

Molecular iodine-catalysed amidation reaction of secondary benzylic and allylic alcohols with carboxamides or sulfonamides

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A highly efficient method for the C–N bond formation *via* 2 mol% of molecular iodine-catalysed amidation reaction of benzylic and allylic alcohols with carboxamides or sulfonamides in MeCN is described, giving the corresponding substituted amides and allylic amides in moderate to excellent yields. The significant features of the procedure include mild and metal-free reaction conditions, operational simplicity, inexpensive reagents, short reaction time, and good yields.

Keywords: molecular iodine, amidation reaction, C–N bond formation

C–N Bond formation is a useful synthetic method for making substituted amines.¹ It is important to develop simple, efficient and environmentally friendly C–N bond forming reactions. Methods for the construction of C–N bond include transition metal catalysed substitution reactions of alcohols or halides with nitrogen nucleophiles,^{2,3} and transition metal catalysed C–H amination reactions.⁴ Among the various strategies, the direct catalytic substitution of alcohols with nitrogen nucleophiles is a salt-free, highly atom-economic, environmentally friendly synthetic method with water as the only byproduct. Although a number of direct substitution reactions of alcohols with amines catalysed by transition metals have been reported,^{5,6} the use of weak nitrogen nucleophiles such as readily available amides are uncommon and require harsh conditions.

A few Lewis acid catalysed amidation reactions of alcohols with amides have been described using NaAuCl₄,⁷ H-Montmorillonite,⁸ MoCl₅,⁹ Bi(OTf)₃-KPF₆,¹⁰ AgOTf¹¹ and FeCl₃.¹² However, the use of expensive and toxic metal catalysts and anhydrous reaction condition reduced the practical application of these methods. Therefore, development of a more practical and economical method for direct substitution of benzylic or allylic alcohols by amides is highly desirable for the synthesis of corresponding substituted benzylic and allylic amides.

Recently, molecular iodine has received considerable attention as an inexpensive and readily available catalyst for various organic transformations due to its moderate Lewis acidity and water-tolerance.^{12–19} In continuation of our studies of molecular iodine-catalysed organic reactions,^{20–22} we now describe a C–N bond formation reaction *via* molecular iodine-

catalysed amidation of secondary benzylic and allylic alcohols with carboxamides and *p*-toluenesulfonamide.

The selected model reaction was carried out with diphenylmethanol (**1a**) and benzamide (**2a**) (Scheme 1). We examined several organic solvents. These were commercially available and were used without further purification or drying (Table 1). We found that a remarkable solvent effect existed in our iodine-catalysed reaction. Only polar solvent such as nitromethane and acetonitrile were good solvents giving a high yield of the corresponding substituted amide (**3a**) (Table 1, entries 3 and 4). Apolar solvents such as THF and dioxane did not give a product (Table 1, entries 1 and 2). Furthermore, the reaction time and the catalyst concentration were reduced to 2 h and 2 mol%, respectively (Table 1, entries 4–8).

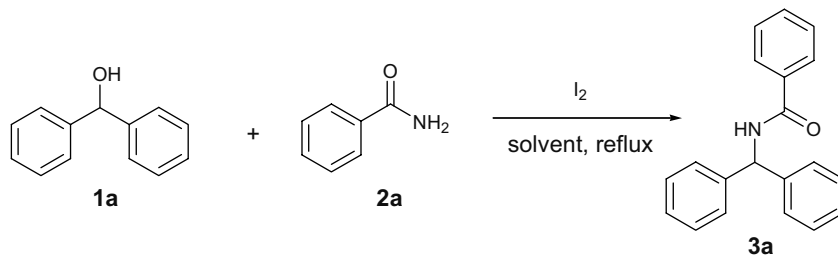
Table 1 Optimisation of reaction conditions

Entry	Catalyst/mol%	Solvent ^b	Time/h	Yield/% ^c
1	5	1, 4-dioxane	5.0	No reaction
2	5	THF	5.0	No reaction
3	5	MeNO ₂	1.0	97
4	5	MeCN	1.0	97
5	0	MeCN	5.0	No reaction
6	2	MeCN	2.0	98
7	10	MeCN	1.5	98
8	20	MeCN	1.0	98

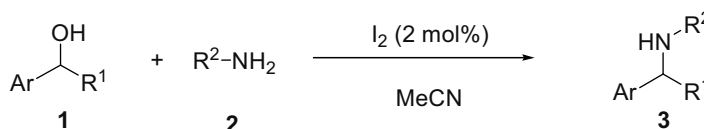
^aReaction condition: diphenylmethanol **1a** (1 mmol), benzamide **2a** (1 mmol), I₂ and solvent (4 mL), under reflux condition.

