Supporting Information for:

Cationic Iron(III) Porphyrin Catalyzed Dehydrative Friedel–Crafts Reaction of Alcohols with Arenes

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**Instrumentation and Chemicals**

All manipulations of oxygen- and moisture-sensitive materials were conducted in a dry box or with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (\(^1\)H, 500 MHz; \(^{13}\)C, 125.7 MHz) spectrometer using tetramethylsilane (\(^1\)H) as an internal standard. \(^1\)H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz), integration, and identification. High-resolution mass spectra were obtained with a Thermo Fisher SCIENTIFIC EXACTIVE spectrometer. Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAI GEL-1H and -2H columns (toluene as an eluent). Infrared spectra (IR) spectra were determined on a SHIMADZU IR Affinity-1 spectrometer. Melting points were determined using a YANAKO MP-500D. TLC analyses were performed by means of Merck Kieselgel 60 F\(_{254}\) (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline KMnO\(_4\) solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–50 μm). Unless otherwise noted, commercially available reagents were used without purification. Xylene was purchased from Wako Pure Chemical Co. stored over slices of sodium.
Experimental Procedure

Preparation of [Fe(TPP)]SbF$_6$.

Fe(TPP)Cl (0.5 mmol, 352 mg) and AgSbF$_6$ (0.5 mmol, 171 mg) was dissolved in dry CH$_2$Cl$_2$ (10mL) and stirred for 2h in dry box. The reaction mixture was filtered and concentrated to dryness. The complex was used without further purification. Other iron porphyrin complexes were also prepared by this procedure.

General procedure for the dehydrative Friedel–Crafts reaction of alcohols with arenes.

To a screw cap vial was added [Fe(TPP)][SbF$_6$] (0.02 mmol, 18 mg), followed by the alcohol (0.4 mmol), the arene (2 mmol) and dry 1,2-dichloroethane (1.0 mL) in a dry box. The vial was sealed and stirred under the indicated condition. The reaction mixture was diluted with hexane/ethyl acetate = 10/1, passed through a short silica gel pad and washed with hexane/ethyl acetate = 1/1, concentrated in vacuo. The crude product was purified by flash column chromatography using basic SiO$_2$ (hexane/ethyl acetate).
Characterization Data of Products

1-Cinnamyl-4-methoxybenzene (3aa).

Yield: 63%, colorless oil. TLC: R_f = 0.62 (hexane/toluene = 2/1).
\[ \delta \]
\[ \begin{align*} & 7.36 (d, J = 7.5 \text{ Hz}, 2\text{H}), 7.31–7.28 (m, 2\text{H}), \\ & 7.22–7.16 (m, 3\text{H}), 6.88–6.86 (m, 1\text{H}), 6.44 (d, J = 16 \text{ Hz}, 1\text{H}), \\ & 6.38–6.32 (m,1\text{H}), 3.81 (s, 3\text{H}), 3.50 (d, J = 6.5 \text{ Hz}, 3\text{H}); \] 13C NMR (CDCl_3) \[ \delta \] 158.1, 137.5, 132.2, 130.7, 129.7, 129.6, 128.5, 127.0, 126.1, 113.9, 55.3, 38.4; IR (neat): 3025, 1511, 1246, 1175, 1035, 966, 827, 729, 692 cm^{-1}; MS m/z (%): 224/25 (100/17) [M^+]; HRMS (APCI) found 225.1269, Calcd for C_{16}H_{16}O [M+H]^+ 225.1274.

1-Cinnamyl-2-methoxybenzene (3aa').

Yield: 9%, colorless oil. TLC: R_f = 0.76 (hexane/toluene = 2/1).
\[ \delta \]
\[ \begin{align*} & 7.35–7.17 (m, 7\text{H}), 6.93–6.88 (m, 2\text{H}), 6.46–6.37 (m, 2\text{H}), 3.86 (s, 3\text{H}), 3.55 (d, J = 6.0 \text{ Hz}, 2\text{H}); \] 13C NMR (CDCl_3) \[ \delta \] 157.3, 137.8, 130.7, 129.8, 128.9, 128.7, 128.4, 127.4, 126.9, 126.1, 120.5 110.4, 55.4, 33.4; IR (neat): 2925, 1491, 1243, 752 cm^{-1}; MS m/z (%): 224/225 (100/18) [M^+]; HRMS (APCI) found 242.1534, Calcd for C_{16}H_{16}O [M+NH_4]^+ 242.1539.

(E)-1-Methoxy-4-(4-phenylbut-3-en-2-yl)benzene (3ba).

