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Direct Synthesis of β-Alkyl *N*-Aryl Aza Baylis–Hillman Adducts via Nitroso-Ene Reaction

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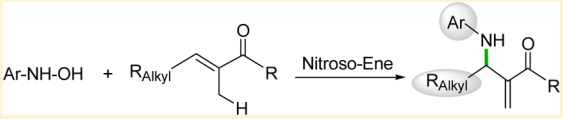
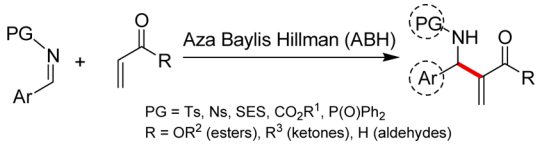
Supporting Information

ABSTRACT: A new approach for the direct Fe-catalyzed synthesis of β-alkyl *N*-aryl aza Baylis–Hillman (ABH) adducts is reported. This approach involves the formation of a C–N bond via a nitroso-ene reaction. This is a simple, fast, and best alternate method to overcome the substrate scope limitations of the ABH reaction, which converts allyl esters and carbonyl compounds to novel ABH adducts. A variety of arylhydroxylamines reacted with esters, aldehydes, ketone, and nitriles to yield the corresponding products in moderate to excellent yields.



The Aza Baylis–Hillman (ABH) or Aza Morita–Baylis–Hillman (AMBH) reaction[1] is an important carbon–carbon bond-forming process that affords densely functionalized β-amino carbonyl compounds, which are widely used for the synthesis of biologically active compounds, medicinal reagents, and natural products.[2] The *N*-aryl β-aminoacid derivatives are key structural elements of many natural products, drug intermediates, β-peptides, β-lactam antibiotics.[3] The *N*-aryl β-aminoacid derivative, SB-214857, is a potent GPIIb/IIIa receptor antagonist currently in phase III clinic trials for the protection of secondary thrombotic events such as heart attack and stroke.[3c,d] The classical ABH reaction uses activated aldimines, α,β-unsaturated carbonyls, and nucleophilic catalysts. The activated aryl aldimines mainly includes tosylimines,[4a] nosylimines,[4b] SES-imines,[4a] phosphinoylimines[4] of their corresponding aromatic aldehydes and amides (Scheme 1).

Scheme 1. Aza Baylis–Hillman Reaction Using Activated Aryl Aldimines



Enormous efforts have been made to this valuable process in the past decades;[1a] nevertheless, the ABH reaction remains to be restricted in substrate scope, protic (polar) solvents[5] and poor reaction rates.[1] Two major limitations of ABH reactions related to the nature of imine substrates are (i) the nitrogen counterpart of imine requires activated/protected amine such as sulfonamides, phosphonamides, etc., and (ii) the aldehyde counterpart of the imines should always be aromatic, i.e. benzaldehydes. The activated aromatic aldimines were well explored for the ABH reaction to prepare *N*-protected β-amino carbonyl compounds (Scheme 1). However, neither activated aliphatic aldimines nor *N*-aryl aromatic aldimines are suitable precursors for the ABH reaction, for which very rare examples[6] have been reported so far, the former due to instability[7] and the latter due to poor reactivity of imine functionality. In ABH reaction, use of aliphatic aldehydes leads to a mixture of β-amino ester resulting from the attack of the enolate on the imine and of the corresponding β-hydroxy ester resulting from a direct attack of the enolate on the aldehyde.[8] In light of this, various research groups have developed multistep approaches for the synthesis of β-alkyl amino carbonyl compounds via the traditional Baylis–Hillman reaction and other alternate methods[1a] (Scheme 2). The use of ABH reaction/products always requires the activation of the intermediate alcohol (BH adduct) function as a bromide, an acetate, a phosphate or as any other leaving group.[9] Another limitation of this method is that the possibility of obtaining a mixture of products via nucleophilic substitution (S_N2) product and allylic substitution (S_N2') (Scheme 2).[10]

