# An Efficient Palladium-Catalyzed Route to *N*-Allylanilines by the Direct Use of Allyl Alcohols

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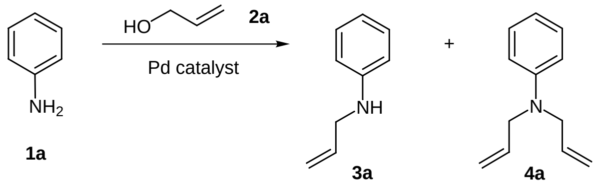
**Abstract:** The direct activation of C–O bonds in allyl alcohols by palladium complexes has been accelerated by carrying out the reactions in the presence of titanium(IV) isopropoxide and molecular sieves (4Å). *N*-Allylation of anilines to give mono- and diallylanilines directly using allyl alcohols has been realized by employing palladium catalysts.

**Key words:** palladium catalysis, *N*-allylation, anilines, allyl alcohol, π-allylpalladium complex

Allyl amines are an important class of compounds not only due to their utility as intermediates in organic synthesis[1] but also because of their physiological properties[2] and their presence in several natural products.[3] A number of synthetic methods for the preparation of allyl amines from alkene derivatives have been developed, but these require severe reaction conditions or several sequential reactions.[4] Palladium-catalyzed allylation is an established, efficient, and highly stereoselective method for the C–C, C–N, and C–O bond formation, which has been widely applied in organic synthesis.[5] The processes have been shown to proceed by attack of nucleophiles on intermediate $\eta^3$-allylpalladium(II) complexes generated by oxidative addition of allylic compounds to a Pd(0) complex.[6] Although halides,[6,7] acetates,[8] carbonates,[9] carbamates,[10] phosphates,[11] and related derivatives[12] of allyl alcohols have frequently been used as substrates, there have been only limited and sporadic reports dealing with the direct cleavage of the C–O bond in allyl alcohols on interaction with a transition metal complex.[13] Successful applications using allyl alcohols directly in catalytic processes are even more limited. This apparently stems from the poor capability of a non-activated hydroxyl to serve as a leaving group.[14] Itoh has reported that palladium-catalyzed nucleophilic substitution of allyl alcohols using zinc enolates can proceed efficiently in the presence of titanium(IV) alkoxides and LiCl;[15] the alkoxides appear to enhance the reactivity of allyl alcohols toward palladium(0) species. Anilines are useful compounds in organic synthesis.[5] We considered that, when anilines are employed as nucleophiles for the reaction, a convenient method to prepare *N*-allylanilines could be realized. We have examined the reaction of allyl alcohols with anilines in the presence of a palladium catalyst and titanium(IV) isopropoxide.

When a mixture of aniline (**1a**) and allyl alcohol (**2a**) was heated in the presence of catalytic amounts of Pd(OAc)$_2$ PPh$_3$ and Ti(OPr-*i*)$_4$ in benzene, *N*-allylaniline (**3a**) was formed (Scheme 1) in 22% yield (Entry 1, Table 1).



**Scheme 1**

The reaction is accompanied by formation of water. Addition of molecular sieves (4Å) for its removal, increased the yields of products **3a** and *N*,*N*-diallylaniline (**4a**) to 66 and 10%, respectively (Entry 2). Addition of molecular

**Table 1** Allylation of Aniline (**1a**) with Allyl Alcohol (**2a**)[a]

| Entry | Catalyst | Additive | Solvent | Yield (%)[e] (**3a**:**4a**) |
|---|---|---|---|---|
| 1[b] | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 22 (100:0) |
| 2 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 76 (87:13) |
| 3 | Pd(OAc)$_2$-PPh$_3$ | – | benzene | 0 |
| 4 | Pd(OAc)$_2$-PPh$_3$ | ET$_3$N | benzene | 0 |
| 5 | Pd(OAc)$_2$-PPh$_3$ | K$_2$CO$_3$ | benzene | 0 |
| 6[c] | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 43 (100:0) |
| 7[d] | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 78 (59:41) |
| 8 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | MeCN | 34 (82:18) |
| 9 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | THF | 36 (81:19) |
| 10 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | HMPA | 52 (83:17) |
| 11 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | DMF | 24 (100:0) |
| 12 | Pd(OAc)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | toluene | 61 (85:15) |
| 13 | Pd(OAc)$_2$ | Ti(OPr-*i*)$_4$ | benzene | 20 (75:25) |
| 14 | Pd(OAc)$_2$-dppe | Ti(OPr-*i*)$_4$ | benzene | 64 (88:12) |
| 15 | Pd(OAc)$_2$-dppp | Ti(OPr-*i*)$_4$ | benzene | 67 (88:12) |
| 16 | Pd(OAc)$_2$-dppb | Ti(OPr-*i*)$_4$ | benzene | 69 (91:9) |
| 17 | Pd(OCOCF$_3$)$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 57 (68:32) |
| 18 | Pd(OCOCF$_3$)$_2$-dppb | Ti(OPr-*i*)$_4$ | benzene | 68 (81:19) |
| 19 | Pd(PPh$_3$)$_4$ | Ti(OPr-*i*)$_4$ | benzene | 51 (90:10) |
| 20 | PdCl$_2$(MeCN)$_2$ | Ti(OPr-*i*)$_4$ | benzene | 70 (86:14) |
| 21 | PdCl$_2$-PPh$_3$ | Ti(OPr-*i*)$_4$ | benzene | 19 (79:21) |
| 22 | PdCl$_2$-dppb | Ti(OPr-*i*)$_4$ | benzene | 18 (100:0) |

[a] Reaction conditions: **1a** (10 mmol), **2a** (12 mmol), Pdcatalyst (0.1 mmol), ligand (0.4 mmol), additive (2.5 mmol) and molecular sieves 4Å (200 mg), solvent (15 mL) at 50 °C for 3 h.

