Res Chem Intermed
DOI 10.1007/s11164-013-1421-8

Direct synthesis of cyanohydrin esters from aroyl chlorides using potassium hexacyanoferrate(II) as an eco-friendly cyanide source

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Received: 29 March 2013 / Accepted: 21 September 2013
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Abstract A direct synthetic method for cyanohydrin esters from aroyl chlorides using potassium hexacyanoferrate(II) as an eco-friendly cyanide source and tributylphosphine as a promoter is described. This protocol has advantages of no use of strong toxic cyanating agents, high yield, and simple work-up procedure.

Keywords Cyanation · Cyanohydrin ester · Eco-friendly cyanide source · Potassium hexacyanoferrate(II) · Green chemistry

Introduction

Cyanohydrin esters are versatile building blocks, which can be utilized for the synthesis of a wide range of fine chemicals, pharmaceuticals, and agrochemicals, and they are therefore an important subject of research [1–6]. The general synthetic methods for cyanohydrin esters include: (1) the reactions of aldehydes with aroyl cyanides catalyzed by bases [7–10]; (2) the reactions of aldehydes with trimethylsilyl cyanide (or potassium cyanide, lithium cyanide) and acid anhydrides (or acid chlorides) [11–15]; (3) the reduction coupling of aroyl cyanides using sodium tetrahydroborate or trimethylphosphine [16, 17]; (4) the reactions of cyanohydrins with benzoic acid (or benzoyl chloride) [18]; (5) the reactions of *O*-trimethylsilyl cyanohydrin with aroyl halides (or acid anhydrides) [19, 20]; and (6) the reactions of 1-(benzotriazol-1-yl)alkyl esters with potassium cyanide [21]. In these synthetic methods, strong toxic cyanating agents, such as KCN, and volatile cyanating agents, such as TMSCN, have been utilized. In addition, the commercially available aroyl cyanides and cyanohydrins are limited and comparatively expensive. Especially, the

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Published online: 05 October 2013

synthesis of aroyl cyanides and cyanohydrins have also had to use strong toxic reagents as original cyanide sources, such as HgCN [22], NaCN [23, 24], CuCN [25], KCN [26, 27], and TMSCN [28, 29], which render the reactions unsafe and environmentally unfriendly. Therefore, there is a need to explore environmentally benign cyanide sources and simple procedures for the synthesis of cyanohydrin esters.

Potassium hexacyanoferrate(II), $K_4[Fe(CN)_6$, is mainly used as a carburizing agent in the iron and steel industry, and it is also 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 of the coal chemical industry and commercially available on a ton scale, and it is even cheaper than KCN. Recently, $K_4[Fe(CN)_6]$ has been used as a cyanide source for some substitution reactions to synthesize benzonitriles [30–47], aroyl cyanides [48], benzyl cyanides [49, 50], and cinnamonitriles [44]. Our current research interests focus on the cyanation of unsaturated compounds including C=O, C=N and/or C=C by nucleophilic addition reactions using $K_4[Fe(CN)_6]$ as an eco-friendly cyanide source [51–56].

Here, we report an efficient method for the direct synthesis of cyanohydrin esters from aroyl chlorides using $K_4[Fe(CN)_6]$ as an eco-friendly original cyanide source.

Experimental

IR spectra were recorded using KBr pellets on an Alpha Centauri FTIR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Mercury-400BB instrument using $CDCl_3$ as solvent and Me_4Si as internal standard. Elemental analyses were performed on a Vario E1 Elemental Analysis instrument. Melting points were observed in an electrothermal melting point apparatus. Potassium hexacyanoferrate(II) was dried at 80 °C under vacuum for 24 h and finely powdered prior to use.

General procedure for the preparation of cyanohydrin esters

The mixture of aroyl chloride (10 mmol) and potassium hexacyanoferrate(II) (0.92 g, 2.5 mmol) was heated at 160 °C under stirring for 3 h. Then, the mixture was cooled to room temperature, and tributylphosphine (2.02 g, 10 mmol) and THF (10 mL) were added. The resulting mixture was further refluxed for 1 h. Then, the solid was removed by filtration, the filtrate was evaporated off the solvent, and the residue was subjected to silica gel flash column chromatography (ethyl acetate, petroleum ether, 1:25) to obtain the pure product. The analytical and spectral data for products are given below.

