ELECTRONIC SUPPLEMENTARY INFORMATION

One Stone Two Birds: Cobalt Catalyzed in situ Generation of Isocyanate and Benzyl alcohol for the Synthesis of N-aryl Carbamates

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A: General Information

The reactions and manipulations were performed under an atmosphere of argon by using standard Schlenk techniques and glovebox (Mikrouna, Supper1220/750). Anhydrous toluene was distilled from sodium benzophenone ketyl prior to use. $^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker-Avance 400 MHz spectrometer. CDCl$_3$ or CD$_3$OD were used as solvent. Chemical shifts ($\delta$) were reported in ppm with tetramethylsilane as internal standard and $J$ values were given in Hz. Melting points were measured on X-4 melting point apparatus and uncorrected. Column chromatography was performed with silica gel using petroleum ether and ethyl acetate as eluents.

B: Procedure of the reactions

Typical procedure for the synthesis of 3aa. CoI$_2$ (0.02 mmol), tris-(4-dimethylaminophenyl)-phosphine (0.048 mmol), Zinc powder (0.6 mmol) and 1 mL of toluene were added to a Schlenk tube under an argon atmosphere in a glovebox. The mixture was stirred at room temperature for 30 min. Then N-Boc protected aniline 1a (0.2 mmol), benzyl formate 2a (0.6 mmol) and toluene (1 mL) were added. The reaction mixture was stirred under argon atmosphere at 120º C for 24 h. After vacuum evaporation of the solvent, the residue was purified by silica gel column chromatography to provide the desired product 3aa (42 mg, 92% yield).
### Table S1: Screening of different Ligands and solvents

- **Entry**
- **Ligand**
- **Zinc (equiv)**
- **Solvent**
- **Temp (°C)**
- **Time (h)**
- **Yield (%)**

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<th>Entry</th>
<th>Ligand</th>
<th>Zinc (equiv)</th>
<th>Solvent</th>
<th>Temp (°C)</th>
<th>Time (h)</th>
<th>Yield (%)</th>
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*Reaction conditions: CoI₂ (10 mol%), monophosphine ligands (24 mol%) or diphosphine ligands (12 mol%) and zinc powder were stirred in solvent (1 mL) for 30 minutes at room temperature under Ar. N-Boc protected aniline 1a (0.2 mmol), benzyl formate 2a (0.6 mmol) and 1 mL solvent were added. The reaction mixture was stirred under argon atmosphere at 100 °C or 120 °C. The reaction was monitored by TLC.

²Isolated yields.

²Mn (3 equiv.) was used instead of Zn.
D: Scheme S1: Control reactions to identify the reaction intermediates

\[ \text{L: } \text{P(phen)} \text{, Col}_2, \text{L} \]

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<td>12% yield, 48 h</td>
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<td>12 h, 89% yield</td>
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S4
E: Characterization Data of Products

Benzyl phenylcarbamate (3aa)

\[
\text{H} \quad \text{O} \quad \text{Ph} \\
\text{N} \quad \text{O} \\
\text{Boc} \quad \text{Ph}
\]

White solid, 42 mg, 92% yield, mp 70-72 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.42-7.29 (m, 9H), 7.07 (t, \(J = 7.4\) Hz, 1H), 6.75 (s, 1H), 5.20 (s, 2H). \(^1^3\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 153.37, 137.77, 136.04, 129.11, 128.67, 128.42, 128.37, 123.56, 118.67, 67.05.

Benzyl (4-methoxyphenyl)carbamate (3ba)

\[
\text{MeO} \quad \text{O} \quad \text{Ph} \\
\text{N} \quad \text{O} \\
\text{Boc} \quad \text{Ph}
\]

White solid, 46 mg, 90% yield, mp 98-100 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.40-7.29 (m, 7H), 6.86-6.82 (m, 2H), 6.76 (s, 1H), 5.19 (s, 2H), 3.78 (s, 3H). \(^1^3\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 155.98, 153.85, 136.22, 130.91, 128.64, 128.33, 120.72, 114.26, 66.94, 55.53.