The usefulness of the ABH reaction has encouraged researchers to employ different alternatives to overcome these existing limitations. Since there is no report of using aldimines of their corresponding aliphatic aldehydes and arylamines to synthesize highly useful β-alkyl *N*-arylamino ABH adducts, we envisaged that it would be possible to access the same adducts via a nitroso-ene reaction[11] using arylhydroxylamines and tiglate esters as substrates (Scheme 3). This method involves a C–N bond formation, whereas C–C bond formation takes place in the ABH reaction. We have been working in the field of iron and copper-catalyzed nitroso-ene reactions using nitrosoarenes, arylhydroxylamines, and simple olefins as well as alkynes.[12] So far, there is no report on nitroso-ene reactions using α,β-unsaturated esters. However, to our surprise, ethyltiglate undergoes a nitroso-ene reaction to yield a β-alkyl *N*-arylamino ABH adduct using our previously developed Fe-catalytic system.[12a,b]

Initially we screened a known set of iron catalysts[12a,b,13] such as Fe(Pc), $FeCl_2.4H_2O$, anhydrous $FeCl_3$, Fe(acac),

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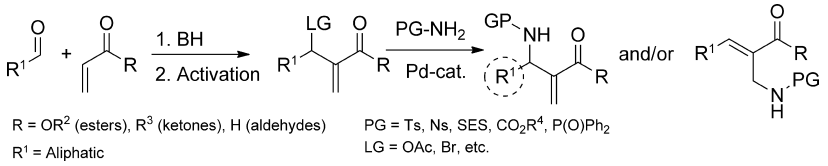
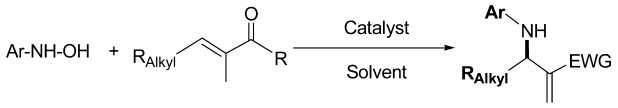
Scheme 2. Alternate Multistep Process to Prepare β-Alkyl ABH Adducts



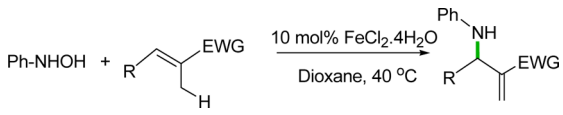
Scheme 3. Direct Synthesis of *N*-Aryl β-Alkyl ABH Adducts



$[Fe(azodioxide)_3]^{2+}$, and copper catalysts[12c−f,14a−c] $[Cu(MeCN)_4]PF_6$, CuCl, $CuCl_2·2H_2O$, and $CuBr·SMe_2$ for the synthesis of ABH adduct using phenylhydroxylamine (nitrogen fragment donor) and ethyltiglate in 1,4-dioxane at a range of temperatures (up to 100 °C). We have found that $FeCl_2·4H_2O$ and $[Fe(azodioxide)_3]^{2+}$ complex[12a,b] afforded ABH adduct in 75 and 78% yield, respectively, at 40 °C in a relatively very short time (6 h) unlike the reported methods, which require longer reaction times, usually 1−3 days.[1a] Because of easy availability and low cost, we preferred $FeCl_2·4H_2O$ catalyst for the present ABH adduct formation. When a series of α,β-unsaturated esters (a−d), ketone (e), aldehydes (f, g), and nitriles (h, i) were subjected to optimized conditions (10 mol % $FeCl_2·4H_2O$, 1,4-dioxane, 40 °C) with phenylhydroxylamine (1), corresponding *N*-phenyl β-alkyl ABH adducts (**1a**−**1i**) were produced in good to excellent yields as shown in Table 1. GC−MS analyses of reaction mixtures indicate that a small amount (<5%) of aniline and azoxybenzene were detected, which are due to reduction and condensation of phenylhydroxylamine (1). To minimize the side product formation, we have used syringe pump to deliver phenylhydroxylamine solution slowly. The α,β-unsaturated esters having allylic C−H bonds, ethyltiglate (a), sec-butyltiglate (b), methy-2-methyl-2-pentenoate (c) reacted well with phenylhydroxylamine (1) and gave the corresponding ABH adducts (**1a**, **1b**, **1c**) in excellent yields.