[b] Without molecular sieves 4Å.

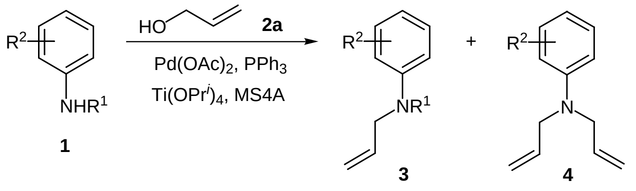
[c] 1 mmol of Ti(OPr-*i*)$_4$ was used.

[d] 5 mmol of Ti(OPr-*i*)$_4$ was used.

[e] Isolated yield.

sieves was needed to prevent catalyst deactivation which is probably due to water formed during the reaction. Without Ti(OPr-*i*)$_4$ (Entry 3) or when it was substituted by $Et_3N$ (Entry 4) or $K_2CO_3$ (Entry 5), allylation products were not obtained. Decreasing the amount of Ti(OPr-*i*)$_4$ afforded only **3a** in 43% yield (Entry 6). Conversely, increasing the amount of Ti(OPr-*i*)$_4$ did not increase the yield of products significantly (Entry 7). Six useful solvents are benzene, MeCN, THF, HMPA, DMF or toluene; benzene gave the best results (Entries 2 and 8–12). Without $PPh_3$ there was low yield of products (entry 13). The bidentate ligand dppe (Entry 14), dppp (Entry 15) or dppb (Entries 16 and 18) did not increase the yield of products. Among the palladium catalysts including $Pd(OCOCF_3)_2$ (Entries 17 and 18), $Pd(PPh_3)_4$ (Entry 19), $PdCl_2(MeCN)_2$ (Entry 20) and $PdCl_2$ (Entries 21 and 22), $Pd(OAc)_2$ was found to be superior (Entry 2).

Results for allylation of a number of anilines substituted by both electron-withdrawing and electron-donating groups **1b–o** with allyl alcohol (**2a**) using $Pd(OAc)_2$, $PPh_3$, Ti(OPr-*i*)$_4$ and molecular sieves (Scheme 2) are summarized in Table 2. All of the anilines examined underwent *N*-allylation smoothly to give the corresponding *N*-allylanilines in overall yields ranging from 48–88%. Allylation of *N*-allylaniline (**1o**) afforded *N*,*N*-diallylaniline (**4a**) in good yield. However, this result reveals that bisallylation could be formed.



Scheme 2

Treatment of 4-chloro-2-methylaniline (**1i**) with crotyl alcohol (**2b**) using $Pd(OAc)_2$, $PPh_3$, Ti(OPr-*i*)$_4$ and molecular sieves gave mixtures of regio- and stereoisomeric anilines **6** and **7** (Scheme 3) in 44 and 36% yields, respectively (Entry 1, Table 3). The 86:14 *E/Z* ratio of **6** was determined by $^1$H NMR and HETCOR spectroscopy, the $CH_2$ signal appeared at $\delta = 3.70$ for the *E*-isomer and at $\delta = 3.78$ for the *Z*-isomer. This stereochemistry was confirmed by the coupling constant of the vinylic protons for this major isomer ($J = 15.2$ Hz) being characteristic of *E*-stereochemistry. Since both regioisomeric alcohols **2b** and **2c** gave identical mixtures of the anilines **6** and **7** in similar ratios, the reaction is considered to proceed via π-allylpalladium intermediates (Entry 2, Table 3). Prior to the attack of the nucleophile, the π-allyl complex **5** can equilibrate between the *syn* and *anti* forms, thus giving rise to three possible products.

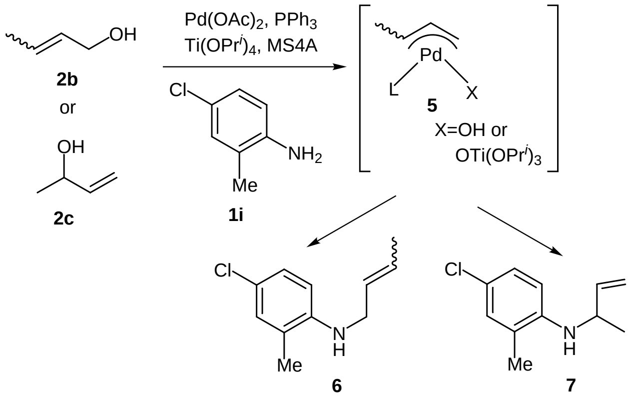
**Table 2** Allylation of Anilines **1b–o** with Allyl Alcohol (**2a**)[a]