Benzyl (2-methoxyphenyl)carbamate (3ca)

\[
\text{O} \quad \text{MeO} \quad \text{Ph} \\
\text{N} \quad \text{O} \\
\text{Boc} \quad \text{Ph}
\]

White solid, 44 mg, 86% yield, mp 72-74 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 8.13 (s, 1H), 7.44-7.33 (m, 6H), 7.03-6.95 (m, 2H), 6.87-6.85 (m, 1H), 5.22 (s, 2H), 3.85 (s, 3H). \(^1^3\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 153.30, 147.60, 136.20, 128.66, 128.39, 127.56, 122.85, 121.14, 118.15, 109.98, 66.96, 55.63.

Benzyl (3-methoxyphenyl)carbamate (3da)

\[
\text{MeO} \quad \text{O} \quad \text{Ph} \\
\text{N} \quad \text{O} \\
\text{Boc} \quad \text{Ph}
\]

Colorless oil, 44 mg, 86% yield. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.41-7.32 (m, 5H), 7.19 (t, \(J = 8.2\), 1H), 7.14 (s, 1H), 6.89-6.84 (m, 2H), 6.63 (dd, \(J = 8.2, 2.3\) Hz, 1H), 5.20 (s, 2H), 3.79 (s, 3H). \(^1^3\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 160.30, 153.33, 139.10, 136.05, 129.78, 128.65, 128.39, 128.32, 110.96, 109.33, 104.44, 67.03, 55.28.
**Benzyl p-tolylcarbamate (3ea)**

![Structure Image]

White solid, 40 mg, 82% yield, mp 77-79 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.39-7.30 (m, 5H), 7.27-7.24 (m, 2H), 7.08 (d, \(J = 8.0\) Hz, 2H), 6.78 (s, 1H), 5.17 (s, 2H), 2.29 (s, 3H). \(^13\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 153.56, 136.19, 135.25, 130.09, 129.57, 128.63, 128.50, 128.34, 118.87, 66.94, 20.78.

**Benzyl (4-(tert-butyl)phenyl)carbamate (3fa)**

![Structure Image]

White solid, 47 mg, 83% yield, mp 102-104 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.42 -7.33 (m, 9H), 6.78 (s, 1H), 5.21 (s, 2H), 1.32 (s, 9H). \(^13\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 153.60, 146.51, 136.21, 135.19, 128.64, 128.34, 128.32, 125.90, 118.65, 66.97, 34.31, 31.43.

**Benzyl (4-hydroxyphenyl)carbamate (3ga)**

![Structure Image]

White solid, 20 mg, 41% yield, mp 160-162 °C. \(^1\)H NMR (400 MHz, Methanol-\(d4\)) \(\delta\) 7.40-7.29 (m, 5H), 7.21-7.20 (m, 2H), 6.73-6.69 (m, 2H), 5.14 (d, \(J = 2.1\) Hz, 2H). \(^13\)C NMR (101 MHz, Methanol-\(d4\)) \(\delta\) 153.20, 136.84, 130.53, 128.11, 127.66, 127.53, 120.72, 114.87, 66.00.

**Benzyl (4-fluorophenyl)carbamate (3ha)**

![Structure Image]

White solid, 46 mg, 94% yield, mp 76-78 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.40-7.32 (m, 7H), 7.02-6.96 (m, 2H), 6.82 (s, 1H), 5.19 (s, 2H). \(^13\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 160.26, 157.85, 153.62, 135.99, 133.79, 128.66, 128.43, 128.33, 120.56, 115.79, 115.57, 67.13.

**Benzyl (4-chlorophenyl)carbamate (3ia)**

![Structure Image]

White solid, 46 mg, 89% yield, mp 109-111 °C. \(^1\)H NMR (400 MHz, Chloroform-\(d\)) \(\delta\) 7.37-7.30(m, 7H), 7.24-7.22 (m, 2H), 6.84 (s, 1H), 5.17(s, 2H). \(^13\)C NMR (101 MHz, Chloroform-\(d\)) \(\delta\) 153.39, 136.45, 135.88, 129.05, 128.68, 128.48, 128.35, 120.02, 67.22.
Benzyl (4-bromophenyl)carbamate (3ju)

White solid, 37 mg, 60% yield, mp 115-117 °C. 1H NMR (400 MHz, Chloroform-d) δ 7.42-7.33 (m, 7H), 7.29-7.27 (m, 2H), 6.72 (s, 1H), 5.19 (s, 2H). 13C NMR (101 MHz, Chloroform-d) δ 153.20, 136.91, 135.81, 132.02, 128.70, 128.40, 120.21, 116.05, 67.25.