^bSolvents were commercially available and used without further purification or drying.

^cIsolated yield.



Scheme 1




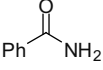
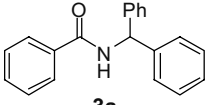
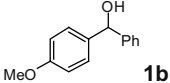
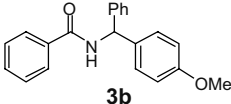

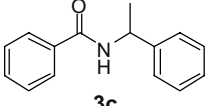
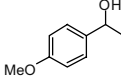
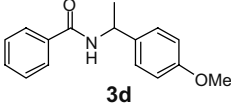
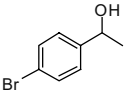
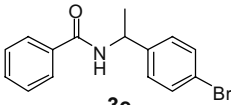
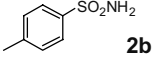
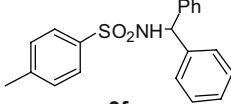
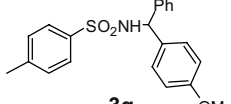
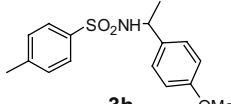
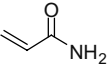
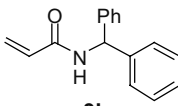
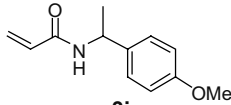

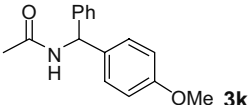
Scheme 2

Using the optimised reaction conditions, we examined a number of secondary benzylic alcohols **1a–e** as well as amide **2a–d** (Scheme 2). We obtained the corresponding substituted amides **3a–k** in 61–98% yields (Table 2). The reaction occurred easily at room temperature when the benzylic alcohols possessed electron-donating groups such as methoxy (Table 2, entries 2, 4, 7, 8 and 10). Aliphatic primary amides such as

acrylamide and acetamide also underwent smooth amidation with benzylic alcohols and gave the desired products (Table 2, entries 9–11). Although this reaction was highly efficient for secondary benzylic alcohols, benzyl alcohol itself did not react under these conditions even on prolonged heating.

Furthermore, we carried out this reaction with allylic alcohols and amides using the established procedure, to

Table 2 Iodine-catalysed amidation reaction of secondary benzylic alcohols with amide^a

Entry	Alcohols	Amides	Products	Time/h	Yield/% ^c
1	 1a	 2a	 3a	2.0	98
2	 1b		 3b	1.0	97
3	 1c		 3c	4.0	61
4	 1d		 3d	2.0	85 ^d
5	 1e		 3e	5.0	65
6	 2b		 3f	2.0	98
7	2b		 3g	1.0	97
8	2b		 3h	2.0	88 ^d
9	 2c		 3i	3.0	76
10	2c		 3j	5.0	68 ^d
11	 2d		 3k	4.0	85

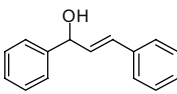
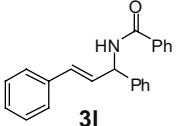
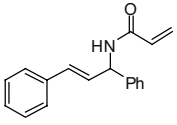
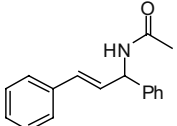
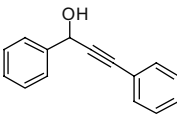
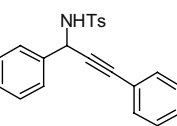
^aReaction condition: benzylic alcohol **1** (2 mmol), amide **2** (2 mmol), I₂ (2 mol%) and MeCN (8 mL), under reflux condition.

^bUsing MeNO₂ as solvent.

^cIsolated yield.

^dAt room temperature.

Table 3 Iodine-catalysed amidation reaction of allylic and propargyl alcohols with amide^a

Entry	Alcohols	Amides	Products	Time/h	Yield/% ^b
1		2a		1.0	98
2	1f	2c		1.0	92
3	1f	2d		2.0	90 ^c
4		2b		3.0	55 ^c

^aReaction conditions: allylic or propargyl alcohols **1** (2 mmol), amide (2 mmol), I₂ (2 mol%), MeCN (8 mL), r. t.

