Direct Esterification of Aromatic Aldehydes with Tetraphenylphosphonium Bromide under Oxidative N-Heterocyclic Carbene Catalysis

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An unconventional reagent, tetraphenylphosphonium bromide, was employed as a phenyl source in the direct transformation of aromatic aldehydes to the corresponding phenyl esters under oxidative N-heterocyclic carbene (NHC) catalysis. The phenyl esters were obtained in moderate yields under mild and organocatalytic conditions.

Introduction. – Tetraphenylphosphonium salts have gained momentous attention due to their eclectic range of applications as ion-pair extractants for the heavy metals [1], phase-transfer catalysts [2][3], herbicides [4], ionic liquids [5], and as conducting materials [6]. Some of the tetraphenylphosphonium salts have been utilized as molecular probes for imaging tumors [7]. Apart from these applications, tetraphenylphosphonium bromide (Ph_4PBr) has also been used as an additive in metal-catalyzed cross-coupling reactions [8–11]. However, very few reports are available in the literature, where tetraphenylphosphonium salts served as an aryl-transfer reagents. *Yamamoto* and co-workers reported Pd-catalyzed arylation of electron-deficient olefins with Ph_4PCl [12]. Recently, *Chang* and co-workers have shown that Ph_4PCl could be effectively utilized as a phenyl source for Pd-catalyzed *Heck*, *Suzuki*, and *Sonagashira* coupling reactions [13].

Oxidative N-heterocyclic carbene catalysis [14][15], a type of N-heterocyclic carbene (NHC) catalysis [16–25], has been emerging as a powerful method for the construction of C–heteroatom bonds. This potential has been explored in a few important transformations, including aerobic oxidation of aldehydes [26][27], esterification [28–36], lactone formation [37–39], and amidation reactions [40]. Recently, we reported an efficient and environmentally friendly synthesis of aryl esters from aromatic aldehydes and aryl boronic acids under oxidative N-heterocyclic carbene catalysis [41]. We also reported that a combination of oxidative N-heterocyclic carbene catalysis and 'click chemistry' was very effective for the one-pot synthesis of ester containing 1,2,3-triazoles from aldehydes [42]. Herein, we report an alternative method for the synthesis of phenyl esters from aromatic aldehydes and Ph_4PBr using N-heterocyclic carbene as a catalyst under aerobic conditions.

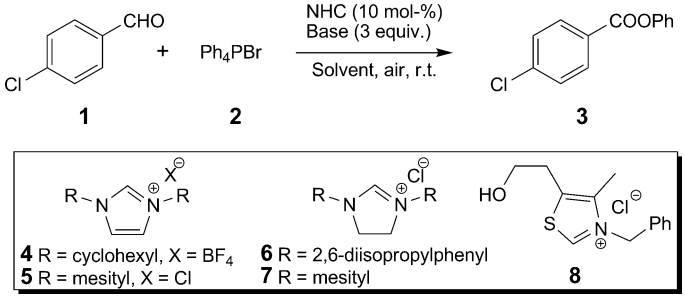
Results and Discussion. – While developing an organocatalytic method for the preparation of aryl esters [41], we came across a few alternative aryl sources, especially phenyl-transfer agents, which include tetraphenylphosphonium salts. Since Ph_4PBr is

commercially available and has not been utilized for esterification reactions so far, we decided to study it as a phenyl source in the oxidative esterification reaction.

The optimization studies were carried out with 4-chlorobenzaldehyde (**1**) as a model substrate and Ph_4PBr (**2**) as a phenyl source. A variety of NHC precursors, **4**–**8**, were screened for this oxidative esterification under different conditions (*Table 1*). All these reactions were carried out under aerobic conditions. Our initial attempt in the optimization studies using NHC precursor **4** was disappointing, as the anticipated product **3** was not observed even after 24 h (*Entry 1*, *Table 1*). However, when the experiment was conducted with **5** as a catalyst, the expected ester **3** was obtained in 20% yield (*Entry 2*). Encouraged by this result, we performed the optimization studies with a few more NHC precursors and different bases, and the results are compiled in *Table 1*. Of the few conditions tried, the best was found as indicated in *Entry 3* (*Table 1*); NHC precursor **6** was used as a catalyst, and the phenyl ester **3** was obtained in 67% yield after 4 h at room temperature.