Yield: 85%, yellow oil. TLC: R_f = 0.76 (hexane/ethyl acetate = 20/1).
\[ \delta \]
\[ \begin{align*} & 7.37–7.35 (m, 2\text{H}), 7.31–7.26 (m, 2\text{H}), \\ & 7.21–7.18 (m, 3\text{H}), 6.88 (d, J = 6.5 \text{ Hz}, 2\text{H}), 6.38 (d, J = 7.0 \text{ Hz}, 2\text{H}), 3.81 (s, 3\text{H}), 3.63–3.58 (m,1\text{H}), 1.45 (d, J = 7.0 \text{ Hz}, 3\text{H}); \] 13C NMR (CDCl_3) \[ \delta \] 158.0, 137.7, 137.6, 135.6, 128.5, 128.2, 128.2, 127.0, 126.1, 113.9, 55.3, 41.7, 21.3 ; IR (neat): 2962, 1511, 1248, 1177, 1035, 828, 693 cm^{-1}; MS m/z (%): 238/239 (66/13) [M^+], 223/224 (100/17) [M^+-Me]; HRMS (APCI) found 238.1421, Calcd for C_{17}H_{18}O [M+H]^+ 238.1430.

(E)-1-Methoxy-4-(3-phenylbut-2-enyl)benzene (3ca).

Yield: 52%, yellow oil. TLC: R_f = 0.22 (hexane/ethyl acetate = 40/1).
\[ \delta \]
\[ \begin{align*} & 7.45–7.43 (m, 2\text{H}), 7.35–7.32 (m, 2\text{H}), \\ & 7.27–7.25 (m, 1\text{H}), 7.20–7.18 (m, 2\text{H}), 6.00–5.97 (m, 1\text{H}), 3.82 (s,3\text{H}), 3.54 (d, J = 7.5 \text{ Hz}, 2\text{H}), 2.17 (s, 3\text{H}); \] 13C NMR (CDCl_3) \[ \delta \] 157.9, 143.7, 135.4, 133.1, 129.3, 128.2, 127.1, 126.7, 125.7, 113.9, 55.3, 34.0, 15.9; IR (neat): 2955, 1511, 1247, 1035, 699 cm^{-1}; MS m/z (%):238/239 (88/16) [M^+], 223/224 (100/18) [M^+-Me]; HRMS (APCI) found 239.1423, Calcd for C_{17}H_{18}O [M+H]^+ 239.1430.
(E)-1-Methoxy-4-(3-methyl-4-phenylbut-3-en-2-yl)benzene (3da).
Yield: 63%, colorless oil. TLC: Rf = 0.44 (hexane/ethyl acetate = 30/1). 
$^1$H NMR (CDCl$_3$) $\delta$ 7.34–7.31 (m, 2H), 7.29–7.26 (m, 2H), 7.22–7.18 (m, 3H), 6.88–6.85 (m, 2H), 6.47 (s, 1H), 3.81 (s, 3H), 3.54 (q, $J$ = 7.0 Hz, 1H), 1.71 (d, $J$ = 1.5 Hz, 3H), 1.46 (d, $J$ = 7.0 Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 157.9, 142.6, 138.5, 137.1, 129.0, 128.4, 128.0, 126.4, 113.7, 55.2, 47.6, 19.7, 16.4; IR (neat): 2964, 1510, 1246, 1176, 1037, 830, 699 cm$^{-1}$; MS m/z (%): 252/253 (81/19) [M$^+$], 237/238 (100/17) [M$^+$–Me]; HRMS (APCI) found 270.1844, Calcd for C$_{18}$H$_{20}$O [M$+$NH$_4^+$] 270.1852.

(Y)-1-Methoxy-2-(3-methyl-4-phenylbut-3-en-2-yl)benzene (3da').
Yield: 8%, colorless oil. TLC: Rf = 0.59 (hexane/ethyl acetate = 30/1). 
$^1$H NMR (CDCl$_3$) $\delta$ 7.33–7.26 (m, 5H), 7.22–7.17 (m, 3H), 6.94–6.87 (m, 2H), 6.42 (s, 1H), 4.01 (q, $J$ = 7.5 Hz, 1H), 3.85 (s, 3H), 1.76 (s, 3H), 1.41 (d, $J$ = 6.5 Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 157.3, 142.3, 138.9, 133.8, 129.0, 128.0, 127.4, 127.0, 125.8, 124.3, 120.1, 110.6, 55.5, 40.3, 19.2, 17.7; IR (neat): 3650, 1507, 1265, 738 cm$^{-1}$; MS m/z (%): 252/253 (80/15) [M$^+$], 237/238 (77/12) [M$^+$–Me], 129 (99), 91/92 (100/10); HRMS (APCI) found 270.1845, Calcd for C$_{18}$H$_{20}$O: [M–H] 270.1852.