| Aniline | $R^1$ | $R^2$ | Products | | Yield (%)[b] (**3**:**4**) |
|---|---|---|---|---|---|
| **1b** | H | 4-Me | **3b** | **4b** | 76 (87:13) |
| **1c** | H | 4-Cl | **3c** | **4c** | 70 (87:13) |
| **1d** | H | 4-OMe | **3d** | **4d** | 56 (91:9) |
| **1e** | H | 4-$CO_2$Et | **3e** | **4e** | 75 (90:10) |
| **1f** | H | 4-CN | **3f** | **4f** | 82 (95:5) |
| **1g** | H | 3,5-OMe | **3g** | **4g** | 67 (87:13) |
| **1h** | H | 2,4-Me | **3h** | **4h** | 82 (90:10) |
| **1i** | H | 2-Me, 4-Cl | **3i** | **4i** | 86 (91:9) |
| **1j** | H | 4-$NO_2$ | **3j** | – | 78 |
| **1k** | H | 2-OMe, 4-$NO_2$ | **3k** | – | 68 |
| **1l**[c] | H | 2-Cl, 4-Br | **3l** | – | 48 |
| **1m** | Me | H | **3m** | – | 86 |
| **1n** | Et | H | **3n** | – | 79 |
| **1o** | $CH_2CH=CH_2$ | H | – | **4a** | 88 |

[a] Reaction conditions: **1** (10 mmol), **2a** (12 mmol), $Pd(OAc)_2$ (0.1 mmol), $PPh_3$ (0.4 mmol), Ti(OPr-*i*)$_4$ (2.5 mmol) and molecular sieves 4Å (200 mg) in benzene (15 mL) at 50 °C for 3 h.

[b] Isolated yield.

[c] Reaction Time: 24 h.



Scheme 3

IR absorption spectra were recorded on Shimadzu IR-27G and Perkin Elmer System 2000 FT-IR spectrophotometers. $^1$H and $^{13}$C NMR were obtained using Varian Gemini-200 and Unity-400 spectrometers. Chemical shifts (δ) are in ppm relative to TMS and *J* val-

**Table 3** Reaction of **1i** with Allyl Alcohols **2b, c**[a]

| Entry | Allyl Alcohol | Yield (%)[b] (**6**:**7**) |
|---|---|---|
| 1 | **2b** | 80 (55:45)<br>(*E*/*Z* = 86/14)[c] |
| 2 | **2c** | 82 (56:44)<br>(*E*/*Z* = 91:9)[c] |

[a] Reaction conditions: 1i (10 mmol), **2** (12 mmol), $Pd(OAc)_2$ (0.25 mmol), $PPh_3$ (1 mmol), Ti(OPr-*i*)$_4$ (2.5 mmol) and molecular sieves 4Å (200 mg) in benzene (15 mL) at 50 °C for 3 h.

[b] Isolated yield.

[c] The *E*/*Z* ratio of 6 was determined by $^1$H NMR spectroscopy.