After having found an appropriate reaction condition, we focused our attention on the scope of this transformation. Consequently, a variety of aromatic aldehydes, **9a**–**9o**, were subjected to the oxidative esterification reaction with Ph_4PBr under optimized conditions, and the results are collected in *Table 2*. In most of the cases, the required esters were obtained in moderate yields. Halogenated aromatic aldehydes such as 4-

Table 1. *Optimization Studies*[a])

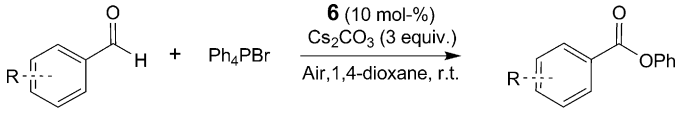


Entry	NHC	Base	Solvent	Time [h]	Yield [%][b])
1	**4**	Cs_2CO_3	Dioxane	24	0
2	**5**	Cs_2CO_3	Dioxane	26	20
3	**6**	**Cs_2CO_3**	**Dioxane**	**4**	**67**
4	**7**	Cs_2CO_3	Dioxane	36	56
5	**8**	Cs_2CO_3	Dioxane	24	Trace
6	**6**	K_2CO_3	Dioxane	36	23
7	**6**	$^iPr_2NEt$	Dioxane	24	0
8	**6**	KO^{t}Bu	Dioxane	24	Trace
9	**6**	Cs_2CO_3	Toluene	14	44
10	**6**	Cs_2CO_3	THF	4	56
12	**6**	Cs_2CO_3	DME	5	33
13	**6**	Cs_2CO_3	DCM	30	24

[a]) Reaction conditions: **1**/**2** 1.3 : 1 equiv. in 0.15M solution. [b]) Yield of isolated **3**; r.t., 32–35°.

bromobenzaldehyde and 4-fluorobenzaldehyde gave the corresponding esters, **10a** and **10b**, in 47 and 55%, respectively. This methodology worked relatively better in the cases of electron-rich aromatic aldehydes, *i.e.*, **10g**, **10j**, and **10k**. In the case of benzaldehyde (**9c**), phenyl benzoate (**10c**) was obtained in 41% yield after 14 h. Electron-poor aromatic aldehydes such as 4-formylbenzonitrile (**9d**) and 3-nitrobenzaldehyde (**9f**) gave the esters **10d** and **10f** in 33 and 32% yields, respectively. 3-Fluorobenzaldehyde (**9o**) gave the corresponding ester, **10o**, in 40% yield in 5 h. The yield of the ester **10e** is slightly better in the case of methyl 4-formylbenzoate. 4-(Trifluoromethyl)-benzaldehyde (**9h**) was also converted to the product **10h** in 55% yield. In the case of alkyl- and aryl-substituted benzaldehydes such as 4-ethylbenzaldehyde (**9i**) and 4-phenylbenzaldehyde (**9n**), the reaction was very slow, and the esters **10i** and **10n** were obtained in 38 and 39% yield, respectively, after 48 h. A few heteroaromatic aldehydes, such as furfural (**9l**) and thiophene-2-carbaldehyde (**9m**) provided the corresponding esters **10l** and **10m** in 48 and 50% yields, respectively. Aliphatic aldehydes, such as dihydrocinnamaldehyde and cyclohexanecarboxaldehyde failed to give the desired products; instead the starting materials were decomposed, since the reaction medium was highly basic. Almost in all the cases, a considerable amount of the corresponding acid was also formed along with ester. This could be due to the competitive oxidation of aldehydes to acids under oxidative NHC-catalyzed conditions [26][27]. It is also known that Ph_4PBr decomposes under strong basic conditions [43]. These are probably the reasons why lower yields were obtained in most of the cases.