*Cyano(phenyl)methyl benzoate (**1**)*

White solid. Mp 56–58 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.67 (s, 1H, CH), 7.44–7.49 (m, 6H, Ar), 7.60–7.64 (m, 2H, Ar), 8.05–8.08 (m, 2H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 63.2, 116.1, 127.7, 128.0, 128.5, 129.1, 130.0, 130.3,

131.7,134.0, 164.5; IR (KBr) ν_{max}/cm^{-1}: 3,054, 2,959, 2,244, 1,727, 1,600, 1,495, 1,452, 1,269, 1,101, 752, 712; Anal. Calcd. for $C_{15}H_{11}NO_2$: C, 75.94; H, 4.67; N, 5.90; Found: C, 75.88; H, 4.66; N, 5.89.

*Cyano(2-chlorophenyl)methyl 2-chlorobenzoate (**2**)*

White solid. Mp 50–52 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.95 (s, 1H, CH), 7.32–7.50 (m, 6H, Ar), 7.80–7.83 (m, 1H, Ar), 7.90 (d, $J = 7.6$ Hz, 1H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.0, 115.0, 126.7, 127.3, 127.6, 129.1, 129.6, 130.2, 131.4, 131.8, 132.0, 133.4, 133.7, 134.6, 163.0; IR (KBr) ν_{max}/cm^{-1}: 3,064, 2,969, 2,256, 1,740, 1,591, 1,476, 1,438, 1,311, 1,232, 1,097, 1,036, 767, 739; Anal. Calcd. for $C_{15}H_9Cl_2NO_2$: C, 58.85; H, 2.96; N, 4.58; Found: C, 58.97; H, 2.96; N, 4.60.

*Cyano (3-chlorophenyl)methyl 3-chlorobenzoate (**3**)*

Light yellow oil. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.62 (s, 1H, CH), 7.40–7.50 (m, 4H, Ar), 7.57–7.61 (m, 2H, Ar), 7.93–7.97 (m, 1H, Ar), 8.01–8.03 (m, 1H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 62.8, 115.3, 126.0, 128.0, 128.1, 129.3, 130.0, 130.1, 130.6, 130.7, 133.1, 134.2, 134.8, 135.2, 163.2; IR (KBr) ν_{max}/cm^{-1}: 3,058, 2,959, 2,247, 1,737, 1,601, 1,484, 1,376, 1,243, 1,089, 1,042, 756, 732; Anal. Calcd. for $C_{15}H_9Cl_2NO_2$: C, 58.85; H, 2.96; N, 4.58; Found: C, 58.78; H, 2.95; N, 4.59.

*Cyano (4-chlorophenyl)methyl 4-chlorobenzoate (**4**)*

Light yellow solid. Mp 64–65 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.62 (s, 1H, CH), 7.43–7.47 (m, 4H, Ar), 7.54 (d, $J = 8.4$ Hz, 2H, Ar), 7.97 (d, $J = 8.4$ Hz, 2H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 62.7, 115.5, 126.1, 129.0, 129.2, 129.4, 130.0, 131.2, 136.6, 140.7, 163.5; IR (KBr) ν_{max}/cm^{-1}: 3,076, 2,954, 2,236, 1743, 1598, 1465, 1369, 1234, 1096, 798, 773; Anal. Calcd. for $C_{15}H_9Cl_2NO_2$: C, 58.85; H, 2.96; N, 4.58; Found: C, 58.80; H, 2.97; N, 4.57.

*Cyano (3-nitrophenyl)methyl 3-nitrobenzoate (**5**)*

Yellow solid. Mp 65–67 °C.^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.25 (s, 1H, CH), 7.43–7.76 (m, 4H, Ar), 7.89–8.12 (m, 2H, Ar), 8.32–8.49 (m, 1H, Ar), 8.62–8.79 (m, 1H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.9, 118.4, 120.9, 124.2, 124.8, 125.4, 128.3, 129.3, 131.2, 134.7, 135.8, 136.2, 147.3, 148.9, 166.3; IR (KBr) ν_{max}/cm^{-1}: 3,048, 2,929, 2,242, 1,768, 1,601, 1,492, 1,363, 1,226, 1,096, 1,022, 746, 712; Anal. Calcd. for $C_{15}H_9N_3O_6$: C, 55.05; H, 2.77; N, 12.84; Found: C, 55.14; H, 2.78; N, 12.80.