^bIsolated yield.

^cUnder reflux condition.

obtain the corresponding allylic amides (Table 3, entries 1–3). All the reactions proceeded smoothly in the presence of 2 mol% iodine in acetonitrile in good yields. We also carried out this reaction with propargyl alcohol **1g** and amide, obtaining the corresponding propargyl amide **3o** in moderate yield (Table 3, entry 4).

Following literature precedent,^{23–26} we consider that iodine catalyses the reaction as a mild Lewis acid via a possible S_N1 mechanism (Scheme 3). A benzyl carbocation might be formed from benzyl alcohol in the presence of molecular iodine, followed by nucleophilic attack by the amide to afford the corresponding substituted amides.

In summary, we have developed a simple and efficient C–N bond formation reaction *via* a molecular iodine-catalysed amidation reaction of secondary benzylic and allylic alcohols with carboxamides or *p*-toluenesulfonamide in acetonitrile. Using this method, propargyl alcohols can also react with amide, giving the corresponding propargyl amides. The notable features of the procedure include mild and metal-free reaction conditions, operational simplicity, use of a catalytic amount of molecular iodine (2 mol%), short reaction time, and good yields.

Experimental

IR spectra were determined as KBr pellets on a Bruker model 470 spectrophotometer. ¹H NMR and ¹³C NMR spectra were recorded using a Bruker 400 MHz spectrometer in CDCl₃ with tetramethylsilane

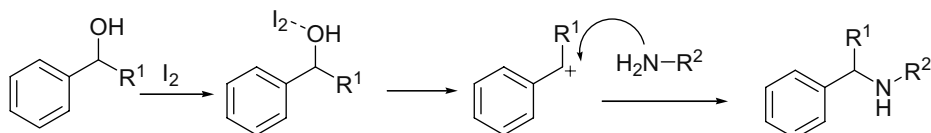
as internal standard. MS was obtained using ESI ionisation. Melting points were obtained on a microscopical instrument and uncorrected. All the products have been previously described.^{7, 12, 27–32}

General procedure for preparation of **3a–o**

A mixture of benzylic or allylic alcohol (2 mmol), amide (2 mmol) and I₂ (2 mol%) in the MeCN (8 mL) was stirred at room temperature or under reflux condition for the appropriate time. After the reaction was completed, the reaction mixture was quenched by 10% aq Na₂S₂O₃ solution (10 mL) and extracted with ethyl acetate (2 × 20 mL). The combined organic layers were dried over Na₂SO₄ and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel column with hexane–EtOAc (4:1) to obtain the corresponding amide product. The spectra data of the compounds **3a–o** are as follows.

N-Benzhydrylbenzamide (**3a**): M.p. 168–169 °C (lit.¹² 170 °C); ¹H NMR (400 MHz, CDCl₃) δ 6.44 (d, 1H, *J* = 7.6 Hz), 6.74 (d, 1H, *J* = 7.2 Hz), 7.24–7.35 (m, 10H), 7.38–7.42 (m, 2H), 7.47–7.52 (m, 1H), 7.80 (d, 2H, *J* = 7.6 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 57.66, 127.3 (2 C), 127.7 (4 C), 127.8 (2 C), 128.8 (2 C), 129.0 (4 C), 131.9, 134.4, 141.7 (2 C), 166.8 ppm; IR (KBr): 3312, 1638, 1521, 1496, 706 cm⁻¹. MS (ESI): *m/z* = 288 ([M + H]⁺).

N-((4-Methoxyphenyl)(phenyl)methyl)benzamide (**3b**): M.p. 183–184 °C (lit.²⁷ 180–181 °C); ¹H NMR (400 MHz, CDCl₃) δ 3.77 (s, 3H), 6.38 (d, 1H, *J* = 8.0 Hz), 6.68 (brs, 1H), 6.85 (d, 2H, *J* = 8.0 Hz), 7.19 (d, 2H, *J* = 8.4 Hz), 7.24–7.49 (m, 8H), 7.79 (d, 2H, *J* = 7.2 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 55.53, 57.13, 114.4 (2 C), 127.3 (2 C), 127.6 (2 C), 127.7, 128.8 (2 C), 128.9 (2 C), 129.0 (2 C), 131.9, 133.9, 134.5, 141.9, 159.2, 166.8 ppm; IR (KBr): 3306, 1631, 1540, 1509, 1247, 1175, 1032, 700 cm⁻¹. MS (ESI): *m/z* = 318 ([M + H]⁺).