Table 2. *Scope with Respect to the Substrate*[a])



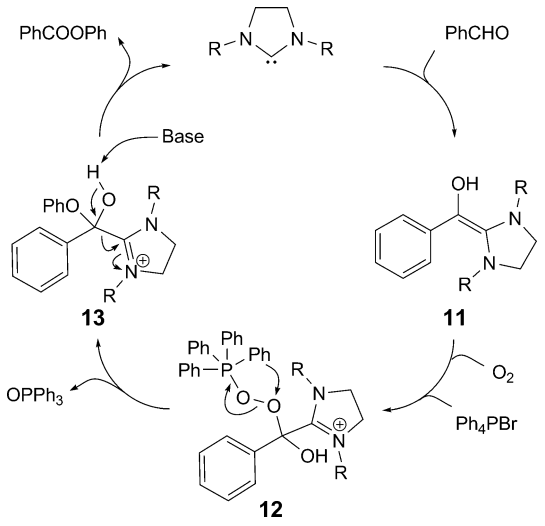
9a – 9o **2** **10a – 10o**

Entry	Product	R	Time [h]	Yield [%]
1	**10a**	4-Br	36	47
2	**10b**	4-F	5	55
3	**10c**	H	14	41
4	**10d**	4-CN	12	33
5	**10e**	4-COOMe	5	43
6	**10f**	3-NO_2	6	32
7	**10g**	3,4-(Methylenedioxy)	28	58
8	**10h**	4-CF_3	9	55
9	**10i**	4-Et	48	38
10	**10j**	4-*t*Bu	36	48
11	**10k**	4-MeO	48	43
12	**10l**	Furan-2-yl[b])	3	48
13	**10m**	Thiophene-2-yl[b])	48	50
14	**10n**	4-Ph	48	39
15	**10o**	3-F	5	40

[a]) Reaction conditions: **9**/**2** 1.3 : 1 equiv. in 0.15M solution in 1,4-dioxane; r.t., 32–35°. [b]) Instead of R–C_6H_4.

At this stage, our attention was shifted towards understanding the mechanism of this reaction. Careful monitoring of the reaction between 4-chlorobenzaldehyde and Ph_4PBr under the standard conditions revealed that triphenylphosphine oxide (Ph_3PO) and 4-chlorobenzoic acid were the by-products. Since 4-chlorobenzoic acid was observed in the reaction, we initially thought that the reaction proceeds *via* acid, which then reacts with Ph_4PBr under basic conditions to give the product and Ph_3PO. To confirm this, an experiment was performed by treating 4-chlorobenzoic acid with Ph_4PBr in 1,4-dioxane using 3 equiv. of Cs_2CO_3 as a base. However, the phenyl ester **3** was not observed even after 24 h at room temperature. This clearly indicates that the reaction does not proceed *via* acid intermediate. Another possible intermediate for this reaction could be PhOH, which might be formed by the decomposition of Ph_4PBr under oxidative conditions. Although PhOH formation was not observed (by TLC) in our experiments, we carried out an experiment, in which Ph_4PBr was exposed to NHC and air (O_2) in 1,4-dioxane under basic condition. However, PhOH was not detected even after stirring the reaction mixture for a prolonged period at room temperature. It is evident from the above mentioned experiments that the reaction involves neither PhCOOH nor PhOH as an intermediate. Based on these observations, we propose a concerted mechanism, which is depicted in the *Scheme*.

Scheme. *Proposed Mechanism*



We presume that the *Breslow* intermediate **11**, formed by the reaction of PhCHO (**9c**) with NHC, reacts with O_2 and Ph_4PBr in a concerted manner to give intermediate **12**, which decomposes readily to intermediate **13** with the expulsion of Ph_3PO. On deprotonation, intermediate **13** releases the product along with NHC.

Conclusions. – We have developed an alternative method for the direct synthesis of phenyl esters from aromatic aldehydes under oxidative N-heterocyclic carbene

catalysis. Although the yield of the esters was moderate in most cases, Ph_4PBr was investigated, for the first time, as a Ph source in this methodology.

