Amidation of 1,3-diarylallylic compounds catalysed by 2,3-dichloro-5,6dicyano-1,4-benzoquinone with molecular oxygen as the terminal oxidant

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An efficient amidation has been developed of 1,3-diarylpropenes by carboxamides, sulfonamides, carbamates and anilines catalysed by 2,3-dichloro-5,6-dicyano-1,4-benzoquinone and benzoyl peroxide with molecular oxygen. The corresponding products were obtained in moderate to good yields.

Keywords: oxidative coupling, oxygen, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone, benzoyl peroxide

Oxidative coupling reactions play an important role in organic chemistry. With the concept of green chemistry,¹ the use of molecular oxygen as the terminal oxidant in an oxidation reaction has attracted much attention owing to its great abundance in nature and water as the only by-product. To accomplish the oxidation, a transition metal or transition-metal complex is usually required to activate the molecular oxygen.²⁻⁴ Recently, there has been a great deal of emphasis on metal-free reactions in organic synthesis because metal residues can pollute the pharmaceutical intermediates and target products. To the best of our knowledge, few efforts have been reported involving metal-free oxidative couplings which simultaneously employ molecular oxygen as the terminal oxidant.

Formation of C-N bonds is an important fundamental reaction in organic synthesis because many bioactive and medicinal molecules are nitrogen-containing compounds.5-7 Allylic amination represents one of the most common C-N bond formation methodologies. An efficient and atom economical approach to allylic amination is to use directly allylic alcohols as electrophiles with catalysts such as transitionmetal complexes or salts and Lewis or Brønsted acids.8-17 Despite the excellent progress which has been made, it would obviously be attractive if allylic amination might be achieved via C-H oxidative activation. Recently, our group has reported the metal-free coupling between 1,3-diarylpropenes and nitrogen nucleophiles promoted by a stoichiometric amount of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ).¹⁸ However, the use of a stoichiometric amount of DDQ caused purification difficulties because of the formation of the by-product DDQH₂. With an interest in developing an application using a catalytic amount of DDQ in the oxidative reaction,19 we wish to report an efficient amidation at a diarylallylic sp3 carbon hydrogen centre catalysed by DDQ with molecular oxygen as the terminal oxidant.

Results and discussion

To begin our study we chose 1,3-diphenylpropene (1a) and benzamide (2a) as model substrates. The reaction was catalysed by 10 mol% DDQ and 10 mol% benzoyl peroxide (BPO) at 100 °C in an atmosphere of oxygen (balloon). The desired coupling product 3a was obtained in 47% yield after 24 h (Table 1, entry 1). Performing the reaction under aerobic conditions or without BPO resulted in a low yield (Table 1, entries 3–4). No coupling product was obtained in the absence of DDQ (Table 1, entry 5). This indicated that DDQ was essential and that molecular oxygen and BPO could promote the reaction. Then the amounts of DDQ and BPO were examined (Table 1, entries 6–11). The best result was obtained when the catalytic

~	\searrow	∼Ph ⁺ PhCONH	D	DQ /Additive		1	NHCOPh I
Ph			NH ₂	O ₂ (balloon)	► Ph	\searrow	Ph
	1a	2a				3a	
Entry		DDQ/mol%	BPO	/mol%	T/°C		Yield/% ^b
1		10		10	100		47
2		10	1	0°	100		39
3		10		0	100		9
4 ^d		10		10	100		10
5		0		10	100		0
6		10		20	100		46
7		20		10	100		42
8		20		20	100		66
9		20		30	100		42
10		30		20	100		44
11		30		30	100		60
12		20		20	80		49
13		20		20	60		42
14		20		20	110		40

 a **1a** (0.24 mmol), **2a** (0.2 mmol), CH₃NO₂(1 mL), O₂ balloon.

Table 1 Optimisation of the reaction conditions^a

^bIsolated yields.

 $^\circ\!\text{Azobisisobutyronitrile}$ (AIBN) was used as an additive.

^dIn the absence of O₂

amounts of both DDQ and BPO were 20 mol%. The yields were 42% and 49% respectively when the temperature was decreased to 60 °C or 80 °C (Table 1, entries 12–13). The coupling product was obtained with 40% yield when the temperature was raised to 110 °C (Table 1, entry 14).