(E)-(3-(4-Methoxyphenyl)prop-1-ene-1,3-diyl)dibenzene (3ea).
Yield: 85%, yellow oil. TLC: Rf = 0.27 (hexane/toluene = 2/1). 
$^1$H NMR (CDCl$_3$) $\delta$ 7.38–7.28 (m, 6H), 7.24–7.14 (m, 6H), 6.86 (d, $J$ = 6.5 Hz, 1H), 4.21 (dd, $J$ = 7.5, 16 Hz, 1H), 6.33 (d, $J$ = 16.5 Hz, 1H), 4.85 (d, $J$ = 7.5 Hz, 1H), 3.80 (s, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 158.1, 143.8, 137.3, 135.6, 132.9, 131.1, 129.6, 128.6, 128.5, 128.4, 127.2, 126.4, 126.3, 113.8, 55.2, 53.3; IR (neat): 3026, 1507, 1248, 1176, 1035, 699 cm$^{-1}$; MS m/z (%): 302/303 (25/5) [M$^+$]; HRMS (APCI) found 301.1581, Calcd for C$_{22}$H$_{20}$O [M+H]$^+$ 301.1587.

1-(Cyclohex-2-enyl)-4-methoxybenzene (3fa).
Yield: 25%, colorless oil. TLC: Rf = 0.25 (hexane/ethyl acetate = 30/1). 
$^1$H NMR (CDCl$_3$) $\delta$ 7.16–7.13 (m, 2H), 6.87–6.84 (m, 2H), 5.90–5.87 (m, 1H), 5.71–5.70 (m, 1H), 3.80 (s, 3H), 3.38–3.36 (m,1H), 2.11–2.00 (m,3H), 1.73–1.72 (m,1H), 1.66–1.53 (m,2H); $^{13}$C NMR (CDCl$_3$) $\delta$ 157.9, 138.8, 130.5, 128.6, 128.1, 113.7, 55.2, 41.0, 32.7, 25.0, 21.1; IR (neat): 2930, 1507, 1245, 1176, 1036, 827 cm$^{-1}$; MS m/z (%): 188/189 (100/14) [M$^+$]; HRMS (APCI) found 189.1272, Calcd for C$_{13}$H$_{18}$O [M+H]$^+$ 189.1274.
1-(Cyclohex-2-enyl)-2-methoxybenzene (3fa').

Yield: 25%, colorless oil. TLC: R$_f$ = 0.67 (hexane/ethyl acetate = 30/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.21–7.18 (m, 2H), 6.94–6.91 (m, 1H), 6.87 (d, $J$ = 8.0 Hz, 1H), 5.94–5.90 (m,1H), 5.69–5.66 (m,1H), 3.85 (s, 3H), 2.09–1.99 (m,4H), 1.69–1.61 (m,2H), 1.53–1.50 (m,1H); $^{13}$C NMR (CDCl$_3$) $\delta$ 156.9, 134.5, 130.3, 128.4, 128.3, 126.9, 120.3 110.2, 55.3, 34.2, 30.1, 25.2, 21.0; IR (neat): 2930, 1490, 1239, 1109, 1031, 754 cm$^{-1}$; MS m/z (%): 188/189 (100/12) [M$^+$]; HRMS (APCI) found 189.1272, Calcd for C$_{13}$H$_{16}$O [M+H]$^+$ 189.1274.

(E)-1-Methoxy-4-(pent-3-en-2-yl)benzene (3ga).

Yield: 64%, colorless oil. TLC: R$_f$ = 0.53 (hexane/toluene = 4/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.14–7.12 (m, 2H), 6.86–6.84 (m, 2H), 5.06–5.57 (m, 1H), 5.47–5.42 (m, 1H), 3.79 (s, 3H), 3.40–3.34 (m, 1H), 1.68 (d, $J$ = 3.0 Hz, 3H), 1.31 (d, $J$ = 7.5 Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 157.8, 138.6, 136.6, 128.0, 123.3, 113.7, 55.2, 41.5, 21.6, 17.9; IR (neat): 2963, 1512, 1247, 1178, 1038, 830 cm$^{-1}$; MS m/z (%): 176/177 (50/8) [M$^+$]; HRMS (APCI) found 177.1272, Calcd for C$_{12}$H$_{16}$O [M+H]$^+$ 177.1274.

1-Methoxy-4-(1-phenylethyl)benzene (3ha).