Scheme 3

N-(1-Phenylethyl)benzamide (**3c**): M.p. 118–120°C (lit.²⁸ 119–120°C); ¹H NMR (400 MHz, CDCl₃) δ 1.61 (d, 3 H, *J* = 6.4 Hz), 5.34 (m, 1 H), 6.45 (brs, 1 H), 7.28 (m, 1 H), 7.33–7.43 (m, 6 H), 7.50 (m, 1 H), 7.77 (d, 2 H, *J* = 7.2 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.95, 49.47, 126.5 (2 C), 127.2 (2 C), 127.7, 128.8 (2 C), 129.0 (2 C), 131.7, 134.8, 143.4, 166.8 ppm; IR (KBr): 3357, 2974, 1634, 1518, 1489, 1323, 700 cm⁻¹. MS (ESI): *m/z* = 226 ([M + H]⁺).

N-(1-(4-Methoxyphenyl)ethyl)benzamide (**3d**): M.p. 112–114°C (lit.²⁷ 113–114°C); ¹H NMR (400 MHz, CDCl₃) δ 1.59 (d, 3 H, *J* = 7.2), 3.80 (s, 3 H), 5.30 (m, 1 H), 6.42 (brs, 1 H), 6.89 (d, 2 H, *J* = 8.4 Hz), 7.32 (d, 2 H, *J* = 8.4 Hz), 7.41–7.50 (m, 3 H), 7.77 (d, 2 H, *J* = 7.6 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.82, 48.87, 55.52, 114.3 (2 C), 127.2 (2 C), 127.7 (2 C), 128.7 (2 C), 131.6, 134.9, 135.5, 159.1, 166.8 ppm; IR (KBr): 3309, 2974, 1635, 1545, 1515, 1253, 832, 695 cm⁻¹. MS (ESI): *m/z* = 256 ([M + H]⁺).

N-(1-(4-Bromophenyl)ethyl)benzamide (**3e**): M.p. 119–121°C (lit.²⁷ 123–124°C); ¹H NMR (400 MHz, CDCl₃) δ 1.55 (d, 3 H, *J* = 6.8), 5.25 (dq, 1 H, *J* = 6.4, 6.8 Hz), 6.38 (d, 1 H, *J* = 6.4 Hz), 7.23 (d, 2 H, *J* = 8.4 Hz), 7.38–7.48 (m, 5 H), 7.74 (d, 2 H, *J* = 7.6 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.95, 48.96, 121.5, 127.2 (2 C), 128.2 (2 C), 128.8 (2 C), 131.8, 132.0 (2 C), 134.6, 142.6, 166.9 ppm; IR (KBr): 3350, 2983, 1635, 1526, 1488, 1316, 1099, 1011, 827 cm⁻¹. MS (ESI): *m/z* = 304 ([M + H]⁺).

N-Benzhydryl-4-methylbenzenesulfonamide (**3f**): M.p. 155–156°C (lit.⁷ 157°C); ¹H NMR (400 MHz, CDCl₃) δ 2.38 (s, 3 H), 5.28 (d, 1 H, *J* = 7.2 Hz), 5.58 (d, 2 H, *J* = 7.2 Hz), 7.10–7.15 (m, 6 H), 7.20–7.22 (m, 6 H), 7.57 (d, 2 H, *J* = 8.0 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.70, 61.57, 127.4 (2 C), 127.6 (4 C), 127.8 (2 C), 128.8 (4 C), 129.6 (2 C), 137.6, 140.8 (2 C), 143.4 ppm; IR (KBr): 3249, 1599, 1495, 1452, 1315, 1161, 1096, 811, 752 cm⁻¹. MS (ESI): *m/z* = 338 ([M + H]⁺).