**Table 4** Spectral Data of Compounds **3, 4, 6** and **7** Prepared

| Product[a] | IR ($CHCl_3$) ν ($cm^{-1}$) | $^1H$ NMR ($CDCl_3$/TMS) δ, *J* (Hz) | $^{13}C$ NMR ($CDCl_3$/TMS) *δ* | MS *m*/*z* | HRMS *m*/*z* |
|---|---|---|---|---|---|
| **3a**[16] | 3400, 1595, 990, 910 | (400 MHz): 3.56 (br s, 1 H, NH), 3.60 (dt, *J* = 1.6, 5.2, 2 H, $CH_2$), 5.07 (dd, *J* = 1.6, 10.4, 1 H, vinyl H), 5.18 (dd, *J* = 1.6, 17.2, 1 H, vinyl H), 5.82 (ddt, *J* = 5.2, 10.4, 17.2, 1 H, vinyl H), 6.50 (d, *J* = 8.0, 2 H, ArH), 6.65 (t, *J* = 7.6, 1 H, ArH), 7.08–7.13 (m, 2 H, ArH) | (100.6 MHz): 46.10 ($CH_2$), 112.66 (CH), 115.67 ($CH_2$), 117.08 (CH), 128.87 (CH), 135.27 (CH), 147.80 (C) | 133 ($M^+$), 118, 106, 91, 77 | Calcd for $C_9H_{11}N$ 133.0891. Found 133.0890. |
| **3b**[17] | 3420, 1615, 990, 915 | (400 MHz): 2.23 (s, 3 H, $CH_3$), 3.62 (br s, 1 H, NH), 3.73 (dt, *J* = 1.6, 5.6, 2 H, $CH_2$), 5.14 (dq, *J* = 1.6, 10.4, 1 H, vinyl H), 5.26 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.94 (ddt, *J* = 5.2, 10.4, 17.2, 1H, vinyl H), 6.54 (d, *J* = 8.4, 2 H, ArH), 6.98 (d, *J* = 8.4, 2 H, ArH) | (100.6 MHz): 20.38 ($CH_3$), 46.93 ($CH_2$), 113.19 (CH), 116.06 ($CH_2$), 126.73 (C), 129.70 (CH), 135.72 (CH), 145.82 (C) | 147 ($M^+$), 132, 120, 106, 91, 77 | Calcd for $C_{10}H_{13}N$ 147.1408. Found 147.1408. |
| **3c**[16] | 3410, 1625, 990, 910 | (200 MHz): 3.61–3.62 (m, 2 H, $CH_2$), 3.72 (br s, 1 H, NH), 5.11 (dq, *J* = 1.6, 10.2, 1 H, vinyl H), 5.19 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.83 (ddt, *J* = 5.0, 10.2, 17.2, 1 H, vinyl H), 6.42 (d, *J* = 9.0, 2 H, ArH), 7.05 (d, *J* = 9.0, 2 H, ArH) | (50.3 MHz): 46.18 ($CH_2$), 113.77 (CH), 116.06 ($CH_2$), 121.47 (C), 128.72 (CH), 134.74 (CH), 146.43 (C) | 169 ($M^+$+2), 167 ($M^+$), 140, 130, 111, 99, 75 | Calcd for $C_9H_{10}ClN$ 167.0503. Found 167.0502. |
| **3d**[16] | 3400, 1620, 990, 910 | (400 MHz): 3.47 (br s, 1 H, NH), 3.60 (dt, *J* = 1.6, 5.2, 2 H, $CH_2$), 3.63 (s, 3 H, $OCH_3$), 5.08 (dq, *J* = 1.6, 10.4, 1H, vinyl H), 5.20 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.86 (ddt, *J* = 5.6, 10.0, 17.2, 1 H, vinyl H), 6.49 (d, *J* = 9.2, 2 H, ArH), 6.72 (d, *J* = 8.8, 2H, ArH) | (100.6 MHz): 46.95 ($CH_2$), 55.17 ($CH_3$), 113.81 (CH), 114.41 (CH), 115.43 ($CH_2$), 135.58 (CH), 142.00 (C), 151.66 (C) | 163 ($M^+$), 148, 136, 122, 108, 95, 77 | Calcd for $C_{10}H_{13}NO$ 163.0997. Found 163.0998. |