*Cyano(4-nitrophenyl)methyl 4-nitrobenzoate (**6**)*

White solid. Mp 72–74 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): 6.32 (s, 1H, CH), 7.26 (d, $J = 8.4$ Hz, 2H, Ar), 8.14 (d, $J = 8.4$ Hz, 2H, Ar), 8.23–8.34 (m, 4H, Ar); ^{13}C

NMR ($CDCl_3$, 100 MHz): δ 65.2, 118.7, 121.7, 125.3, 129.1, 130.6, 136.2,139.6, 147.9, 154.6, 166.2; IR (KBr) ν_{max}/cm^{-1}: 3,039, 2,918, 2,247, 1,736, 1,601, 1,489, 1,345, 1,218, 1,122, 774, 734; Anal. Calcd. for $C_{15}H_9N_3O_6$: C, 55.05; H, 2.77; N, 12.84; Found: C, 54.98; H, 2.77; N, 12.81.

*Cyano(4-methylphenyl)methyl 4-methylbenzoate (**7**)*

White solid. Mp 112–114 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 2.41 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 6.63 (s, 1H, CH), 7.27 (d, $J = 6.4$ Hz, 4H, Ar), 7.51 (d, $J = 8.4$ Hz, 2H, Ar), 7.95 (d, $J = 8.4$ Hz, 2H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.3, 21.8, 63.1, 116.5, 125.4, 127.9, 129.0, 129.4,129.9, 130.1, 140.6, 144.9, 164.7; IR (KBr) ν_{max}/cm^{-1}: 3,069, 2,938, 2,241, 1,756, 1,611, 1,479, 1,375, 1,242, 1,102, 787, 754; Anal. Calcd. for $C_{17}H_{15}NO_2$: C, 76.96; H, 5.70; N, 5.28; Found: C, 76.84; H, 5.68; N, 5.26.

*Cyano(3,5-dimethylphenyl)methyl 3,5-dimethylbenzoate (**8**)*

Light yellow oil. ^1H NMR ($CDCl_3$, 400 MHz): δ 2.36 (s, 6H, CH_3), 2.37 (s, 6H, CH_3), 6.61 (s, 1H, CH), 7.11 (s, 1H, Ar), 7.22 (s, 2H, Ar), 7.25 (s, 1H, Ar), 7.68 (s, 2H, Ar). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.1, 21.2, 63.3, 116.5, 125.7, 127.7, 127.9, 131.8, 131.9, 135.7, 138.3, 138.9, 164.9; IR (KBr) ν_{max}/cm^{-1}: 3,010, 2,922, 2,859, 2,242, 1,727, 1,609, 1,462, 1,301, 1,241, 1,121, 792, 748; Anal. Calcd. for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77; Found: C, 77.85; H, 6.51; N, 4.75.

*Cyano(3,4,5-trimethoxyphenyl)methyl 3,4,5-trimethoxybenzoate (**9**)*

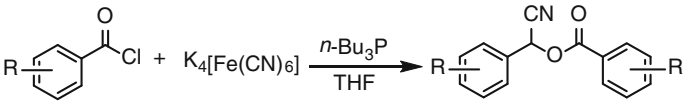
Yellow solid. Mp 136–138 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 3.87 (s, 3H, OCH_3), 3.90 (s, 6H, OCH_3), 3.91 (s, 6H, OCH_3), 3.92 (s, 3H, OCH_3), 6.62 (s, 1H, CH), 6.81 (s, 2H, Ar), 7.30 (s, 2H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 56.0, 56.1, 60.6, 60.7, 63.4, 105.0, 107.1, 116.0, 122.6, 127.0, 139.3, 143.0, 152.8, 153.5, 164.1; IR (KBr) ν_{max}/cm^{-1}: 3,044, 2,942, 2,239, 1,728, 1,597, 1,469, 1,332, 1,216, 1,143, 765, 743; Anal. Calcd. for $C_{21}H_{23}NO_8$: C, 60.43; H, 5.55; N, 3.36; Found: C, 60.35; H, 5.56; N, 3.35.