N-((4-Methoxyphenyl)(phenyl)methyl)-4-methylbenzenesulfonamide (**3g**): M.p. 129–130°C (lit.²⁷ 129–130°C); ¹H NMR (400 MHz, CDCl₃) δ 2.38 (s, 3 H), 2.76 (s, 3 H), 5.22 (d, 1 H, *J* = 6.8 Hz), 5.53 (d, 2 H, *J* = 6.8 Hz), 6.74 (d, 2 H, *J* = 8.8 Hz), 7.01 (d, 2 H, *J* = 8.0 Hz), 7.10–7.15 (m, 4 H), 7.19–7.22 (m, 3 H), 7.56 (d, 2 H, *J* = 8.0 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.70, 55.49, 61.07, 114.1 (2 C), 127.4 (2 C), 127.5 (2 C), 127.7, 128.7 (2 C), 128.9 (2 C), 129.6 (2 C), 133.0, 137.7, 141.0, 143.3, 159.2 ppm; IR (KBr): 3240, 1610, 1511, 1448, 1433, 1323, 1252, 1159, 1048 cm⁻¹. MS (ESI): *m/z* = 368 ([M + H]⁺).

N-(1-(4-Methoxyphenyl)ethyl)-4-methylbenzenesulfonamide (**3h**): M.p. 88–89°C (lit.²⁹ 86–88°C); ¹H NMR (400 MHz, CDCl₃) δ 1.40 (d, 3 H, *J* = 6.8 Hz), 2.40 (s, 3 H), 3.76 (s, 3 H), 4.42 (m, 1 H), 4.96 (d, 1 H, *J* = 7.2 Hz), 6.72 (d, 2 H, *J* = 8.4 Hz), 7.02 (d, 2 H, *J* = 8.4 Hz), 7.20 (d, 2 H, *J* = 8.4 Hz), 7.63 (d, 2 H, *J* = 8.4 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.69, 23.64, 55.33, 55.49, 114.1 (2 C), 127.4 (2 C), 127.6 (2 C), 129.7 (2 C), 134.4, 138.0, 143.3, 159.2 ppm; IR (KBr): 3275, 2960, 1614, 1513, 1321, 1247, 1178, 1160, 1090, 1031, 957 cm⁻¹. MS (ESI): *m/z* = 306 ([M + H]⁺).

N-Benzhydrylacrylamide (**3i**): 175–177°C (lit.³⁰ 177–178°C); ¹H NMR (400 MHz, CDCl₃) δ 5.65 (d, 1 H, *J* = 10.4 Hz), 6.16 (dd, 1 H, *J* = 16.8, 10.4 Hz), 6.23–6.26 (m, 3 H), 7.22–7.35 (m, 10 H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 57.28, 127.4, 127.6 (4 C), 127.7 (2 C), 128.9 (4 C), 130.8, 141.2 (2 C), 164.8 ppm; IR (KBr): 3279, 3026, 1651, 1619, 1536, 1494, 1408, 1232, 987 cm⁻¹. MS (ESI): *m/z* = 238 ([M + H]⁺).

N-(1-(4-Methoxyphenyl)ethyl)acrylamide (**3j**): 104–106°C (lit.³¹ 107.5°C); ¹H NMR (400 MHz, CDCl₃) δ 1.50 (d, 3 H, *J* = 7.2 Hz), 3.78 (s, 3 H), 5.15 (m, 1 H), 5.61 (d, 1 H, *J* = 10.0 Hz), 5.89 (brs, 1 H), 6.07 (dd, 1 H, *J* = 18.8, 10.0 Hz), 6.27 (d, 1 H, *J* = 16.8 Hz), 6.86 (d, 2 H, *J* = 8.4 Hz), 7.25 (d, 2 H, *J* = 8.8 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.70, 48.46, 55.53, 114.3 (2 C), 126.7, 127.7 (2 C), 131.2, 135.3, 159.1, 164.7 ppm; IR (KBr): 3286, 2970, 1655, 1625, 1546, 1515, 1411, 1246, 1180, 828 cm⁻¹. MS (ESI): *m/z* = 206 ([M + H]⁺).

N-((4-Methoxyphenyl)(phenyl)methyl)acetamide (**3k**): 159–160°C (lit.²⁷ 159–160°C); ¹H NMR (400 MHz, CDCl₃) δ 2.03 (s, 3 H), 3.79 (s, 3 H), 6.20 (m, 2 H), 6.85 (d, 2 H, *J* = 9.2 Hz), 7.14 (d, 2 H, *J* = 8.4 Hz), 7.23–7.32 (m, 5 H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 23.55, 55.51, 56.63, 114.2 (2 C), 127.5 (2 C), 127.6 (2 C), 128.8 (2 C), 128.9, 134.0, 142.0, 159.1, 169.3 ppm; IR (KBr): 3310, 3061, 1638, 1545, 1513, 1448, 1370, 1249, 1185, 1031 cm⁻¹. MS (ESI): *m/z* = 256 ([M + H]⁺).