Though methyl methacrylate (d) is structurally similar to the other esters, the reaction gave a moderate yield of adduct **1d** (57%) along with aniline (21%) as a side product. Other functional groups such as the ketone and aldehydes are well tolerated under the present reaction conditions. 3-Methyl-3-penten-2-one (e), 2-methyl-2-pentenal (f), 2-methyl-2-butenal (g) gave the expected products (**1e**, **1f**, **1g**) in good yields, which are better than previously reported for similar ABH reaction adducts.[15] A relatively lower yield of aldehydes is due to the possible formation of a condensation product of PhNHOH with aldehydes. To diminish the condensation process, reactions of aldehydes (f, g) were carried out at 30 °C. The reaction of methacrylonitrile (h) was not effective (**1h**, <5%) in the present reaction condition, possibly because of the strong coordination of the nitrile to metal thereby making the catalyst less active. As we expected, an increase in the reaction temperature (up to 70 °C) and substrate ratio (PhNHOH/nitrile: 1/5) improved the yield up to 12% (GC), but the formation of other side products were also observed in this reaction. Unsuccessful attempts were made to purify this product by column chromatography. Surprisingly, when we replaced the methacrylonitrile (h) with 2-methyl-2-butene nitrile (i), a very good improvement in the product yield (**1i**, 42%) was observed at 70 °C. We have carried out an experiment with phenylhydroxylamine in large scale (5 g) using ethyltiglate to give ABH adduct (**1a**) in 62% yield, which confirms the scalability of the present reaction.

Table 1. β-Alkyl *N*-Aryl ABH Adducts Formation Using Phenylhydroxylamine[a]



Substrate	ABH adduct	% Yield[b]
(a)	(1a)	75
(b)	(1b)	83
(c)	(1c)	88
(d)	(1d)	57
(e)	(1e)	69
(f)	(1f)	54[c]
(g)	(1g)	61[c]
(h)	(1h)	12[d,e]
(i)	(1i)	42[d]

[a]All reactions were performed at 40 °C with 1:3 substrate ratio (PhNHOH:substrate). [b]Isolated yields. [c]Reactions performed at 30 °C. [d]Reactions performed at 70 °C. [e]GC yield.

Having established this new approach with various α,β-unsaturated carbonyls and nitriles using phenylhydroxylamine (1), we chose to evaluate other substituted arylhydroxylamines (2, 3, 4) by keeping ethyltiglate (a) constant (Table 2). *p*-tolylhydroxylamine (2) reacted very similar to phenylhydroxylamine to give the target product (**2a**) in 72% yield. This methodology was successfully applied to other 2-haloaryl hydroxylamines (3 and 4) and afforded halogenated ABH adducts (**3a** and **4a**) without any side reaction. The formation

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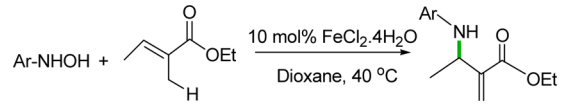
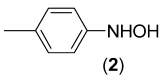
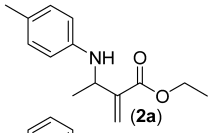
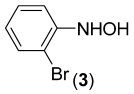
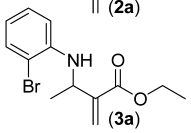
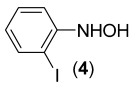
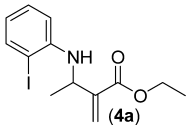
Table 2. β-Alkyl *N*-Aryl ABH Adducts Formation Using Ethyltiglate[a]



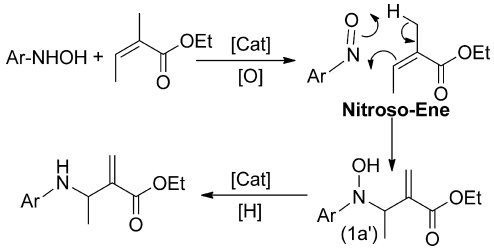
Substrate	ABH adduct	% Yield[b]
 (2)	 (2a)	72
 (3)	 (3a)	78
 (4)	 (4a)	81

[a]All reactions were performed at 40 °C with 1:3 substrate ratio (ArNHOH:substrate) unless otherwise mentioned. [b]Isolated yields.

of these halogenated ABH product can undergo further coupling reactions for the elaboration of more complex compounds, such as N-heterocycles.[16]

To confirm the catalyst involvement in the reaction, we performed two experiments without using catalyst: (i) reaction of phenylhydroxylamine and ethyltiglate and (ii) reaction of nitrosobenzene and ethyltiglate. First reaction did not give any ABH adduct, where as the second reaction gave an *N*-hydroxy ABH adduct (**1a**′, Scheme 4) in lower yield (32%), which is an