| **3e** | 3386, 1701, 1616, 1033, 928 | (400 MHz): 1.33 (t, *J* =7 .2, 3 H, $CH_3$), 3.76 (dt, *J* = 1.6, 5.2, 2 H, $CH_2$), 4.29 (q, *J* = 7.2, 2 H, $CH_2CH_3$), 4.59 (br s, 1 H, NH), 5.14 (dq, *J* = 1.6, 10.4, 1 H, vinyl H), 5.23 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.87 (ddt, *J* = 5.2, 10.2, 17.2, 1 H, vinyl H), 6.54 (d, *J* = 9.2, 2 H, ArH), 7.86 (d, *J* = 8.8, 2 H, ArH) | (100.6 MHz): 14.19 ($CH_3$), 45.47 ($CH_2$), 59.92 ($CH_2$), 111.34 (CH), 116.20 ($CH_2$), 118.21 (C), 131.17 (CH), 134.17 (CH), 151.70 (C), 166.71 (C) | 205 ($M^+$), 178, 160, 150, 132, 117, 105, 91, 77 | Calcd for $C_{12}H_{15}NO_2$ 205.1103. Found 205.1102. |
| **3f**[17] | 3386, 1621, 1003, 928 | (200 MHz): 3.81 (dt, *J* = 1.6, 5.2, 2 H, $CH_2$), 4.53 (br s, 1 H, NH), 5.20 (dq, *J* = 1.5, 10.1, 1 H, vinyl H), 5.27 (dq, *J* = 1.5, 17.1, 1 H, vinyl H), 5.90 (ddt, *J* = 5.2, 10.2, 17.2, 1 H, vinyl H), 6.57 (d, *J* = 8.9, 2 H, ArH), 7.40 (d, *J* = 8.9, 2 H, ArH) | (50.3 MHz): 45.59 ($CH_2$), 98.64 (C), 112.40 (CH), 116.93 ($CH_2$), 120.52 (C), 133.62 (CH), 133.83 (CH), 151.25 (C) | 158 ($M^+$),142, 131, 116,102, 91, 77 | Calcd for $C_{10}H_{10}N_2$ 158.0844. Found 158.0843. |
| **3g** | 3406, 1616, 998, 918 | (400 MHz): 3.75 (m, 2 H, $CH_2$), 3.77 (s, 6 H, 2 × $OCH_3$), 4.07 (br s, 1 H, NH), 5.21 (dq, *J* = 1.5, 10.3, 1 H, vinyl H), 5.32 (dq, *J* = 1.6, 17.2, 1H, vinyl H), 5.88 (d, *J* = 2.1, 2 H, ArH), 5.90–6.07 (m, 1 H, vinyl H), 5.97 (t, *J* = 2.1, 1 H, ArH) | (100.6 MHz): 45.99 ($CH_2$), 54.51 ($CH_3$), 89.27 (CH), 91.31 (CH), 115.47 ($CH_2$), 135.16 (CH), 149.79 (C), 161.27 (C) | 193 ($M^+$), 178, 166, 164, 147, 138, 125, 107, 77 | Calcd for $C_{11}H_{15}NO_2$ 193.1103. Found 193.1102. |
| **3h** | 3410, 1610, 990, 910 | (400 MHz): 2.12 (s, 3 H, $CH_3$), 2.20 (s, 3 H, $CH_3$), 3.46 (br s, 1 H, NH), 3.78 (dt, *J* = 1.6, 5.6, 2 H, $CH_2$), 5.15 (dq, *J* = 1.6, 10.4, 1 H, vinyl H), 5.27 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.98 (ddt, *J* = 5.2, 10.4, 17.2, 1 H, vinyl H), 6.52 (d, *J* = 8.4, 1 H, ArH), 6.88 (s, 1 H, ArH), 6.90 (d, *J* = 8.0, 1 H, ArH) | (100.6 MHz): 17.40 ($CH_3$), 20.32 ($CH_3$), 46.80 ($CH_2$), 110.29 (CH), 116.04 ($CH_2$), 122.15 (C), 126.25 (C), 127.31 (CH), 130.97 (CH), 135.81 (CH), 143.68 (C) | 161 ($M^+$), 146, 134, 120, 91, 77 | Calcd for $C_{11}H_{15}N$ 161.1207. Found 161.1204. |
| **3i** | 3440, 1600, 990, 920 | (200 MHz): 2.02 (s, 3 H, $CH_3$), 3.53 (br s, 1 H, NH), 3.69 (dt, *J* = 1.6, 5.2, 2 H, $CH_2$), 5.13 (dq, *J* = 1.5, 10.2, 1 H, vinyl H), 5.21 (dq, *J* = 1.5, 17.2, 1 H, vinyl H), 5.89 (ddt, *J* = 5.2, 10.2, 17.2, 1 H, vinyl H), 6.41 (d, *J* = 8.4, 1 H, ArH), 6.94–7.02 (m, 2 H, ArH) | (50.3 MHz): 17.02 ($CH_3$), 46.21 ($CH_2$), 110.77 (CH), 116.09 ($CH_2$), 121.15 (C), 123.44 (C), 126.41 (CH), 129.45 (CH), 134.93 (CH), 144.35 (C) | 183 ($M^+$+2), 181 ($M^+$), 154, 140, 117, 89, 77 | Calcd for $C_{10}H_{12}ClN$ 181.0655. Found 181.0658. |