Yield: 71%, colorless oil. TLC: R$_f$ = 0.55 (hexane/toluene = 2/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.30–7.27 (m, 2H), 7.23–7.21 (m, 2H), 7.20–7.18 (m, 1H), 7.17–7.13 (m, 2H), 6.85–6.82 (m, 2H), 4.15 (q, $J$ = 7 Hz, 1H), 3.78 (s, 3H), 1.62 (d, $J$ = 7 Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 157.9, 146.8, 138.6, 128.5, 128.3, 127.6, 125.9, 113.7, 55.2, 43.9, 22.0; IR (neat): 2965, 1506, 1246, 1177, 1027, 832, 699 cm$^{-1}$; MS m/z (%): 212/213 (47/8) [M$^+$], 197/198 (100/16) [M$^+$–Me]; HRMS (APCI) found 230.1539, Calcd for C$_{15}$H$_{16}$O [M+NH$_4$]$^+$ 230.1539.

1-Methoxy-2-(1-phenylethyl)benzene (3ha').

Yield: 18%, colorless oil. TLC: R$_f$ = 0.73 (hexane/toluene = 2/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.28–7.23 (m, 4H), 7.19–7.14 (m, 3H), 6.92–6.89 (m, 1H), 6.84 (d, $J$ = 8.0 Hz, 1H), 4.57 (q, $J$ = 7.5 Hz, 1H), 3.77 (s,3H), 1.58 (d, $J$ = 3.5 Hz, 1H); $^{13}$C NMR (CDCl$_3$) $\delta$ 156.9, 146.4, 135.0, 128.1, 127.7, 127.0, 125.7, 120.5, 110.7, 109.8, 55.4, 37.4, 20.9; IR (neat): 2965, 1490, 1242, 1124, 1026, 752, 699 cm$^{-1}$; MS m/z (%): 212/213 (42/8) [M$^+$], 197/198 (51/8) [M$^+$–Me], 91/92 (100/8); HRMS (APCI) found 230.1539, Calcd for C$_{15}$H$_{16}$O [M+NH$_4$]$^+$ 230.1539.
1-(1-(4-Methoxyphenyl)ethyl)-2-methylbenzene (3ia).

Yield: 84%, yellow oil. TLC: Rf = 0.33 (hexane/toluene = 5/1). 1H NMR (CDCl3) δ 7.27 (d, J = 6.5 Hz, 1H), 7.22–7.19 (m, 1H), 7.14–7.13 (m, 2H), 7.09–7.07 (m, 2H), 6.83–6.80 (m, 2H), 4.28 (q, J = 7.0 Hz, 1H), 3.78 (s, 3H), 2.25 (s, 3H), 1.59 (d, J = 7.0 Hz, 3H); 13C NMR (CDCl3) δ 157.6, 144.2, 138.3, 136.0, 130.4, 128.5, 126.0, 126.0, 113.6, 55.2, 40.1, 22.2, 19.7; IR (neat): 2965, 1511, 1246, 1177, 1031, 831, 770 cm⁻¹; MS m/z (%): 226/227 (44/7) [M⁺], 211/212 (100/17) [M⁺–Me]; HRMS (APCI) found 244.1692, Calcd for C16H18O [M+NH₄⁺]⁺ 244.1696.

1-Methoxy-2-(1-o-tolylethyl)benzene (3ia').

Yield: 8%, colorless oil. TLC: Rf = 0.53 (hexane/toluene = 5/1). 1H NMR (CDCl3) δ 7.27 (d, J = 6.5 Hz, 1H), 7.19–7.15 (m, 2H), 7.13–7.09 (m, 2H), 6.99 (d, J = 7.5 Hz, 1H), 6.87–6.84 (m, 1H), 4.69 (q, J = 7.0 Hz, 1H), 3.81 (s, 3H), 2.22 (s, 3H), 1.53 (d, J = 7.0 Hz, 3H); 13C NMR (CDCl3) δ 156.7, 144.3, 136.3, 134.8, 130.1, 127.5, 126.8, 126.5, 125.7, 125.7, 120.5, 110.3, 55.4, 33.7, 20.6, 19.3; IR (neat): 2964, 2930, 1490, 1459, 1437, 1241, 1030, 752 cm⁻¹; MS m/z (%): 226/227 (45/8) [M⁺], 211/212 (40/7) [M⁺–Me], 105/106 (100/9); HRMS (APCI) found 244.1693, Calcd for C16H18O [M+NH₄⁺]⁺ 244.1696.

1-(1-(4-Methoxyphenyl)ethyl)-3-methylbenzene (3ja).