Scheme 4. Nitroso-Ene Reaction Pathway to Access β-Alkyl *N*-Aryl ABH Adducts



intermediate in the present method. From these observations, we concluded that the catalyst is required for the present reaction. We have already established the mechanism of allylic amination of unactivated olefins in which metal−nitroso complexes are found to be the intermediates.[12a,b] We believe that the present reaction also follows a similar pathway, which basically involves three consecutive steps, i.e., oxidation, nitroso-ene, and reduction as shown in Scheme 4. The efficiency and high product yields of this method are mainly due to high reactivity of the esters and carbonyl compounds when compared to simple allylic substrates.[8] This catalytic process involves both oxidation and reduction steps, thereby helping to regenerate the catalyst to complete the catalytic cycle.

In conclusion, we have described a convenient and efficient method for the formation of new ABH adducts using arylhydroxylamines under mild reaction conditions. This reaction works with a variety of carbonyl derivatives such as esters, aldehydes, a ketone and nitriles. The present approach is based on C−N bond formation via a nitroso-ene reaction in which nitroso compounds are generated in situ from the corresponding arylhydroxylamines. This is the best alternate method to Aza Baylis−Hillman reaction to access β-alkyl *N*-arylamino ABH adducts. This new strategy is operationally simple and provides a practical solution to access novel ABH adducts, and it should allow for the future advances in the field of aza Baylis−Hillman reactions.

EXPERIMENTAL SECTION

General Information. All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous sodium sulfate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (60−120 mesh size) was used for the column chromatography. Reactions were monitored by TLC on silica gel 60 F_{254} (0.25 mm). NMR spectra were recorded in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard for ^{1}H NMR (400 MHz) and for ^{13}C NMR (100 MHz). Mass spectra were recorded by electro spray ionization (ESI) with a Q-TOF.

General Procedure for the Preparation of β-Alkyl *N*-Aryl Aza Baylis−Hillman Adduct (1a). To the solution of $FeCl_2{\cdot}4H_2O$ (5 mg, 0.025 mmol), ethyltiglate (80 μL, 0.75 mmol) in dioxane (3 mL) was added the phenylhydroxylamine (0.25 mmol) solution slowly in dioxane (4 mL) via syringe pump over 4 h at 40 °C. Reactions were allowed to continue for two more hours to get complete consumption of phenylhydroxylamine. After that, the mixture was filtered through Celite, and the filtrate was concentrated to dryness. The crude product was purified over a short column of silica gel (hexane/ethylacetate eluents) to give the corresponding ABH adduct in 75% (41 mg) isolated yield, which was then directly analyzed by GC−MS, NMR, IR, and ESI-MS analysis.

Ethyl 2-Methylene-3-(phenylamino)butanoate (1a). 41 mg, 75% yield: Colorless gum; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.32 (t, 3H, J = 7.2 Hz), 1.40 (d, 3H, J = 6.4 Hz), 3.92 (brs, 1H), 4.24 (q, 2H, J = 7.2 Hz), 4.39 (q, 1H, J = 6.4 Hz), 5.75 (s, 1H), 6.17 (s, 1H), 6.53 (d, 2H, J = 6.4 Hz), 6.68 (t, 1H, J = 7.2 Hz), 7.14 (t, 2H, J = 6.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.1, 20.9, 49.1, 59.7, 112.4, 116.5, 123.3, 128.1, 141.3, 145.6, 165.5; IR (KBr) 3402, 2975, 2932, 1710, 1619, 1514, 1283, 1182, 1105, 1023, 953, 810 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{18}NO_2$ (M + H$^+$), 220.1338, found 220.1336.

sec-Butyl 2-Methylene-3-(phenylamino)butanoate (1b). 52 mg, 83% yield: Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) 0.93 (q, 3H, J = 7.6 Hz), 1.26 (t, 3H, J = 7.2 Hz), 1.40 (d, 3H, J = 6.8 Hz), 1.58−1.67 (m, 2H), 3.97 (brs, 1H), 4.40 (q, 1H, J = 6.4 Hz), 4.95−4.98 (m, 1H), 5.71 (s, 1H), 6.15 (s, 1H), 6.54 (d, 2H, J = 7.6 Hz), 6.68 (t, 1H, J = 7.2 Hz), 7.13 (t, 2H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 9.7, 19.4, 21.9, 28.8, 50.1, 72.6, 113.4, 117.5, 123.9, 129.1, 142.8, 146.7, 166.2; IR (KBr) 3403, 3020, 2972, 2934, 2878, 1704, 1602, 1505, 1455, 1375, 1282, 1091, 749, 692 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{22}NO_2$, 248.1651, found 248.1648.