Benzyl (4-nitrophenyl)carbamate (3ka)

White solid, 44 mg, 80% yield, mp 163-165 °C. 1H NMR (400 MHz, Chloroform-d) δ 8.21-8.17 (m, 2H), 7.56-7.54 (m, 2H), 7.41-7.35 (m, 5H), 7.13 (s, 1H), 5.23 (s, 2H). 13C NMR (101 MHz, Chloroform-d) δ 152.64, 143.81, 143.09, 135.33, 128.75, 128.71, 128.48, 125.24, 117.78, 67.75.

Benzyl naphthalen-1-ylcarbamate (3la)

White solid, 40 mg, 72% yield, mp 130-132 °C. 1H NMR (400 MHz, Chloroform-d) δ 7.85-7.81 (m, 3H), 7.63 (d, J = 8.1 Hz, 1H), 7.47-7.32 (m, 8H), 7.08 (s, 1H), 5.23 (s, 2H). 13C NMR (101 MHz, Chloroform-d) δ 154.33, 136.11, 134.09, 132.44, 128.77, 128.69, 128.45, 126.29, 126.06, 125.85, 125.15, 120.51, 67.35.

Benzyl cyclohexylcarbamate (3ma)

White solid, 38 mg, 82% yield, mp 64-66 °C. 1H NMR (400 MHz, Chloroform-d) δ 7.37-7.28 (m, 5H), 5.08 (s, 2H), 4.71 (d, J = 4.9 Hz, 1H), 3.54-3.47 (m, 1H), 1.94-1.91 (m, 2H), 1.72-1.66 (m, 2H), 1.60-1.57 (m, 1H), 1.38-1.25 (m, 2H), 1.20-1.07 (m, 3H). 13C NMR (101 MHz, Chloroform-d) δ 155.58, 136.70, 128.54, 128.18, 128.09, 66.48, 49.90, 33.41, 25.49, 24.82.

Benzyl benzylicarbamate (3na)

White solid, 26 mg, 54% yield, mp 64-66 °C. 1H NMR (400 MHz, Chloroform-d) δ 7.37-7.26 (m, 10H), 5.14 (s, 3H), 4.38 (d, J = 5.6 Hz, 2H). 13C NMR (101 MHz, Chloroform-d) δ 156.46, 138.41, 136.49, 128.70, 128.56, 128.18, 127.54, 66.90, 45.16.
2-chlorobenzyl phenylcarbamate (3ab)

White solid, 34 mg, 64% yield, mp 82-84 °C. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.37-7.28 (m, 4H), 7.22-7.13 (m, 4H), 6.97 (t, $J = 7.4$ Hz, 1H), 6.79 (s, 1H), 5.22 (s, 2H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 153.21, 137.73, 133.80, 133.78, 130.12, 129.69, 129.63, 129.12, 126.98, 123.63, 118.70, 64.33.

3-chlorobenzyl phenylcarbamate (3ac)

White solid, 20 mg, 38% yield, mp 91-93 °C. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.39-7.25 (m, 8H), 7.09-7.05 (m, 1H), 6.75 (s, 1H), 5.17 (s, 2H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 153.10, 138.11, 137.59, 134.50, 129.92, 129.13, 128.47, 128.19, 126.19, 123.71, 118.76, 66.05.

4-chlorobenzyl phenylcarbamate (3ad)

White solid, 34 mg, 65% yield, mp 94-96 °C. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.40-7.28 (m, 9H), 7.08 (t, $J = 7.3$ Hz, 1H), 6.83 (s, 1H), 5.15 (s, 2H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 153.25, 137.65, 134.61, 134.25, 129.68, 129.11, 128.81, 123.68, 118.77, 66.16.

4-bromobenzyl phenylcarbamate (3ae)

White solid, 48 mg, 79% yield, mp 95-96 °C. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.52-7.48 (m, 2H), 7.38-7.36 (m, 2H), 7.32-7.27 (m, 4H), 7.09-7.06 (m, 1H), 6.66 (s, 1H), 5.15 (s, 2H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 153.28, 137.67, 135.14, 131.78, 129.96, 129.12, 123.71, 122.40, 118.83, 66.19.