With the best reaction conditions identified, various amides were examined and the results are summarised in Table 2. Carboxamides including aromatic, heteroaromatic and cyclic were compatible with the present procedure and good yields were obtained (Table 2, entries 2-7). Benzene and methylbenzene sulfamides were also suitable substrates and the coupling products were obtained with moderate yields (Table 2, entries 10-11). The reaction of aniline and 1,3-diphenylpropene was carried out under the optimised condition but no desired coupling product was detected. When 1,4-dioxane was used as solvent instead of nitromethane, the product could be obtained in low yield. According to the literature,18 the addition sequence of the substrates was changed. To a solution of DDQ, BPO and 1,3-diarylpropene in 1,4-dioxane, after stirring for about 5 minutes at room temperature, aniline was added. The yields were promoted to 46-49% (Table 2, entries 12-13). As a result of this, the more useful but acid-sensitive carbamates such as CH₂OCO-, Boc- and Cbz- participated in the reaction to provide

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Table 2	2 Oxidative	coupling	reaction	of 1	1,3-diary	lpro	penes
					, , ,		

R ₁	$R_2 + R_3 - 1$	-NH ₂ DDQ (20 mol% BPO (20 mol% CH ₃ NO ₂ , 100 ° O ₂ (balloon)	$\frac{1}{C}$ R ₁	
Entry	R ₁ , R ₂ (1)	R ₃ (2)	Product (3)	Yield/% ^b
1	C ₆ H ₅ , C ₆ H ₅	C ₆ H ₅ CO	3a	66
2	$C_{6}H_{5}, C_{6}H_{5}$	4-CH ₃ C ₆ H ₄ CO	3b	90
3	$C_{6}H_{5}, C_{6}H_{5}$	4-CH ₃ OC ₆ H ₄ CO	3c	73
4	C_6H_5, C_6H_5	4-FC ₆ H ₄ CO	3d	70
5	$C_{6}H_{5}, C_{6}H_{5}$	3-CIC ₆ H ₄ CO	3e	71
6	C ₆ H ₅ , C ₆ H5	2-thienyICO	3f	57
7	$C_{6}H_{5}, C_{6}H_{5}$	$(CH_2)_3C(0)$	3g	54
8	C ₆ H ₅ , 4-BrC ₆ H ₄	C ₆ H ₅ CO	3h°	90
9	C ₆ H ₅ , 4-CH ₃ OC ₆ H ₄	C ₆ H ₅ CO	3i ⁰	41
10	$C_{6}H_{5}, C_{6}H_{5}$	$C_6H_5SO_2$	3j	40
11	$C_{6}H_{5}, C_{6}H_{5}$	$4-CH_3C_6H_4SO_2$	3k	61
12 ^d	$C_{6}H_{5}, C_{6}H_{5}$	C_6H_5	31	46
13 ^d	$C_{6}H_{5}, C_{6}H_{5}$	4-BrC ₆ H ₄	3m	49
14 ^d	$C_{6}H_{5}, C_{6}H_{5}$	CH ₃ OC(0)	3n	53
15 ^d	$C_{6}H_{5}, C_{6}H_{5}$	Boc	30	45
16 ^d	C_6H_5 , C_6H_5	Cbz	3p	57

 a1 (0.24 mmol), **2** (0.2 mmol), DDQ (20 mol%), BPO (20 mol%), CH $_{\rm 3}\rm NO_{2}$ (1 mL), 100 °C, 24 h, O $_{\rm 2}$ balloon.

^bIsolated yields.

 $^{\mathrm{c}}\text{A}$ mixture of $\alpha\text{-}$ and $\gamma\text{-}\text{products}$ was obtained.

^d1,4-Dioxane instead of CH₂NO₂.

the corresponding products with moderate yields (Table 2, entries 14–16).