(*E*)-*N*-(1,3-Diphenylallyl)benzamide (**3l**): 156–157°C (lit.²⁷ 157–158°C); ¹H NMR (400 MHz, CDCl₃) δ 6.04 (m, 1 H), 6.46 (dd, 1 H, *J* = 16.0, 6.4 Hz), 6.63 (d, 1 H, *J* = 16.0 Hz), 6.64 (brs, 1 H), 7.25–7.52 (m, 14 H), 7.84 (d, 2 H, *J* = 7.6 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 55.47, 126.8 (2 C), 127.3 (2 C), 127.5 (2 C), 128.0, 128.1, 128.8 (2 C), 128.9 (2 C), 129.0, 129.1 (2 C), 131.9, 132.0, 134.6, 136.6, 141.1, 166.7 ppm; IR (KBr): 3351, 3031, 1633, 1516, 1488, 1314, 967, 749 cm⁻¹. MS (ESI): *m/z* = 314 ([M + H]⁺).

(*E*)-*N*-(1,3-Diphenylallyl)acrylamide (**3m**): M.p. 121–122°C (lit.¹² 123°C); ¹H NMR (400 MHz, CDCl₃) δ 5.65 (d, 1 H, *J* = 10.4 Hz), 5.88 (m, 1 H), 6.15 (brs, 1 H), 6.20 (dd, 1 H, *J* = 16.0, 10.4 Hz), 6.32 (d, 1 H, *J* = 16.0 Hz), 6.35 (dd, 1 H, *J* = 16.0, 6.0 Hz), 6.54 (d, 1 H, *J* = 16.0 Hz), 7.19–7.37 (m, 10 H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 55.07, 126.8 (2 C), 127.3, 127.5 (2 C), 128.0, 128.1, 128.8 (2 C), 128.9, 129.1 (2 C), 130.9, 131.9, 136.6, 140.9, 164.9 ppm; IR (KBr): 3269, 3029, 1656, 1619, 1541, 1494, 1449, 1408, 1235, 954, 749, 695 cm⁻¹. MS (ESI): *m/z* = 264 ([M + H]⁺).

(*E*)-*N*-(1,3-Diphenylallyl)acetamide (**3n**): 126–128°C (lit.²⁷ 126–127°C); ¹H NMR (400 MHz, CDCl₃) δ 2.05 (s, 3 H), 5.82 (m, 1 H), 6.03 (d, 1 H, *J* = 7.6 Hz), 6.34 (dd, 1 H, *J* = 16.0, 6.0 Hz), 6.54 (d, 1 H, *J* = 16.0 Hz), 7.20–7.38 (m, 10 H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 23.68, 55.03, 126.8 (2 C), 127.4 (2 C), 127.9, 128.0, 128.8 (2 C), 129.1 (2 C), 129.2, 131.6, 136.7, 141.1, 169.3 ppm; IR (KBr): 3292, 3030, 1640, 1535, 1493, 1449, 1371, 1308, 971, 747, 695 cm⁻¹. MS (ESI): *m/z* = 252 ([M + H]⁺).

N-(1,3-Diphenylprop-2-ynyl)-4-methylbenzenesulfonamide (**3o**): 184–185°C (lit.³² 186–188°C); ¹H NMR (400 MHz, CDCl₃) δ 2.32 (s, 3 H), 4.97 (d, 1 H, *J* = 9.2 Hz), 5.56 (d, 1 H, *J* = 9.2 Hz), 7.12 (d, 2 H, *J* = 7.2 Hz), 7.22–7.38 (m, 8 H), 7.56 (d, 2 H, *J* = 6.8 Hz), 7.82 (d, 2 H, *J* = 8.0 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 21.67, 50.04, 85.74, 86.95, 122.2, 127.6 (2 C), 127.8 (2 C), 128.4 (2 C), 128.7, 128.8, 129.0 (2 C), 129.8 (2 C), 131.8 (2 C), 137.65, 137.67, 143.8 ppm; IR (KBr): 3268, 1597, 1491, 1430, 1332, 1155, 1091, 1050, 760, 698 cm⁻¹. MS (ESI): *m/z* = 362 ([M + H]⁺).

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