**Table 4** (continued)

| Product[a] | IR (CHCl$_3$) ν (cm$^{-1}$) | $^1$H NMR (CDCl$_3$/TMS) δ, *J* (Hz) | $^{13}$C NMR (CDCl$_3$/TMS) *δ* | MS *m*/*z* | HRMS *m*/*z* |
|---|---|---|---|---|---|
| **3j** | 3440, 1600, 990, 920 | (200 MHz): 3.87 (dt, *J* = 1.3, 5.1, 2 H, CH$_2$), 5.04 (br s, 1 H, NH), 5.20 (dq, *J* = 1.3, 10.2, 1 H, vinyl H), 5.28 (dq, *J* = 1.3, 17.2, 1 H, vinyl H), 5.90 (ddt, *J* = 5.1, 10.2, 17.2, 1H, vinyl H), 6.55 (d, *J* = 9.2, 2 H, ArH), 8.06 (d, *J* = 9.2, 2 H, ArH) | (50.3 MHz): 45.70 (CH$_2$), 111.30 (CH), 117.12 (CH$_2$), 126.36 (CH), 133.41 (CH), 137.78 (C), 153.51 (C) | 178 (M$^+$), 161, 151, 130, 117, 105, 91, 77 | Calcd for $C_9H_{10}N_2O_2$ 178.0742. Found 187.0743. |
| **3k** | 3426, 1606, 1003, 928 | (200 MHz): 3.89 (dt, *J* = 1.6, 5.6, 2 H, CH$_2$), 3.92 (s, 3 H, CH$_3$), 5.22 (dq, *J* = 1.6, 10.2, 1 H, vinyl H), 5.27 (br s, 1 H, NH), 5.28 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.92 (ddt, *J* = 5.2, 10.2, 17.2, 1 H, vinyl H), 6.48 (d, *J* = 8.9, 1 H, ArH), 7.61 (d, *J* = 2.4, 1 H, ArH), 7.87 (dd, *J* = 2.4, 8.9, 1 H, ArH) | (50.3 MHz): 45.37 (CH$_2$), 55.90 (CH$_3$), 104.68 (CH), 106.97 (CH), 117.07 (CH$_2$), 119.77 (CH), 133.50 (CH), 137.18 (C), 144.04 (C), 145.20 (C) | 208 (M$^+$), 193, 181, 179, 165, 146, 118, 91 | Calcd for $C_{10}H_{12}N_2O_3$ 208.0848. Found 208.0846. |
| **3l** | 3410, 1620, 990, 910 | (400 MHz): 3.72 (m, 2 H, CH$_2$), 4.42 (br s, 1 H, NH), 5.15 (dq, *J* = 1.6, 10.4, 1 H, vinyl H), 5.22 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.86 (ddt, *J* = 5.2, 10.4, 17.2, 1 H, vinyl H), 6.42 (d, *J* = 8.4, 1 H, ArH), 7.15 (dd, *J* = 2.0, 8.8, 1 H, ArH), 7.32 (d, *J* = 2.0, 1 H, ArH) | (100.6 MHz): 45.86 (CH$_2$), 107.58 (C), 112.42 (CH), 116.52 (CH$_2$), 119.54 (C), 130.43 (CH), 131.14 (CH), 134.11 (CH), 142.81 (C) | 249 (M$^+$+4), 247 (M$^+$+2), 245 (M$^+$), 220, 218, 212, 210, 208, 206, 166, 164, 131 | Calcd for $C_9H_9BrClN$ 244.9607. Found 244.9608. |
| **3m**[18] | 1616, 1003, 933 | (200 MHz): 2.90 (s, 3 H, CH$_3$), 3.88 (dt, *J* = 1.7, 5.1, 2 H, CH$_2$), 5.12 (dq, *J* = 1.7, 10.4, 1 H, vinyl H), 5.15 (dq, *J* = 1.7, 17.0, 1 H, vinyl H), 5.82 (ddt, *J* = 5.1, 10.4, 17.0, 1 H, vinyl H), 6.64–6.72 (m, 3 H, ArH), 7.16–7.24 (m, 2 H, ArH) | (50.3 MHz): 37.93 (CH$_3$), 55.23 (CH$_2$), 112.45 (CH), 116.08 (CH$_2$), 116.41 (CH), 129.08 (CH), 133.82 (CH), 149.48 (C) | 147 (M$^+$), 132, 120, 104, 91, 77 | Calcd for $C_{10}H_{13}N$ 147.1048. Found 147.1048. |
| **3n**[19] | 1606, 1003, 933 | (400 MHz): 1.14 (t, *J* = 7.2, 3 H, CH$_3$), 3.35 (q, *J* = 7.2, 2 H, CH$_2$CH$_3$), 3.86 (dt, *J* = 1.6, 4.8, 2 H, CH$_2$), 5.11 (dq, *J* = 1.6, 10.0, 1 H, vinyl H), 5.15 (dq, *J* = 1.6, 17.2, 1 H, vinyl H), 5.84 (ddt, *J* = 4.8, 10.0, 17.2, 1 H, vinyl H), 6.62–6.69 (m, 3 H, ArH), 7.15–7.20 (m, 2 H, ArH) | (100.6 MHz): 12.21 (CH$_3$), 44.61 (CH$_2$), 52.55 (CH$_2$), 112.04 (CH), 115.64 (CH$_2$), 115.79 (CH), 129.06 (CH), 134.42 (CH), 148.11 (C) | 161 (M$^+$), 146, 134, 118, 104, 91, 77 | Calcd for $C_{11}H_{15}N$ 161.1204. Found 161.1203. |