Yield: 72%, yellow oil. TLC: Rf = 0.34 (hexane/toluene = 5/1). 1H NMR (CDCl3) δ 7.20–7.14 (m, 3H), 7.03–6.99 (m, 3H), 6.85–6.83 (m, 2H), 4.08 (q, J = 7.5 Hz, 1H), 3.79 (s, 3H), 2.39 (m, 3H), 1.61 (d, J = 7.5 Hz, 3H); 13C NMR (CDCl3) δ 157.7, 146.7, 138.6, 137.8, 128.5, 128.3, 128.2, 126.7, 124.5, 113.7, 55.2, 43.9, 22.1, 21.5; IR (neat): 2964, 1609, 1511, 1247, 1178, 1034, 831 cm⁻¹; MS m/z (%): 226/227 (38/8) [M⁺], 211/212 (100/16) [M⁺–Me]; HRMS (APCI) found 244.1692, Calcd for C16H18O [M+NH₄⁺]⁺ 244.1696.

1-Methoxy-2-(1-m-tolylethyl)benzene (3ja').

Yield: 18%, yellow oil. TLC: Rf = 0.48 (hexane/toluene = 5/1). 1H NMR (CDCl3) δ 7.21–7.16 (m, 3H), 7.08–7.07 (m, 2H), 7.00 (d, J = 7.0 Hz, 1H), 6.93 (t, J = 7.5 Hz, 1H), 6.87 (d, J = 7.5 Hz, 1H), 4.57 (q, J = 7.5 Hz, 1H), 3.81 (s, 3H), 2.33 (s, 3H), 1.59 (s, 3H); 13C NMR (CDCl3) δ 156.7, 146.2, 137.5, 135.0, 128.5, 127.9, 127.7, 127.6, 126.9, 126.5, 124.7, 120.5, 110.5, 55.4, 37.2, 21.5, 20.9; IR (neat): 2965, 2931, 1490, 1242, 1123, 1030, 752, 701 cm⁻¹; MS m/z (%): 226/227 (27/4) [M⁺], 211/212 (32/6) [M⁺–Me], 105/106 (100/9); HRMS (APCI) found 244.1692, Calcd for C16H18O [M+NH₄⁺]⁺ 244.1696.
1-Methoxy-4-(1-p-tolylethyl)benzene (3ka).

Yield: 87%, colorless oil. TLC: Rₜ = 0.32 (hexane/toluene = 5/1). ¹H NMR (CDCl₃) δ 7.15–7.08 (m, 6H), 6.84–6.83 (m, 2H), 4.08 (q, J = 7.5 Hz, 1H), 3.79 (s, 3H), 2.32 (s, 3H), 1.61 (d, J = 7.5 Hz, 1H); ¹³C NMR (CDCl₃) δ 157.7, 143.8, 138.8, 135.4, 129.0, 128.4, 127.4, 113.7, 55.2, 43.5, 22.1, 21.0; IR (neat): 2964, 2930, 1509, 1246, 1177, 1034, 834, 814 cm⁻¹; MS m/z (%): 226/227 (36/5) [M⁺], 211/212 (100/16) [M⁺–Me]; HRMS (APCI) found 244.1693, Calcd for C₁₆H₁₈O [M+NH₄]⁺ 244.1696.

1-Methoxy-2-(1-p-tolylethyl)benzene (3ka').

Yield: 8%, yellow oil. TLC: Rₜ = 0.47 (hexane/toluene = 5/1). ¹H NMR (CDCl₃) δ 7.19–7.13 (m, 4H), 7.08 (d, J = 8.0 Hz, 2H), 6.90 (t, J = 8.0 Hz, 1H), 6.85 (d, J = 24 Hz, 1H), 4.55 (q, J = 7.5 Hz, 1H), 3.79 (s, 3H), 2.31 (s, 3H), 1.57 (d, J = 7.5 Hz, 3H); ¹³C NMR (CDCl₃) δ 156.7, 143.3, 135.1, 128.8, 128.8, 127.6, 127.6, 126.9, 120.5, 110.5, 55.4, 55.4, 36.9, 21.0; IR (neat): 2964, 2931, 1491, 1241, 752 cm⁻¹; MS m/z (%): 226 (27) [M⁺], 211 (38) [M⁺–Me], 105/106 (100/10); HRMS (APCI) 244.1693, Calcd for C₁₆H₁₈O [M+NH₄]⁺ 244.1696.

4,4'-(Ethane-1,1-diyl)bis(methoxybenzene) (3la).