A possible mechanism of the reaction was proposed (Scheme 1). In the experiment, the yield of coupling product was decreased dramatically when (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) was added to the reaction mixture. Byproduct 1,3-diphenylpropen-3-one, which might be generated by further oxidation of 1,3-diphenylpropene, was obtained. The reaction of asymmetrical 1,3-diarylpropene with benzamide gave a mixture of the corresponding α - and γ -products (Table 2, entries 8–9). According to the literature²⁰ and the above results, this indicated that the reaction is likely to proceed through a radical process. In the presence of DDQ, 1,3-diphenylpropene might generate an allylic cation intermediate, which will react further with nitrogen nucleophiles to produce the coupled products. The allylic cation can afford isomeric products by reaction at the α - and γ -positions when an asymmetrical 1,3-diarylpropene is used as substrate.

In summary, we have developed an amidation of 1,3-diarylallylic compounds with nitrogen nucleophiles mediated by a catalytic amount of DDQ and BPO with molecular oxygen as the terminal oxidant. It may be used with carboxamides, sulfonamides, carbamates and anilines. Moderate to good yields are obtained.

Experimental

Column chromatography was carried out on silica gel (200–300 mesh). ¹H NMR spectra were recorded on a Bruker AMX-500 MHz instrument and the chemical shifts are reported in parts per million (δ) relative to the internal standard TMS (0 ppm) for CDCl₃. The coupling constants, *J*, are reported in Hertz (Hz). Low (high) resolution MS was obtained using ESI ionisation on an Agilent 6210 TOF LC/MS instrument. Melting points are uncorrected. All reagents were weighed and handled in air at room temperature and all reactions were performed without exclusion of air or moisture.

Oxidative coupling reaction of 1,3-diarylpropenes **1**; general procedure DDQ (20 mol%, 9.08 mg, 0.04 mmol) and BPO (20 mol%, 9.69 mg, 0.04 mmol) were added to a mixture of 1,3-diarylpropene **1** (0.24 mmol) and substrate **2** (0.2 mmol) in CH₃NO₂ (1 mL). The reaction was carried out at 100 °C under an oxygen atmosphere (oxygen balloon) for 24 h. The resulting mixture was purified by flash column chromatography on silica gel (petroleum ether:ethyl acetate = 10:1–20:1) to give the desired pure product **3a–k**.

$\label{eq:procedure} \textit{Procedure for the preparation of compounds 3l-p}$

A mixture of DDQ (20 mol%, 9.08 mg, 0.04 mmol), BPO (20 mol%, 9.69 mg, 0.04 mmol) and 1,3-diarylpropene (0.2 mmol) in 1,4-dioxane (1 mL) was stirred for about 5 minutes. Aniline (0.55 mmol) in 1,4-dioxane (0.5 mL) was added to the mixture. Stirring was continued under an oxygen atmosphere (oxygen balloon) for 24 h. The resulting mixture was purified by flash column chromatography on silica gel (petroleum ether–ethyl acetate) to give the desired named products **31–p**. (E)-N-(*1*,3-Diphenylallyl)benzamide (**3a**): White solid; m.p.

162–163 °C (lit.¹⁵ 163–164 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.85 (d, J = 10.8 Hz, 2H), 7.53 (t, J = 7.4 Hz, 1H), 7.47–7.25 (m, 12H), 6.66–6.62 (m, 2H), 6.47 (dd, J = 6.1, 15.9 Hz, 1H), 6.06–6.04 (m, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 166.5, 141.0, 136.5, 134.5, 131.9, 131.6, 128.9, 128.63, 128.59, 127.85, 127.79, 127.2, 127.1, 126.6, 55.3.

(E)-N-(*1*,3-Diphenylallyl)-4-methylbenzamide (**3b**): White solid; m.p. 156–157 °C (lit.²¹ 156–158 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.75 (d, *J* = 8.1 Hz, 2H), 7.45–7.38 (m, 6H), 7.35–7.31 (m, 3H), 7.29–7.24 (m, 3H), 6.64–6.55 (m, 2H), 6.46 (dd, *J* = 6.1, 15.9 Hz, 1H), 6.06–6.03 (m, 1H), 2.41 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 166.5, 142.1, 141.0, 136.5, 131.7, 131.5, 129.3, 128.9, 128.87, 128.6, 127.8, 127.7, 127.3, 127.1, 126.6, 55.2, 21.5.