*Cyano(furan-2-yl)methyl furan-2-carboxylate (**10**)*

Yellow solid. Mp 120–122 °C. ^1H NMR ($CDCl_3$, 400 MHz): δ 6.47–6.49 (m, 1H, Fu), 6.54–6.56 (m, 1H, Fu), 6.72 (s, 1H, CH), 6.75–6.77 (m, 1H, Fu), 7.33–7.34 (m, 1H, Fu), 7.52–7.54 (m, 1H, Fu), 7.65–7.67 (m, 1H, Fu); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 55.7, 111.1, 112.2, 113.0, 116.8, 120.6, 142.4, 145.1, 146.3, 147.7, 156.2; IR (KBr) ν_{max}/cm^{-1}: 3,038, 2,923, 2,228, 1,732, 1,496, 1,354, 1,280, 1,098, 782, 743; Anal. Calcd. for $C_{11}H_7NO_4$: C, 60.83; H, 3.25; N, 6.45; Found: C, 60.89; H, 3.24; N, 6.47.

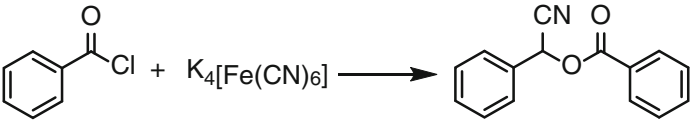
Results and discussion

Initially, the reaction of benzoyl chloride with $K_4[Fe(CN)_6]$ was selected to examine the feasibility for the synthesis of corresponding cyanohydrin ester under different conditions (Scheme 1, R=H). It was found that no corresponding product was observed in the absence of a promoter (Table 1, entry 1). In later research, some Lewis bases, such as pyridine, triethylamine, triphenylphosphine, and tributylphosphine, were selected as promoters. Pyridine and triethylamine had no effect on the reaction, and no cyanohydrin ester was produced (Table 1, entries 2–3). However, triphenylphosphine and tributylphosphine can promote the reactions in different solvents under refluxing conditions. Among them, the best yield for cyanohydrin ester was obtained when tributylphosphine was used as a promoter and THF as solvent under refluxing condition (Table 1, entry 9). In addition, the optimal mole ratio of benzoyl chloride to potassium hexacyanoferrate(II) was 4:1 for the reaction, which indicated that an excess of $K_4[Fe(CN)_6]$ (based on the number of CN^-) was needed in the reaction. In fact, the reaction may undergo a one-pot two-step



Scheme 1 Synthesis of cyanohydrin esters

Table 1 The effect of the reaction conditions on the yield of cyanohydrin ester



Entry	Promoter	Solvent	Reaction time (h)	Yield (%)[a]
1	–	THF	6	0
2	Pyridine	THF	6	0
3	Et_3N	THF	6	0
4	Ph_3P	CH_2Cl_2	3	11
5	Ph_3P	Toluene	3	36
6	Ph_3P	THF	3	40
7	*n*-Bu_3P	CH_2Cl_2	1	66
8	*n*-Bu_3P	Toluene	1	78
9	*n*-Bu_3P	THF	1	88

All reactions were carried out first using benzoyl chloride (10 mmol) and $K_4[Fe(CN)_6]$ (2.5 mmol) at 160 °C, then at refluxing condition after addition of promoter (10 mmol) and different solvents (10 mL)

[a] Isolated yields

procedure to give product: firstly, benzoyl chloride reacted with $K_4[Fe(CN)_6]$ under solvent-free condition at 160 °C to give benzoyl cyanide as an intermediate, then 2 mol of benzoyl cyanide, through reductive coupling in THF at refluxing condition in the presence of tributylphosphine, afforded cyanohydrin ester as a final product.

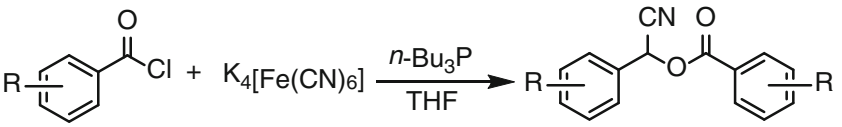
Based on the above promising findings, and to explore the generality and scope of the synthetic method, various substituted aroyl chlorides were examined for the reactions under the optimal conditions (Scheme 1). The results are summarized in Table 2. It was found that aroyl chlorides bearing electron-withdrawing substituents, such as chloro and nitro groups, on the aromatic rings gave the corresponding products in higher yield (Table 2, entries 2–6). In contrast, aroyl chlorides bearing electron-donating substituents, such as methyl and methoxy groups, on the aromatic rings afforded the corresponding products in slightly lower yield under similar conditions (Table 2, entries 7–9). In addition, for *ortho*-substituted aroyl chlorides (Table 2, entry 2), the corresponding products were obtained in slightly lower yield than *para*-substituted ones (Table 2, entry 4), presumably due to the steric hindrance (Table 2, entry 2). Heteroaroyl chloride, such as furoyl chloride, was also very efficient for the reaction (Table 2, entry 10). Aliphatic acyl chlorides, such as acetyl chloride and oxalyl chloride, and vinylic acyl chloride, such as acryloyl chloride, were also tested for the similar reactions. However, no corresponding cyanohydrin esters were observed because the high volatilities and low stabilities of acyl chlorides made the reactions with $K_4[Fe(CN)_6]$ difficult to proceed at high temperature.