| **4a**[20] | 1600, 980, 910 | (400 MHz): 3.91 (dt, *J* = 1.6, 4.8, 4 H, 2 × CH$_2$), 5.14 (dq, *J* = 1.6, 10.4, 2 H, vinyl H), 5.17 (dq, *J* = 1.6, 17.2, 2H, vinyl H), 5.85 (ddt, *J* = 5.2, 10.4, 17.2, 2 H, vinyl H), 6.65–6.71 (m, 3 H, ArH), 7.17–7.21 (m, 2 H, ArH) | (100.6 MHz): 52.71 (CH$_2$), 112.33 (CH), 115.94 (CH$_2$), 116.28 (CH), 129.02 (CH), 134.03 (CH), 148.68 (C) | 173 (M$^+$), 158, 146, 130, 118, 104, 91, 77 | Calcd for $C_{12}H_{15}N$ 173.1204. Found 173.1205. |
| **4b** | 1610, 990, 920 | (400 MHz): 2.22 (s, 3 H, CH$_3$), 3.86 (dt, *J* = 1.6, 4.8, 4 H, 2 × CH$_2$), 5.12 (dq, *J* = 1.6, 10.0, 2 H, vinyl H), 5.15 (dq, *J* = 1.6, 16.8, 2 H, vinyl H), 5.83 (ddt, *J* = 4.8, 10.0, 16.8, 2 H, vinyl H), 6.46 (d, *J* = 8.8, 2 H, ArH), 6.99 (d, *J* = 8.8, 2H, ArH) | (100.6 MHz): 20.15 (CH$_3$), 52.90 (CH$_2$), 112.66 (CH), 115.84 (CH$_2$), 125.42 (C), 129.52 (CH), 134.27 (CH), 146.58 (C) | 187 (M$^+$), 172, 160, 144, 130, 118, 91, 77 | Calcd for $C_{13}H_{17}N$ 187.1359. Found 187.1360. |
| **4c** | 1595, 990, 920 | (200 MHz): 3.89 (dt, *J* = 1.6, 4.8, 4 H, 2 × CH$_2$), 5.14 (dq, *J* = 1.7, 9.7, 2 H, vinyl H), 5.15 (dq, *J* = 1.7, 17.6, 2H, vinyl H), 5.83 (ddt, *J* = 4.8, 9.6, 17.6, 2 H, vinyl H), 6.59 (d, *J* = 9.2, 2 H, ArH), 7.12 (d, *J* = 9.2, 2 H, ArH) | (50.3 MHz): 52.94 (CH$_2$), 113.45 (CH), 116.16 (CH$_2$), 121.01 (C), 128.79 (CH), 133.51 (CH), 147.37 (C) | 209 (M$^+$+2), 207 (M$^+$), 180, 138, 130, 111, 75 | Calcd for $C_{12}H_{14}ClN$ 207.0817. Found 207.0815. |
| **4d** | 1635, 995, 910 | (400 MHz): 3.74 (s, 3 H, OCH$_3$), 3.85 (dt, *J* = 1.6, 5.2, 4 H, 2 × CH$_2$), 5.14 (dq, *J* = 1.6, 10.4, 2 H, vinyl H), 5.17 (dq, *J* = 1.6, 17.2, 2 H, vinyl H), 5.84 (ddt, *J* = 5.2, 10.4, 17.2, 2 H, vinyl H), 6.68 (d, *J* = 9.2, 2 H, ArH), 6.80 (d, *J* = 8.8, 2 H, ArH) | (100.6 MHz): 53.59 (CH$_2$), 55.75 (CH$_3$), 114.52 (CH), 114.63 (CH), 116.08 (CH$_2$), 134.55 (CH), 143.50 (C), 151.54 (C) | 203 (M$^+$), 188, 176, 162, 135, 120, 92, 77 | Calcd for $C_{13}H_{17}NO$ 203.1310. Found 203.1311. |
| **4e** | 1711, 1611, 1038, 928 | (200 MHz): 1.35 (t, *J* = 7.1, 3 H, CH$_3$), 3.97 (dt, *J* = 1.7, 4.6, 4 H, 2 × CH$_2$), 4.31 (q, *J* = 7.1, 2 H, CH$_2$CH$_3$), 5.15 (dq, *J* = 1.7, 16.8, 2 H, vinyl H), 5.17 (dq, *J* = 1.6, 10.7, 2 H, vinyl H), 5.84 (ddt, *J* = 4.7, 10.7, 16.8, 2 H, vinyl H), 6.64 (d, *J* = 9.2, 2 H, ArH), 7.88 (d, *J* = 9.2, 2 H, ArH) | (50.3 MHz): 14.50 (CH$_3$), 52.65 (CH$_2$), 60.09 (CH$_2$), 110.98 (CH), 116.42 (CH$_2$), 117.72 (C), 131.26 (CH), 132.81 (CH), 151.92 (C), 166.89 (C) | 233 (M$^+$), 218, 204, 192, 190, 177, 165, 137, 122, 107, 77 | Calcd for $C_{15}H_{19}NO_2$ 245.1416. Found 245.1414. |