Methyl 2-Methylene-3-(phenylamino)pentanoate (1c). 48 mg, 88% yield: White solid, Mp. 92−94 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.97 (t, 3H, J = 7.6 Hz), 1.61−1.67 (m, 1H), 1.72−1.85 (m, 1H), 3.76 (s, 3H), 4.17 (t, 1H, J = 6.0 Hz), 5.71 (s, 1H), 6.19 (s, 1H), 6.52 (d, 2H, J = 7.6 Hz), 6.66 (t, 1H, J = 7.2 Hz), 7.12 (t, 2H, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.0, 28.6, 52.0, 56.5, 113.6, 117.6, 125.7, 129.3, 140.8, 147.2, 167.2; IR (KBr) 3395, 2970, 2958, 1700, 1630, 1435, 1309, 1279, 1125, 747, 691 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{18}NO_2$, 220.1338, found 220.1332.

Methyl 2-Methylene-3-(phenylamino)propanoate (1d). 27 mg, 57% yield: Brown color Oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 3H), 4.04 (s, 2H), 4.20 (brs, 1H), 5.80 (s, 1H), 6.27 (s, 1H), 6.60 (d, 2H, J = 7.6 Hz), 6.71 (t, 1H, J = 7.2 Hz), 7.17 (t, 2H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 44.8, 51.9, 113.0, 117.7, 126.0, 129.2, 137.2, 147.4, 166.9.; IR (KBr) 3413, 3052, 3022, 2951, 2848, 1714,

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1634, 1603, 1506, 1437, 1327, 1263, 1152, 1101, 817, 751, 693 cm^{-1}; HRMS (ESI) calcd for $C_{11}H_{14}NO_2$, 192.1025, found 192.1019.

3-Methylene-4-(phenylamino)pentan-2-one (1e). 32 mg, 69% yield: Yellow Gum; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (d, 3H, J = 6.8 Hz), 2.37 (s, 3H), 3.93 (brs, 1H), 4.47 (q, 1H, J = 6.4 Hz), 5.97 (s, 1H), 6.05 (s, 1H), 6.47 (d, 2H, J = 8.0 Hz), 6.67 (t, 1H, J = 7.2 Hz), 7.12 (t, 2H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.2, 26.7, 48.9, 113.3, 117.5, 125.0, 129.1, 146.6, 150.3, 199.9.; IR (KBr) 3389, 3051, 3020, 2925, 2857, 1663, 1602, 1504, 1389, 1281, 1131, 750, 693 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_{16}NO$, 190.1232, found 190.1227.

2-Methylene-3-(phenylamino)pentanal (1f). 25 mg, 54% yield: Colorless gum;^1H NMR (400 MHz, $CDCl_3$) δ 0.96 (t, 3H, J = 8.0 Hz), 1.63 (m, 1H), 1.78 (m, 1H), 3.94 (s, 1H), 4.20 (t, 1H, J = 6.4 Hz), 6.04 (s, 1H), 6.35 (s, 1H), 6.47 (d, 2H, J = 7.2 Hz), 6.66 (t, 1H, J = 7.2 Hz), 7.11 (t, 2H, J = 8.4 Hz), 9.61 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.9, 28.3, 53.9, 113.5, 117.8, 129.4, 135.4, 147.0, 150.4, 194.6; IR (KBr) 3392, 3022, 2965, 2931, 2873, 1683, 1599, 1504, 1315, 1259, 1031, 751, 693 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_{16}NO$, 190.1232, found 190.1223.

2-Methylene-3-(phenylamino)butanal (1g). 27 mg, 61% yield: Colorless gum; ^1H NMR (400 MHz, $CDCl_3$) δ 1.40 (d, 3H, J = 6.8 Hz), 3.92 (brs, 1H), 4.40 (q, 1H, J = 6.8 Hz), 6.03 (s, 1H), 6.39 (s, 1H), 6.48 (d, 2H, J = 8.0 Hz), 6.69 (t, 1H, J = 7.6 Hz), 7.13 (t, 2H, J = 8.0 Hz), 9.65 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.8, 47.9, 113.5, 117.9, 129.4, 134.5, 146.6, 151.7, 194.6; IR (KBr) 33399, 2964, 2893, 1684, 1601, 1504, 1317, 1261, 1027, 799, 751, 693 cm^{-1}; HRMS (ESI) calcd for $C_{11}H_{14}NO$, 176.1075, found 176.1062.