(E)-N-(*1*,3-Diphenylallyl)-4-methoxybenzamide (**3c**): White solid; m.p. 149–151 °C (lit.²² 150–151 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.82



Scheme 1 Possible mechanism of the coupling reaction.

(d, J = 8.9 Hz, 2H), 7.44–7.38 (m, 6H), 7.34–7.24 (m, 4H), 6.95–6.93 (m, 2H), 6.64–6.61 (m, 1H), 6.53–6.52 (m, 1H), 6.46 (dd, J = 6.1, 15.9 Hz, 1H), 6.04–6.02 (m, 1H), 3.86 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 166.1, 162.3, 141.1, 136.5, 131.6, 129.1, 129.0, 128.8, 128.5, 127.7, 127.6, 127.2, 126.59, 126.57, 113.7, 55.4, 55.2.

(E)-N-(*1*,3-Diphenylallyl)-4-fluorobenzamide (**3d**): White solid; m.p. 139–140 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.86–7.83 (m, 2H), 7.43–7.25 (m, 10H), 7.07 (t, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.0 Hz, 1H), 6.60 (d, *J* = 15.9 Hz, 1H), 6.44 (dd, *J* = 6.2, 15.9 Hz, 1H), 6.02–5.99 (m, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 165.7, 165.5, 163.7, 140.7, 136.3, 131.8, 130.43, 130.41, 129.5, 129.4, 128.8, 128.61, 128.55, 127.9, 127.8, 127.2, 126.5, 115.6, 115.5, 55.4; HRMS (ESI) calcd for C₂₂H₁₈FNNaO [M + Na⁺]: 354.1265; found: 354.1271.

(E)-N-(*1*,3-*Diphenylallyl*)-3-*chlorobenzamide* (**3e**): White solid; m.p. 149–150 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.82 (s, 1H), 7.70 (d, *J* = 7.6 Hz, 1H), 7.49–7.25 (m, 12H), 6.72 (d, *J* = 7.6 Hz, 1H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.44 (dd, *J* = 6.1, 15.9 Hz, 1H), 6.02–5.99 (m, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 165.2, 140.6, 136.3, 136.1, 134.8, 132.0, 131.6, 129.9, 128.9, 128.6, 128.4, 127.9, 127.88, 127.4, 127.2, 126.6, 125.2, 55.4; HRMS (ESI) calcd for C₂₂H₁₈CINNaO [M + Na⁺]: 370.0969; found: 370.0961.

(E)-N-(1,3-Diphenylallyl)thiophene-2-carboxamide (**3f**): White solid; m.p. 182–184 °C; ¹H NMR (500 MHz, CDCl₃): δ 7.58–7.57 (m, 1H), 7.51–7.50 (m, 1H), 7.45–7.39 (m, 6H), 7.35–7.31 (m, 3H), 7.28–7.25 (m, 1H), 7.10–7.09 (m, 1H), 6.63 (d, *J* = 16.0 Hz, 1H), 6.47–6.41 (m, 2H), 6.02–5.99 (m, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 161.0, 140.7, 138.7, 136.4, 133.6, 132.0, 130.2, 128.9, 128.59, 128.55, 128.46, 128.3, 127.9, 127.86, 127.7, 127.3, 126.6, 55.2; HRMS (ESI) calcd for C₂₀H₁₇NNaOS: [M + Na⁺]: 342.0923; found: 342.0924.

(E)-*1*-(*1*,*3*-*Diphenylallyl*)*pyrrolidin*-2-*one* (**3g**)²³: Colourless oil; ¹H NMR (500 MHz, CDCl₃): δ 7.48–7.28 (m, 10H), 6.64 (d, *J* = 15.9 Hz, 1H), 6.47 (dd, *J* = 6.8, 15.9 Hz, 1H), 6.12 (d, *J* = 6.7 Hz, 1H), 3.48–3.43 (m, 1H), 3.18–3.13 (m, 1H), 2.53–2.49 (m, 2H), 2.10–1.99 (m, 2H); ¹³C NMR (125 MHz, CDCl₃): δ 174.8, 138.8, 136.6, 133.6, 130.0, 128.7, 128.6, 128.3, 127.9, 127.8, 129.7, 126.6, 125.5, 56.3, 43.5, 31.2, 18.2.