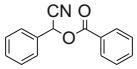
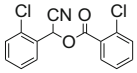
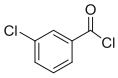
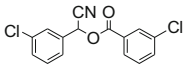
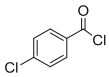
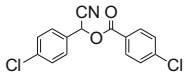
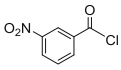
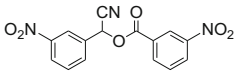
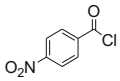
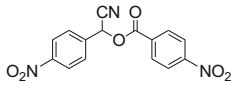
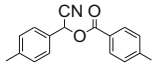
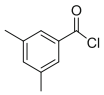
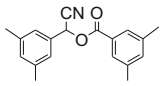
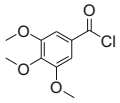
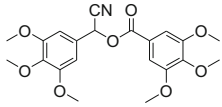
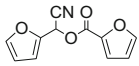
A plausible mechanism for these reactions is shown in Scheme 2. Initially, potassium hexacyanoferrate(II) reacted with aroyl chlorides to form aroyl cyanides [51], which were probably activated by 0.5 equiv. of tributylphosphine to yield intermediates **A**. Intermediates **A** were in equilibrium with the corresponding intermediates **B** [57]. Then, the nucleophilic attack of aroyl cyanides by intermediates **B** in the following step gave intermediates **C**. The subsequent one hydride transfer from tributylphosphine to the carbon connected with the CN group took place to give cyanohydrin esters by ambient moisture through the loss of tributylphosphine oxide and hydrogen cyanide. In addition, the hydrogen cyanide produced was quenched by other 0.5 equiv. of tributylphosphine during the reaction.

Conclusion

An efficient direct synthetic route from aroyl chlorides to cyanohydrin esters using potassium hexacyanoferrate(II) as an eco-friendly cyanide source has been developed. The protocol has the advantages of no use of toxic cyanating agents, high yield, and simple work-up procedure.

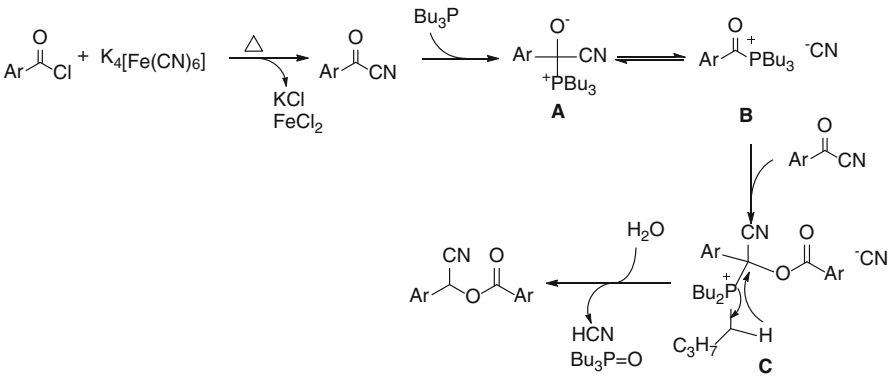
Table 2 Synthesis of cyanohydrin esters from aroyl chlorides



Entry	Aroyl chloride	Product	Yield (%)[b]
1			88
2			80
3			82
4			86
5			82
6			87
7			79
8			75
9			73
10			78

[a] All reactions were carried out first using benzoyl chloride (10 mmol) and $K_4[Fe(CN)_6]$ (2.5 mmol) at 160 °C, then at refluxing condition after addition of *n*-Bu_3P (10 mmol) and THF (10 mL)

[b] Isolated yields



Scheme 2 The mechanism for the synthesis of cyanohydrin esters from aroyl chlorides

Acknowledgment The authors thank the National Natural Science Foundation of China (21162024) for the financial support of this work.

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