Experimental Part

General. Most of the reagents and starting materials used were purchased from commercial sources and used as such. TLC: *Merck* silica gel *60 F_{254}* plates with AcOEt/hexane as an eluent. Column chromatography (CC): silica gel (SiO_2; 100–200 mesh). IR Spectra: *PerkinElmer* FT-IR spectrometer; with KBr, in cm^{-1}. ^1H- and ^{13}C-NMR spectra: in $CDCl_3$ on 400-MHz *Brucker* FT-NMR spectrometer; chemical shifts (δ) in ppm relative to TMS, and coupling constants (J) in Hz.

General Procedure for the Oxidative Esterification of Aromatic Aldehydes with Ph_4PBr. Aromatic aldehyde (0.37 mmol) was added to a suspension of Ph_4PBr (0.29 mmol), **6** (0.029 mmol), and Cs_2CO_3 (0.86 mmol) in 1,4-dioxane (2 ml) at r.t. (32–35°). After completion of the reaction, the reaction mass was filtered, washed with AcOEt (10 ml), and dried (Na_2SO_4). The solvent was removed under reduced pressure, and the residue was purified by CC (SiO_2; hexane/AcOEt (5%)) to give the pure ester.

Phenyl 4-Chlorobenzoate (**3**) [41]. Yield: 67%. White solid. M.p. 104–106°. IR (KBr): 1732. ^1H-NMR: 8.15 (*d*, *J* = 8.7, 2 H); 7.49 (*d*, *J* = 8.7, 2 H); 7.47–7.42 (*m*, 2 H); 7.31–7.27 (*m*, 1 H); 7.23–7.19 (*m*, 2 H). ^{13}C-NMR: 164.5; 150.9; 140.3; 131.7; 129.7; 129.1; 128.2; 126.2; 121.8.

Phenyl 4-Bromobenzoate (**10a**) [41]. Yield: 47%. White solid. M.p. 117–118°. IR (KBr): 1731. ^1H-NMR: 8.07 (*d*, *J* = 8.6, 2 H); 7.66 (*d*, *J* = 8.6, 2 H); 7.46–7.41 (*m*, 2 H); 7.31–7.27 (*m*, 1 H); 7.22–7.20 (*m*, 2 H). ^{13}C-NMR: 164.7; 150.9; 132.1; 131.8; 129.7; 129.0; 128.6; 126.2; 121.8.

Phenyl 4-Fluorobenzoate (**10b**) [44]. Yield: 55%. White solid. M.p. 63–65°. IR (KBr): 1734. ^1H-NMR: 8.26–8.21 (*m*, 2 H); 7.46–7.41 (*m*, 2 H); 7.30–7.26 (*m*, 1 H); 7.22–7.16 (*m*, 4 H). ^{13}C-NMR: 166.3 (*d*, *J* = 250.5); 164.4; 151.0; 132.9 (*d*, *J* = 9.4); 129.7; 128.9; 126.0 (*d*, *J* = 19.2); 121.8; 115.9 (*d*, *J* = 21.8).

Phenyl Benzoate (**10c**) [41]. Yield: 41%. White solid. M.p. 66–68°. IR (KBr): 1731. ^1H-NMR: 8.24–8.21 (*m*, 2 H); 7.67–7.63 (*m*, 1 H); 7.54–7.51 (*m*, 2 H); 7.47–7.42 (*m*, 2 H); 7.31–7.27 (*m*, 1 H); 7.24–7.21 (*m*, 2 H). ^{13}C-NMR: 165.3; 151.1; 133.7; 130.3; 129.7; 129.6; 128.7; 126.0; 121.9.