(E)-N-[*1*-(*4*-Bromophenyl)-3-phenylallyl]-4-methylbenzamide (**3h**): As a mixture of α- and γ-isomers; white solid; m.p. 151–153 °C; 'H NMR (500 MHz, CDCl₃): δ 7.74 (d, *J* = 7.8 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.44–7.23 (m, 10H), 6.68–6.53 (m, 2H), 6.46–6.38 (m, 1H), 6.01–5.95 (m, 1H), 2.41 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 166.62, 166.58, 142.23, 142.18, 140.7, 140.1, 136.2, 135.5, 132.3, 131.8, 131.7, 131.3, 131.2, 130.4, 129.8, 129.3, 128.95, 128.92, 128.6, 128.3, 128.1, 128.0, 127.9, 127.3, 127.2, 127.1, 126.6, 121.6, 55.2, 54.8, 21.5; HRMS (ESI) calcd for $C_{23}H_{21}BrNO [M + H^+]$: 406.0807; found: 406.0818.

(E)-N-[*I*-(*4-Methoxylphenyl*)-*3-phenylallyl*]-*4-methylbenzamide* (**3i**): As a mixture of α- and γ-isomers; pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.75–7.73 (m, 2H), 7.44–7.24 (m, 9H), 6.93–6.91 (m, 1H), 6.85 (d, *J* = 8.7 Hz, 1H), 6.62–6.52 (m, 2H), 6.45 (dd, *J* = 5.8, 15.9 Hz, 0.5 × 1H), 6.31 (dd, *J* = 6.2, 15.9 Hz, 0.5 × 1H), 6.02–5.97 (m, 1H), 3.82, 3.81 (ss, 3H), 2.41 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): 166.4, 166.39, 159.4, 159.1, 142.1, 141.2, 136.5, 133.0, 131.5, 131.3, 131.25, 129.3, 129.1, 128.8, 128.6, 128.5, 127.8, 127.7, 127.65, 127.2, 127.1, 126.7, 126.6, 114.2, 114.0, 58.4, 55.34, 55.30, 55.22, 54.6, 21.5, 18.4; HRMS (ESI): calcd for $C_{24}H_{24}NO_2$ [M + H⁺]: 358.1807; found: 358.1807.

(E)-N-(*1*,3-Diphenylallyl)benzenesulfonamide (**3j**): White solid; m.p. 109–111 °C (lit.²⁴ 111–114 °C); ¹H NMR (500 MHz, (CD₃)₂CO): δ 7.83 (d, *J* = 7.3 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.48–7.43 (m, 2H), 7.34 (d, *J* = 7.3 Hz, 2H), 7.29–7.21 (m, 9H), 6.40 (d, *J* = 15.8 Hz, 1H), 6.26 (dd, *J* = 7.0, 15.8 Hz, 1H), 5.21–5.18 (m, 1H); ¹³C NMR (CD₃)₂CO): δ 143.1, 141.8, 137.6, 132.9, 132.1, 130.1, 129.7, 129.4, 129.3, 128.6, 128.2, 128.0, 127.9, 127.3, 60.7.

(E)-N-(*1*,3-*Diphenylallyl*)-4-methylbenzenesulfonamide (**3k**): White solid; m.p. 138–140 °C (lit.¹⁵ 140–141 °C); ¹H NMR (500 MHz, CDCl₃): δ 7.68 (d, *J* = 8.3 Hz, 2H), 7.29–7.15 (m, 12H), 6.36 (d, *J* = 15.8 Hz, 1H), 6.09 (dd, *J* = 6.8, 15.8 Hz, 1H), 5.23 (d, *J* = 7.3 Hz, 1H), 5.14–5.12 (m, 1H), 2.33 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 143.2, 139.7, 137.7, 136.1, 132.1, 129.4, 128.7, 128.4, 128.2, 127.9, 127.8, 127.3, 127.1, 126.5, 59.8, 21.4.