2-Methylene-3-(phenylamino)butanenitrile (1i). 18 mg, 42% yield: Brownish gum; ^1H NMR (400 MHz, $CDCl_3$) δ 1.50 (t, 3H, J = 3.6 Hz), 3.80 (d, 1H, J = 4.8 Hz), 4.10 (m, 1H), 5.94 (s, 1H), 5.95 (s, 1H), 6.55 (d, 2H, J = 7.6 Hz), 6.76 (t, 1H, J = 7.6 Hz), 7.18 (t, 2H, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 52.4, 113.7, 117.6, 118.7, 126.6, 129.5, 129.8, 145.7; IR (KBr) 3392, 3052, 2972, 2905, 2221, 1602, 1505, 1315, 1259, 1157, 1029, 945, 750 cm^{-1}; HRMS (ESI) calcd for $C_{11}H_{13}N_2$, 173.1079, found 173.1067.

Ethyl 3-(*p*-Tolylamino)-2-methylenebutanoate (2a). 41 mg, 72% yield: Colorless gum; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (t, 3H, J = 7.2 Hz), 1.40 (d, 3H, J = 6.8 Hz), 2.22 (s, 3H), 4.24 (q, 2H, J = 7.2 Hz), 4.36 (q, 1H, J = 6.4 Hz), 5.73 (s, 1H), 6.16 (s, 1H), 6.46 (d, 2H, J = 7.2 Hz), 6.95 (d, 2H, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 20.6, 22.2, 50.7, 60.9, 113.8, 124.5, 126.9, 129.8, 142.6, 144.6, 166.8.; IR (KBr) 3401, 2978, 2893, 1709, 1617, 1519, 1373, 1284, 1182, 1105, 808 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{19}NO_2$ ($M + H^+$) 234.1494, found 234.1486.

Ethyl 3-(2-Bromophenylamino)-2-methylenebutanoate (3a). 58 mg, 78% yield: Brownish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 1.33 (t, 3H, J = 6.8 Hz), 1.46 (d, 3H, J = 6.8 Hz), 4.25 (q, 2H, J = 6.8 Hz), 4.47 (t, 1H, J = 6.8 Hz), 4.58 (s, 1H), 5.70 (s, 1H), 6.19 (s, 1H), 6.45 (d, 1H, J = 8.0 Hz), 6.54 (t, 1H, J = 6.8 Hz), 7.10 (t, 1H, J = 7.2 Hz), 7.41 (d, 1H, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 22.2, 50.0, 61.0, 109.9, 112.7, 118.1, 124.4, 128.5, 132.5, 142.1, 143.5, 166.6; IR (KBr) 3411, 3068, 2979, 2933, 1713, 1595, 1505, 1459, 1321, 1282, 1128, 1105, 1019, 954, 815, 742 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{17}BrNO_2$ ($M + H^+$), 298.0443, 300.0422, found 298.0436, 300.0418.

Ethyl 3-(2-Iodophenylamino)-2-methylenebutanoate (4a). 69 mg, 81% yield: Brownish oil; ^1H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, 3H, J = 7.2 Hz), 1.40 (d, 3H, J = 6.0 Hz), 4.20 (q, 2H, J = 7.2 Hz), 4.36 (brs, 2H), 5.62 (s, 1H), 6.12 (s, 1H), 6.30–6.37 (m, 2H), 7.06 (t, 1H, J = 6.8 Hz), 7.58 (d, 1H, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.2, 21.0, 49.1, 59.8, 84.4, 110.8, 117.8, 123.3, 128.3, 138.0, 141.0, 144.6, 165.4; IR (KBr) 3392, 3064, 2970, 2927, 1716, 1589, 1506, 1449, 1270, 1124, 1102, 1005, 742 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_{17}INO_2$, 346.0304, found 346.0307.

■ ASSOCIATED CONTENT

Supporting Information

General information and copies of ^1H, ^{13}C NMR spectra for all products. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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