Phenyl 4-Cyanobenzoate (**10d**) [41]. Yield: 33%. White solid. M.p. 94–96°. IR (KBr): 1742, 2365. ^1H-NMR: 8.31 (*d*, *J* = 8.7, 2 H); 7.83 (*d*, *J* = 8.7, 2 H); 7.48–7.44 (*m*, 2 H); 7.33–7.29 (*m*, 1 H); 7.24–7.20 (*m*, 2 H). ^{13}C-NMR: 163.7; 150.7; 133.6; 132.6; 130.8; 129.8; 126.5; 121.6; 118.0; 117.6.

Methyl Phenyl Benzene-1,4-Dicarboxylate (**10e**) [41]. Yield: 43%. White solid. M.p. 106–108°. IR (KBr): 1734. ^1H-NMR: 8.27 (*d*, *J* = 8.6, 2 H); 8.18 (*d*, *J* = 8.6, 2 H); 7.47–7.42 (*m*, 2 H); 7.32–7.28 (*m*, 1 H); 7.24–7.21 (*m*, 2 H); 4.0 (*s*, 3 H). ^{13}C-NMR: 166.4; 164.6; 150.9; 134.6; 133.5; 130.3; 129.9; 129.7;126.3; 121.7; 52.7.

Phenyl 3-Nitrobenzoate (**10f**) [45]. Yield: 32%. Pale-yellow solid. M.p. 156–158°. IR (KBr): 1728, 2923. ^1H-NMR: 9.05–9.04 (*m*, 1 H); 8.55–8.49 (*m*, 2 H); 7.76–7.72 (*m*, 1 H); 7.49–7.44 (*m*, 2 H); 7.34–7.3 (*m*, 1 H); 7.26–7.22 (*m*, 2 H). ^{13}C-NMR: 163.1; 150.6; 148.5; 135.9; 131.5; 130.3; 129.8; 128.1; 126.6; 125.3; 121.6.

Phenyl 1,3-Benzodioxole-5-carboxylate (**10g**) [41]. Yield: 58%. White solid. M.p. 80–82°. IR (KBr): 1715. ^1H-NMR: 7.83 (*dd*, *J* = 8.2, 1.7, 1 H); 7.62 (*d*, *J* = 1.7, 1 H); 7.45–7.40 (*m*, 2 H); 7.29–7.24 (*m*, 1 H); 7.21–7.18 (*m*, 2 H); 6.91 (*d*, *J* = 8.1, 1 H); 6.08 (*s*, 2 H). ^{13}C-NMR: 164.7; 152.3; 151.1; 148.1; 129.6; 126.4; 126.0; 123.6; 121.9; 110.1; 108.3; 102.1.

Phenyl 4-(Trifluoromethyl)benzoate (**10h**) [45]. Yield: 55%. Pale-yellow solid. M.p. 81–83°. IR (KBr): 1733. ^1H-NMR: 8.33 (*d*, *J* = 8.04, 2 H); 7.79 (*d*, *J* = 8.12, 2 H); 7.48–7.43 (*m*, 2 H); 7.33–7.29 (*m*, 1 H); 7.25–7.21 (*m*, 2 H). ^{13}C-NMR: 164.2; 150.8; 135.2 (*q*, *J* = 32.9); 133.0 (*q*, *J* = 1.4); 130.7; 129.8; 126.4; 125.8 (*q*, *J* = 3.66); 123.7 (*q*, *J* = 270.6); 121.7.

Phenyl 4-Ethylbenzoate (**10i**) [41]. Yield: 38%. White solid. M.p. 62–63°. IR (KBr): 1726. ^1H-NMR: 8.13 (*d*, *J* = 8.1, 2 H); 7.45–7.41 (*m*, 2 H); 7.34 (*d*, *J* = 8.0, 2 H); 7.29–7.26 (*m*, 1 H); 7.22 (*d*, *J* = 8.0, 2 H); 2.75 (*q*, *J* = 7.6, 2 H); 1.29 (*t*, *J* = 7.6, 3 H). ^{13}C-NMR: 165.4; 151.2; 150.7; 130.5; 129.6; 128.2; 127.2; 125.9; 121.9; 29.2; 15.4.