(E)-N-(*1*,3-*Diphenylallyl*)*aniline* (**3**):¹⁸ Pale yellow oil; ¹H NMR (500 MHz, CDCl₂): δ 7.51 (d, *J* = 7.6 Hz, 2H). 7.45–7.42 (m, 4H), 7.38–7.35 (m,

3H), 7.31–7.28 (m, 1H), 7.24–7.21 (m, 2H), 6.80–6.77 (m, 1H), 6.72–6.68 (m, 3H), 6.47 (dd, J = 6.2, 15.9 Hz, 1H), 5.16 (d, J = 6.1 Hz, 1H), 4.19 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 146.2, 141.1, 135.6, 130.1, 129.7, 128.1, 127.8, 127.5, 126.6, 126.5, 126.2, 125.5, 116.7, 112.6, 59.7.

(E)-4-Bromo-N-(1, 3-diphenylallyl)aniline (**3m**):¹⁸ Pale yellow oil; ¹H NMR (500 MHz, CDCl₃): δ 7.46–7.40 (m, 6H), 7.36–7.33 (m, 3H), 7.29–7.24 (m, 3H), 6.64 (d, *J* = 15.9 Hz, 1H), 6.56–6.53 (m, 2H), 6.41 (dd, *J* = 6.2, 15.9 Hz, 1H), 5.08 (d, *J* = 6.1 Hz, 1H), 4.19 (s, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 145.1, 140.5, 135.4, 130.8, 130.4, 129.1, 127.9, 127.6, 126.8, 126.7, 126.1, 125.5, 114.2, 108.4, 59.6.

(E)-N-(*1*,3-*Diphenylallyl*)*methylcarbamate* (**3n**): White solid; m.p. 104–105 °C (lit.²³ 104–106 °C); ¹H NMR (500 MHz, (CD₃)₂CO): δ 7.44 (d, *J* = 8.0 Hz, 4H), 7.38–7.23 (m, 6H), 6.95 (s, 1H), 6.64 (d, *J* = 16.1 Hz, 1H), 6.49 (dd, *J* = 6.6, 15.9 Hz, 1H), 5.93–5.50 (m, 1H), 3.61 (s, 3H); ¹³C NMR (125 MHz, (CD₃)₂CO): δ 157.1, 143.3, 137.9, 131.4, 131.1, 129.5, 129.4, 128.5, 128.1, 127.9, 127.4, 58.0, 52.1.

(E)-N-(*1*,3-*Diphenylallyl*)-tert-*butylcarbamate* (**30**): White solid, m.p. 114–116 °C (lit.¹⁵ 117 °C); ¹H NMR (500 MHz, (CD₃)₂CO): δ 7.44 (d, *J* = 8.0 Hz, 4H), 7.38–7.31 (m, 4H), 7.28–7.23 (m, 2H), 6.68(s, 1H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.48 (dd, *J* = 6.8, 15.9 Hz, 1H), 5.48 (s, 1H), 1.42 (s, 9H); ¹³C NMR (125 MHz, (CD₃)₂CO): δ 155.9, 143.6, 138.0, 131.4, 131.2, 129.5, 129.3, 128.4, 127.93, 127.86, 127.3, 79.1, 57.5, 28.7.

(E)-N-(*1*,3-*Diphenylallyl*)*benzylcarbamate* (**3p**): White solid, m.p. 107–109 °C (lit.¹⁵ 109 °C); ¹H NMR (500 MHz, (CD₃)₂CO): δ 7.47–7.23 (m, 15H), 7.09 (d, *J* = 6.4 Hz, 1H), 6.65 (d, *J* = 15.9 Hz, 1H), 6.50 (dd, *J* = 6.7, 15.9 Hz, 1H), 5.56–5.53 (m, 1H), 5.14–5.07 (m, 2H); ¹³C NMR (125 MHz, (CD₃)₂CO): δ 156.6, 143.2, 138.4, 137.9, 131.5, 131.0, 129.5, 129.4, 129.2, 128.7, 128.6, 128.5, 128.1, 127.9, 127.4, 66.8, 58.1.

Electronic Supplementary Information

The ESI (NMR spectra for all compounds) is available through: stl.publisher.ingentaconnect.com/content/stl/jcr/supp-data

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