Yield: 73%, pale yellow solid. Mp. 71 °C. TLC: Rₜ = 0.39 (hexane/ethyl acetate = 20/1). ¹H NMR (CDCl₃) δ 7.12 (d, J = 8.5 Hz, 4H), 6.82 (d, J = 7.0 Hz, 4H), 4.06 (q, J = 7.0 Hz, 1H), 3.78 (s, 6H), 1.58 (d, J = 7.5 Hz, 3H); ¹³C NMR (CDCl₃) δ 157.7, 138.9, 128.4, 113.6, 55.2, 43.0, 22.2; IR (KBr): 2960, 1606, 1511, 1378, 1262, 1173, 1122, 1026, 825, 752, 592, 549 cm⁻¹; MS m/z (%): 242/243 (26/4) [M⁺], 227/228 (100/17) [M⁺–Me]; HRMS (APCI) found 260.1642, Calcd for C₁₆H₁₈O₂ [M+NH₄]⁺ 260.1645.

1-Bromo-4-(1-(4-methoxyphenyl)ethyl)benzene (3ma).

Yield: 35%, colorless oil. TLC: Rₜ = 0.29 (hexane/toluene = 5/1). ¹H NMR (CDCl₃) δ 7.39 (d, J = 8.5 Hz, 2H), 7.11–7.06 (m, 4H), 6.84–6.82 (m, 2H), 4.06 (q, J = 7.0 Hz, 1H), 3.78 (s, 3H), 1.58 (d, J = 7.5 Hz, 3H); ¹³C NMR (CDCl₃) δ 157.9, 145.8, 137.8, 131.4, 129.3, 128.4, 119.7, 113.8, 55.2, 43.4, 21.9; IR (neat): 2965, 1511, 1247, 1177, 1008, 826 cm⁻¹; MS m/z (%): 290/291/292/293 (36/34/4/34) [M⁺], [M⁺–Me], 275/276/277/278 (100/14/97/14); HRMS (APCI) found 289.0217, Calcd for C₁₆H₁₃BrO [M+H⁺] 289.0234.
1-(1-(4-Bromophenyl)ethyl)-2-methoxybenzene (3ma').

Yield: 10%, colorless oil. TLC: R_f = 0.58 (hexane/toluene = 5/1). ^1H NMR (CDCl_3) δ 7.37–7.35 (m, 2H), 7.19 (t, J = 8.5 Hz, 1H), 7.14 (d, J = 8.5 Hz, 1H), 7.11–7.09 (m, 2H), 6.91 (t, J = 7.5 Hz, 1H), 6.84 (d, J = 7.0 Hz, 1H), 4.45 (q, J = 7.0 Hz, 1H), 3.76 (s, 3H), 1.55 (d, J = 7.5 Hz, 3H); ^13C NMR (CDCl_3) δ 156.7, 145.5, 134.1, 131.1, 129.4, 127.4, 127.3, 120.5, 119.3, 110.6, 55.3, 37.0, 20.7; IR (neat): 2965, 1490, 1243, 1009, 829, 752 cm⁻¹; MS m/z (%): 290/291/292/293 (50/10/49/10) [M⁺], 275/276/277/278 (65/9/61/9) [M⁺–Me], 169/170/171/172 (100/8/98/7); HRMS (APCI) found 308.0638, Calcd for C_{15}H_{15}BrO [M+NH₄]^⁺ 308.0645.

1-Tert-butyl-4-(1-(4-methoxyphenyl)ethyl)benzene (3na).

Yield: 81%, yellow solid. Mp. 53 °C. TLC: R_f = 0.23 (hexane/toluene = 5/1). ^1H NMR (CDCl_3) δ 7.31–7.29 (m, 2H), 7.17–7.14 (m, 4H), 6.85–6.83 (m, 2H), 4.09 (q, J = 7.5 Hz, 1H), 3.79 (s, 3H), 1.62 (d, J = 7.0 Hz, 3H), 1.30 (s, 9H); ^13C NMR (CDCl_3) δ 157.7, 148.6, 143.6, 138.8, 128.5, 127.0, 125.2, 113.6, 55.2, 43.4, 34.3, 31.4, 22.1; IR (KBr): 2965, 1609, 1453, 1364, 1246, 1177, 1112, 1016, 842, 581 cm⁻¹; MS m/z (%): 268/269 (26/5) [M⁺], 253/254 (100/18) [M⁺–Me]; HRMS (APCI) found 286.2160, Calcd for C_{19}H_{24}O [M+NH₄]^⁺ 286.2165.

1-(1-(4-Tert-butylphenyl)ethyl)-2-methoxybenzene (3na').