Phenyl 4-(tert-*Butyl)benzoate* (**10j**) [46]. Yield: 48%. White solid. M.p. 142–146°. IR (KBr): 1729. ^{1}H-NMR: 8.18 (*d*, *J* = 8.4, 2 H); 7.57 (*d*, *J* = 8.4, 2 H); 7.49–7.45 (*m*, 2 H); 7.33–7.24 (*m*, 3 H); 1.41 (*s*, 9 H). ^{13}C-NMR: 165.3; 157.5; 151.2; 130.2; 129.6; 126.9; 125.9; 125.7; 121.9; 35.3; 31.3.

Phenyl 4-Methoxybenzoate (**10k**) [41]. Yield: 43%. White solid. M.p. 75–77°. IR (KBr): 1727. ^{1}H-NMR: 8.14 (*d*, *J* = 8.8, 2 H); 7.42–7.39 (*m*, 2 H); 7.26–7.22 (*m*, 1 H); 7.20–7.18 (*m*, 2 H); 6.97 (*d*, *J* = 8.8, 2 H); 3.88 (*s*, 3 H). ^{13}C-NMR: 165.9; 164.0; 151.2; 132.4; 129.6; 125.9; 122.0; 121.9; 114.0; 55.7.

Phenyl Furan-2-carboxylate (**10l**) [41]. Yield: 48%. White solid. M.p. 54–56°. IR (KBr): 1736. ^{1}H-NMR: 7.68 (*dd*, *J* = 1.7, 0.8, 1 H); 7.45–7.40 (*m*, 2 H); 7.39 (*dd*, *J* = 3.5, 0.8, 1 H); 7.30–7.25 (*m*, 1 H); 7.23–7.20 (*m*, 2 H); 7.60 (*dd*, *J* = 3.5, 1.8, 1 H). ^{13}C-NMR: 157.1; 150.3; 147.3; 144.2; 129.7; 126.2; 121.8; 119.6; 112.3.

Phenyl Thiophene-2-carboxylate (**10m**) [41]. Yield: 50%. Semisolid. IR (KBr): 1738. ^{1}H-NMR: 7.99 (*dd*, *J* = 3.8, 1.3, 1 H); 7.67 (*dd*, *J* = 5.0, 1.3, 1 H); 7.45–7.40 (*m*, 2 H); 7.30–7.25 (*m*, 1 H); 7.24–7.21 (*m*, 2 H); 7.18 (*dd*, *J* = 5.0, 3.8, 1 H). ^{13}C-NMR: 160.7; 150.7; 134.8; 133.6; 133.1; 129.6; 128.2; 126.1; 121.8.

Phenyl 1,1′-Biphenyl-4-carboxylate (**10n**) [41]. Yield: 39%. White solid. M.p. 158–160°. IR (KBr): 1731. ^{1}H-NMR: 8.28 (*d*, *J* = 8.2, 2 H); 7.74 (*d*, *J* = 8.2, 2 H); 7.67 (*d*, *J* = 7.7, 2 H); 7.52–7.41 (*m*, 5 H); 7.31–7.24 (*m*, 3 H). ^{13}C-NMR: 165.2; 151.2; 146.5; 140.0; 130.9; 129.7; 129.1; 128.5; 128.4; 127.5; 127.4; 126.0; 121.9.

Phenyl 3-Fluorobenzoate (**10o**) [41]. Yield: 40%. White solid. M.p. 58–59°. IR (KBr): 1736. ^{1}H-NMR: 8.02–8.00 (*m*, 1 H); 7.91–7.87 (*m*, 1 H); 7.37–7.27 (*m*, 2 H); 7.23–7.20 (*m*, 2 H); 7.53–7.42 (*m*, 3 H). ^{13}C-NMR: 164.1 (*d*, *J* = 25.4); 161.5; 150.9; 131.9 (*d*, *J* = 3.8); 130.4 (*d*, *J* = 7.8); 129.7; 126.3; 126.1 (*d*, *J* = 3.1); 121.7; 120.9 (*d*, *J* = 21.2); 117.2 (*d*, *J* = 22.9).

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