**Table 4** (continued)

| Product[a] | IR ($CHCl_3$) ν ($cm^{-1}$) | $^1H$ NMR ($CDCl_3$/TMS) δ, *J* (Hz) | $^{13}C$ NMR ($CDCl_3$/TMS) *δ* | MS *m*/*z* | HRMS *m*/*z* |
|---|---|---|---|---|---|
| **4f** | 1611, 1008, 933 | (400 MHz): 3.96 (dt, *J* = 2.0, 4.4, 4 H, 2 × $CH_2$), 5.14 (dq, *J* = 1.6, 17.2, 2 H, vinyl H), 5.19 (dq, *J* = 1.6, 10.4, 2 H, vinyl H), 5.82 (ddt, *J* = 4.8, 10.4, 17.2, 2 H, vinyl H), 6.64 (d, *J* = 9.2, 2 H, ArH), 7.40 (d, *J* = 9.2, 2 H, ArH) | (100.6 MHz): 52.63 ($CH_2$), 97.68 (C), 111.81 (CH), 116.61 ($CH_2$), 120.55 (C), 132.23 (CH), 133.38 (CH), 151.31 (C) | 198 ($M^+$), 183, 171, 155, 129, 102 | Calcd for $C_{13}H_{14}N_2$ 198.1157. Found 198.1157. |
| **4g** | 1615, 990, 915 | (200 MHz): 3.78 (s, 6 H, 2 × $OCH_3$), 3.92 (dt, *J* = 1.6, 4.8, 4 H, 2 × $CH_2$), 5.18 (dq, *J* = 1.6, 10.3, 2 H, vinyl H), 5.22 (dq, *J* = 1.7, 17.1, 2 H, vinyl H), 5.88 (ddt, *J* = 5.2, 10.3, 17.2, 2 H, vinyl H), 5.93 (s, 3 H, ArH) | (50.3 MHz): 52.85 ($CH_2$), 54.97 ($CH_3$), 88.36 (CH), 91.62 (CH), 115.90 ($CH_2$), 133.89 (CH), 150.55 (C), 161.47 (C) | 233 ($M^+$), 218, 204, 192, 190, 177, 165, 137, 122, 107, 77 | Calcd for $C_{14}H_{19}NO_2$ 233.1416. Found 233.1417. |
| **4h** | 1605, 990, 915 | (400 MHz): 2.25 (s, 3 H, $CH_3$), 2.28 (s, 3 H, $CH_3$), 3.53 (dt, *J* = 1.2, 6.0, 4 H, 2 × $CH_2$), 5.07 (dq, *J* = 1.6, 10.0, 2 H, vinyl H), 5.14 (dq, *J* = 1.6, 16.8, 2 H, vinyl H), 5.78 (ddt, *J* = 6.4, 10.0, 16.8, 2 H, vinyl H), 6.90 (s, 1 H, ArH), 6.91 (s, 1 H, ArH), 6.98 (s, 1 H, ArH) | (100.6 MHz): 18.15 ($CH_3$), 20.73 ($CH_3$), 55.98 ($CH_2$), 116.84 ($CH_2$), 121.92 (C), 126.47 (C), 131.75 (CH), 132.51 (CH), 133.82 (CH), 135.55 (CH), 147.34 (C) | 201 ($M^+$), 186, 174, 160, 144, 132, 117, 105, 91, 77 | Calcd for $C_{14}H_{19}N$ 201.1517. Found 201.1516 |
| **4i** | 1635, 990, 920 | (400 MHz): 2.28 (s, 3 H, $CH_3$), 3.53 (dt, *J* = 1.6, 6.4, 4 H, 2 × $CH_2$), 5.09 (dq, *J* = 1.6, 10.4, 2 H, vinyl H), 5.15 (dq, *J* = 1.6, 17.2, 2 H, vinyl H), 5.75 (ddt, *J* = 6.0, 10.0, 17.2, 2 H, vinyl H), 6.91 (d, *J* = 8.8, 1 H, ArH), 7.06 (dd, *J* = 2.4, 8.4, 1 H, ArH), 7.14 (d, *J* = 2.4, 1 H, ArH) | (100.6 MHz): 18.18 ($CH_3$), 55.71 ($CH_2$), 117.32 ($CH_2$), 123.28 (CH), 125.81 (CH), 128.14 (C), 130.76 (CH), 134.93 (CH), 135.86 (C), 148.42 (C) | 223 ($M^+$+2), 221 ($M^+$), 194, 180, 152, 144, 117, 89 | Calcd for $C_{13}H_{16}ClN$ 221.0971. Found 221.0970. |
| **6** | 3446, 1616, 978, 893 | (400 MHz), *E*-isomer: 1.71 (dq, *J* = 1.2, 6.0, 3 H, $CH_3$), 2.11 (s, 3 H, $CH_3$), 3.49 (br s, 1 H, NH), 3.70 (d, *J* = 6.0, 2 H, $CH_2$), 5.60 (dtq, *J* = 1.6, 6.0, 15.2, 1 H, vinyl H), 5.72 (dtq, *J* = 1.2, 6.4, 15.2, 1 H, vinyl H), 6.51 (d, *J* = 8.4, 1 H, ArH), 7.02 (d, *J* = 2.4, 1 H, ArH), 7.05 (dd, *J* = 2.4, 8.4, 1 H, ArH).<br>*Z*-isomer: 1.73 (dq, *J* = 1.2, 6.0, 3 H, $CH_3$), 2.10 (s, 3 H, $CH_3$), 3.52 (br s, 1 H, NH), 3.78 (d, *J* = 6.4, 2 H, $CH_2$), 5.53–5.71 (m, 2 H, vinyl H), 6.52 (d, *J* = 8.4, 1 H, ArH), 7.02 (d, *J* = 2.4, 1 H, ArH), 7.07 (dd, *J* = 2.4, 8.4, 1 H, ArH) | (100.6 MHz), *E*-isomer: 17.32 ($CH_3$), 17.77 ($CH_3$), 46.06 ($CH_2$), 110.96 (CH), 121.42 (C), 123.64 (C), 126.66 (CH), 127.69 (CH), 128.29 (CH), 129.68 (CH), 144.68 (C).<br>*Z*-isomer: 17.30 ($CH_3$), 17.80 ($CH_3$), 46.09 ($CH_2$), 110.85 (CH), 121.45 (C), 123.65 (C), 126.64 (CH), 127.37 (CH), 127.60 (CH), 129.70 (CH), 144.79 (C) | 197 ($M^+$+2), 195 ($M^+$), 180, 154, 141, 117, 106, 89, 77 | Calcd for $C_{11}H_{14}ClN$ 195.0815. Found 195.0814. |
| **7** | 3436, 1606, 993, 923, 894 | (200 MHz): 1.36 (d, *J* = 6.6, 3 H, $CH_3$), 2.13 (s, 3 H, $CH_3$), 3.45 (br s, 1 H, NH), 3.99 (dq, *J* = 5.8, 6.5, 1 H, CH), 5.11 (dt, *J* = 1.3, 10.3, 1 H, vinyl H), 5.19 (dt, *J* = 1.4, 17.2, 1 H, vinyl H), 5.84 (ddd, *J* = 5.5, 10.3, 17.2, 1 H, vinyl H), 6.52 (d, *J* = 9.4, 1 H, ArH), 7.03–7.07 (m, 2 H, ArH) | (50.3 MHz): 17.36 ($CH_3$), 21.76 ($CH_3$), 51.04 (CH), 111.87 (CH), 114.23 ($CH_2$), 121.20 (C), 123.37 (C), 126.51 (CH), 129.69 (CH), 140.88 (CH), 143.80 (C) | 197 ($M^+$+2), 195 ($M^+$), 180, 168, 144, 130, 117, 106, 89, 77 | Calcd for $C_{11}H_{14}ClN$ 195.0815. Found 195.0813. |

[a] Satisfactory elemental analyses obtained (C ± 0.21, H ± 0.18, N ± 0.24).

ues are in Hz. MS and high resolution mass spectra (HRMS) were recorded on a Hewlett Packard 5989A or JEOL JMS D-100 instrument, with a direct inlet system.

### *N*-Allylation of Anilines 1 with Allyl Alcohols 2; General Procedure

A mixture of **1** (10 mmol), **2** (12 mmol), $Pd(OAc)_2$ (23 mg, 0.1 mmol), $PPh_3$ (105 mg, 0.4 mmol), Ti(OPr-*i*)$_4$ (0.75 mL, 2.5 mmol), molecular sieves (4Å) (200 mg) and benzene (15 mL) was stirred under $N_2$ at 50°C for 3 h. After cooling, the mixture was poured into aq 10% HCl and extracted with $Et_2O$. The aqueous layer was mixed with aq 10% NaOH solution and extracted with $Et_2O$. The $Et_2O$ layers were combined, dried ($Na_2SO_4$) and concentrated. Column chromatography (hexane/EtOAc, 5:1) of the residue afforded products. Spectroscopic data for *N*-allylanilines **3**, **4**, **6** and **7** are given in Table 4.

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