Yield: 10%, pink oil. TLC: R_f = 0.38 (hexane/toluene = 5/1). ^1H NMR (CDCl_3) δ 7.29–7.26 (m, 2H), 7.19–7.13 (m, 4H), 6.91–6.84 (m, 2H), 4.57 (q, J = 7.0 Hz, 1H), 3.80 (s, 3H), 1.57 (d, J = 7.5 Hz, 3H), 1.29 (s, 9H); ^13C NMR (CDCl_3) δ 148.3, 143.1, 135.2, 127.7, 127.3, 126.9, 125.1, 124.9, 120.5, 110.5, 55.4, 36.6, 34.3, 31.4, 20.9; IR (neat): 2962, 1507, 1490, 1241 cm⁻¹; MS m/z (%): 268/269 (59/11) [M⁺], 253/254 (92/22) [M⁺–Me], 91 (100); HRMS (APCI) found 286.2160, Calcd for C_{19}H_{24}O [M+NH₄]^⁺ 286.2165.

1-(1-(4-Methoxyphenyl)ethyl)naphthalene (3oa).

Yield: 83%, pink solid. Mp. 75 °C. TLC: R_f = 0.19 (hexane/toluene = 5/1). ^1H NMR (CDCl_3) δ 8.06–8.04 (m, 1H), 7.86–7.84 (m, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.48–7.40 (m, 4H), 7.16–7.12 (m, 2H), 6.82–6.78 (m, 2H), 4.89 (q, J = 7.0 Hz, 1H), 3.76 (s, 3H), 1.75 (d, J = 7.0 Hz, 3H); ^13C NMR (CDCl_3) δ 157.7, 141.9, 139.8, 133.9, 131.6, 128.7, 128.5, 126.9, 125.8, 125.4, 125.3, 124.1, 124.0, 113.7, 55.2, 39.7, 22.7; IR (KBr): 2961, 1606, 1512, 1439, 1396, 1376, 1285, 1250,
1177, 1030, 837, 804, 777, 562, 447 cm⁻¹; MS m/z (%): 262/263 (47/10) [M⁺], 247/248 (100/20) [M⁺–Me]; HRMS (APCI) found 280.1691, Calcd for C₁₉H₁₈O [M+NH₄]⁺ 280.1696.

1-(1-(2-Methoxyphenyl)ethyl)naphthalene (3oa').

Yield: 10%, pink solid. Mp. 76 °C. TLC: Rᵣ = 0.30 (hexane/toluene = 5/1). ¹H NMR (CDCl₃) δ 8.00 (d, J = 9.0 Hz, 1H), 7.82 (d, J = 7.0 Hz, 1H), 7.73 (d, J = 7.5 Hz, 1H), 7.49–7.45 (m, 2H), 7.42–7.39 (m, 2H), 7.14 (t, J = 8.0 Hz, 1H), 6.91 (t, J = 7.5 Hz, 2H), 6.77 (t, J = 7.5 Hz, 1H), 5.32 (q, J = 7.0 Hz, 1H), 3.89 (s, 3H), 1.67 (d, J = 7.0 Hz, 3H); ¹³C NMR (CDCl₃) δ 156.2, 141.9, 135.3, 133.8, 131.8, 128.5, 127.8, 127.0, 126.7, 125.7, 125.3, 125.2, 124.1, 123.8, 120.6, 110.3, 55.5, 33.0, 21.0; IR (KBr): 2964, 1490, 1245, 1026, 782, 569 cm⁻¹; MS m/z (%): 262/263 (41/9) [M⁺], 247/248 (36/6) [M⁺–Me], 141/142 (100/13); HRMS (APCI) found 280.1691, Calcd for C₁₉H₁₈O [M+NH₄]⁺ 280.1696.

1-(4-Methoxyphenyl)-1,2,3,4-tetrahydronaphthalene (3pa).

Yield: 58%, yellow oil. TLC: Rᵣ = 0.45 (hexane/ethyl acetate = 30/1). ¹H NMR (CDCl₃) δ 7.14–7.10 (m, 2H), 7.05–7.01 (m, 3H), 6.87–6.82 (m, 3H), 4.08 (t, J = 7.0 Hz, 1H), 3.80 (s, 3H), 2.90–2.83 (m, 2H), 2.14–2.12 (m, 1H), 1.90–1.82 (m, 2H), 1.80–1.77 (m, 1H); ¹³C NMR (CDCl₃) δ 157.8, 139.7, 139.7, 137.5, 130.1, 129.7, 128.9, 125.8, 125.6, 113.6, 55.2, 44.7, 33.3, 29.8, 20.9; IR (neat): 2931, 1510, 1245, 1175, 1036, 828, 740 cm⁻¹; MS m/z (%): 238/239 (30/4) [M⁺], 130/131 (100/12); HRMS (APCI) found 239.1423, Calcd for C₁₇H₁₈O [M+H]⁺ 239.1430.