4-nitrobenzyl phenylcarbamate (3af)

White solid, 47 mg, 87% yield, mp 124-126 °C. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 8.23 (d, $J = 8.7$Hz, 2H), 7.55 (d, $J = 8.6$ Hz, 2H), 7.40-7.38 (m, 2H), 7.32 (t, $J = 7.4$ Hz, 2H), 7.09 (t, $J = 7.3$ Hz, 1H), 6.78 (s, 1H), 5.29 (s, 2H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 147.73, 143.47, 137.34, 129.20, 128.38, 123.88, 118.76, 65.44.
4-methylbenzyl phenylcarbamate (3ag)

White solid, 29 mg, 62% yield, mp 76-78°C. ¹H NMR (400 MHz, Chloroform-d) δ 7.40-7.38 (m, 2H), 7.33-7.28 (m, 4H), 7.19 (d, J = 7.9 Hz, 2H), 7.07 (t, J = 7.4 Hz, 1H), 6.74 (s, 1H), 5.17 (s, 2H), 2.37 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 153.44, 138.30, 137.83, 133.02, 129.35, 129.09, 128.56, 123.50, 118.66, 67.02, 21.28.

Phenethyl phenylcarbamate (3ah)

White solid, 29 mg, 62% yield, mp 77-79°C. ¹H NMR (400 MHz, Chloroform-d) δ 7.34-7.31 (m, 9H), 7.07-7.02 (m, 1H), 6.67 (s, 1H), 4.38 (t, J = 7.0 Hz, 2H), 2.98 (t, J = 7.0 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-d) δ 153.50, 137.82, 129.08, 128.96, 128.59, 126.65, 123.49, 118.68, 65.65, 35.41.

3-methoxybenzyl phenylcarbamate (3aj)

Colorless oil, 45 mg, 87% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.41-7.39 (m, 2H), 7.33-7.28 (m, 3H), 7.09-7.05 (m, 1H), 6.99 (d, J = 7.6 Hz, 1H), 6.95-6.94 (m, 1H), 6.90-6.86 (m, 2H), 5.18 (s, 2H), 3.81 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 159.8, 153.4, 137.8, 137.6, 129.7, 129.1, 123.5, 120.5, 118.7, 113.9, 113.7, 66.9, 55.3.

4-fluorobenzyl phenylcarbamate (5ab)

White solid, 47 mg, 96% yield, mp 79-80°C. ¹H NMR (400 MHz, Chloroform-d) δ 7.40-7.37 (m, 4H), 7.31 (t, J = 7.4 Hz, 2H), 7.09-7.03 (m, 3H), 6.71 (s, 1H), 5.16 (s, 2H). ¹³C NMR (101 MHz, Chloroform-d) δ 163.9, 161.5, 153.2, 153.1, 137.7, 131.9, 131.9, 130.4, 130.3, 129.1, 123.6, 118.7, 115.6, 115.4, 66.3.

Cyclohexyl phenylcarbamate (5ac)

Colorless oil, 43 mg, 98% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.40-7.38 (m, 2H), 7.31-7.27 (m, 2H), 7.07-7.02 (m, 1H), 6.71 (s, 1H), 4.78-4.72 (m, 1H), 1.96-1.91 (m, 2H), 1.77-1.71 (m, 2H), 1.58-1.53 (m, 1H), 1.50-1.33 (m, 4H), 1.30-1.21 (m, 1H). ¹³C NMR (101 MHz, Chloroform-d) δ 153.3, 138.2, 129.0, 123.2, 118.5, 73.6, 31.9, 25.4, 23.8.
Ethyl phenylcarbamate (5ad)

Colorless oil, 27 mg, 82% yield. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.40-7.35 (m, 2H), 7.32-7.27 (m, 2H), 7.08-7.03 (m, 1H), 6.77 (s, 1H), 4.22 (q, $J = 7.1$ Hz, 2H), 1.31 (t, $J = 7.1$ Hz, 3H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 153.7, 138.0, 129.1, 123.3, 118.6, 61.2, 28.4, 14.6.
F: NMR Spectra of Products