$: C, 69.14; H, 4.93; N, 12.09. Found: C, 69.22; H, 4.92; N, 12.12%.

N-*[Cyano(3,4-dimethylphenyl)methyl]*-N-*(thiazol-2-yl)-3-methylbenzamide* (entry 10): White solid; m.p. 103–105 °C; IR (KBr, ν_{max}/cm^{-1}): 2249 (CN), 1672 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42–6.96 (m, 9H, PhH and ThH), 6.92 (s, 1H, CH), 2.23 (s, 3H, CH_3), 2.14 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 169.8, 159.3, 138.9, 138.6, 137.8, 137.3, 133.1, 132.3, 130.0, 128.9, 128.7, 128.4, 128.3, 125.0, 124.5, 118.5, 116.1, 52.3, 21.2, 19.7, 19.4. Anal. Calcd for $C_{21}H_{19}N_3OS$: C, 69.78; H, 5.30; N, 11.63. Found: C, 69.69; H, 5.32; N, 11.58%.

N-*[Cyano(3,4-dimethylphenyl)methyl]*-N-*(thiazol-2-yl)-4-fluorobenzamide* (entry 11): White solid; m.p. 113–115 °C; IR (KBr, ν_{max}/cm^{-1}): 2249 (CN), 1674 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.53–7.00 (m, 9H, PhH and ThH), 6.97 (s, 1H, CH), 2.23 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 168.6, 165.6, 163.1, 159.4, 139.5, 138.1, 137.4, 130.9, 130.8, 130.1, 129.5, 128.6, 128.5, 124.8, 119.7, 116.0, 115.8, 115.6, 52.0, 19.7, 19.5. Anal. Calcd for $C_{20}H_{16}FN_3OS$: C, 65.74; H, 4.41; N, 11.50. Found: C, 65.67; H, 4.40; N, 11.47%.

N-*[Cyano(phenyl)methyl]*-N-*(thiazol-2-yl)-4-chlorobenzamide* (entry 12): White solid; m.p. 125–126 °C; IR (KBr, ν_{max}/cm^{-1}): 2242 (CN), 1678 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.45–7.03 (m, 11H, PhH and ThH), 7.02 (s, 1H, CH); ^{13}C NMR ($CDCl_3$, 100 MHz): 168.6, 159.2, 139.6, 138.0, 131.6, 131.4, 129.8, 129.5, 129.0, 128.8, 127.5, 119.8, 115.7, 52.2. Anal. Calcd for $C_{18}H_{12}ClN_3OS$: C, 61.10; H, 3.42; N, 11.88. Found: C, 61.04; H, 3.41; N, 11.85%.

N-*[Cyano(4-tolyl)methyl]*-N-*(thiazol-2-yl)-4-chlorobenzamide* (entry 13): White solid; m.p. 152–154 °C; IR (KBr, ν_{max}/cm^{-1}): 2240 (CN), 1682 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46–7.02 (m, 10H, PhH and ThH), 6.99 (s, 1H, CH), 2.27 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 168.6, 159.2, 139.7, 139.6, 137.9, 131.7, 129.9, 129.7, 128.8, 128.4, 127.5, 119.9, 115.9, 52.0, 21.2. Anal. Calcd for $C_{19}H_{14}ClN_3OS$: C, 62.04; H, 3.84; N, 11.42. Found: C, 62.14; H, 3.83; N, 11.38%.

N-*[Cyano(3,4-dimethylphenyl)methyl]*-N-*(thiazol-2-yl)-4-chlorobenzamide* (entry 14): White solid; m.p. 146–148 °C; IR (KBr, ν_{max}/cm^{-1}): 2239 (CN), 1678 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46–7.02 (m, 9H, PhH and ThH), 6.97 (s, 1H, CH), 2.16 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 168.6, 159.3, 139.6, 138.2, 137.8, 137.5, 131.8, 130.2, 129.8, 128.8, 128.6, 128.6, 124.9, 119.8, 116.0, 52.0, 19.8, 19.5. Anal. Calcd for $C_{20}H_{16}ClN_3OS$: C, 62.90; H, 4.22; N, 11.00. Found: C, 62.79; H, 4.23; N, 12.97%.

Electronic Supplementary Information

Copies of the IR, ^{1}H and ^{13}C NMR spectra of all the compounds above are provided in the electronic supplementary information available through stl.publisher.ingentaconnect.com/content/stl/jcr/supp-data.

The authors thank the National Natural Science Foundation of China (21162024) and Provincial Natural Science Foundation of Gansu (1107RJZA189) for financial support of this work.

Received 12 September 2012; accepted 14 October 2012
Paper 1201510 doi: 10.3184/174751912X13518642070088
Published online: 5 December 2012

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