1-(2-Methoxyphenyl)-1,2,3,4-tetrahydronaphthalene (3pa').

Yield: 9%, colorless oil. TLC: Rᵣ = 0.52 (hexane/ethyl acetate = 30/1). ¹H NMR (CDCl₃) δ 7.19–7.16 (m, 1H), 7.14–7.09 (m, 2H), 7.04–7.01 (m, 1H), 6.91–6.90 (m, 1H), 6.85–6.81 (m, 2H), 6.75–6.74 (m, 1H), 4.59 (t, J = 6.0 Hz, 1H), 3.86 (s, 3H), 2.92–2.84 (m, 2H), 2.09–2.05 (m, 1H), 1.89–1.82 (m, 2H), 1.78–1.74 (m, 1H); ¹³C NMR (CDCl₃) δ 157.0, 139.7, 138.0, 135.8, 130.2, 130.0, 128.8, 126.9, 125.6, 120.3, 110.3, 55.4, 38.0, 30.5, 29.8, 20.7; IR (neat): 2931, 1490, 1240, 1031, 752 cm⁻¹; MS m/z (%): 382/239 (46/9) [M⁺], 134/135 (100/10); HRMS (APCI) found 256.1691, Calcd for C₁₇H₁₈O [M+NH₄]⁺ 256.1696.
((4-Methoxyphenyl)methylene)dibenzene (3qa).

Yield: 87%, colorless oil. TLC: R_f = 0.31 (hexane/toluene = 5/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.30–7.26 (m, 4H), 7.23–7.20 (m, 2H), 7.13–7.10 (m, 4H), 7.05–7.02 (m, 2H), 6.85–6.82 (m, 2H), 5.51 (s, 1H), 3.79 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 158.0, 144.2, 136.1, 130.3, 129.4, 128.3, 126.2, 113.6, 56.0, 55.2; IR (neat): 3025, 1510, 1248, 1178, 1032, 699 cm\(^{-1}\); MS m/z (%): 274/275 (100/22) [M\(^+\)], 197/198 (86/12) [M\(^+\)-Ph], 121/122 (100/14); HRMS (APCI) found 275.1422, Calcd for C\(_{20}\)H\(_{18}\)O [M+H]\(^+\) 275.1430.

((2-Methoxyphenyl)methylene)dibenzene (3qa').

Yield: 11%, white powder. TLC: R_f = 0.20 (hexane/toluene = 5/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.27–7.24 (m, 4H), 7.23 –7.18 (m, 3H), 7.09 (d, \(J = 7.5\) Hz, 4H), 6.88–6.866 (m, 3H), 5.93 (s, 1H), 3.71 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 157.1, 143.9, 132.6, 130.3, 129.4, 128.1, 127.5, 126.0, 120.2, 110.7, 55.6, 49.6; IR (KBr): 2930, 1596, 1488, 1452, 1330, 1289, 1246, 1160, 1024, 753, 705, 611 cm\(^{-1}\); MS m/z (%): 274/275 (100/22) [M\(^+\)], 259/260 (46/8) [M\(^+\)-Me], 197/198 (86/12) [M\(^+\)-Ph], 165 (62), 91 (69); HRMS (APCI) found 292.1692, Calcd for C\(_{20}\)H\(_{18}\)O [M+NH\(_4\)]\(^+\) 292.1696.

(1-(4-Methoxyphenyl)propane-1,3-diyl)dibenzene (3ra).

Yield: 84%, colorless oil. TLC: R_f = 0.21 (hexane/toluene = 2/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.30–7.22 (m, 6H), 7.20–7.13 (m, 6H), 6.84–6.82 (m, 2H), 3.87 (t, \(J = 15.5\) Hz, 1H), 3.78 (s,3H), 2.57 (t, \(J = 15.5\) Hz, 2H), 2.37–2.32 (m, 2H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 157.8, 145.2, 142.1, 136.9, 128.8, 128.4, 128.4, 128.3, 127.7, 126.0, 125.8, 126.0, 125.8, 113.8, 55.2, 49.7, 37.5, 34.1; IR (neat): 2930, 1509, 1248, 698 cm\(^{-1}\); MS m/z (%): 302/303 (10/3) [M\(^+\)], 197 (100); HRMS (APCI) found 320.2002, Calcd for C\(_{22}\)H\(_{22}\)O [M+NH\(_4\)]\(^+\) 320.2009.

(1-(2-Methoxyphenyl)propane-1,3-diyl)dibenzene (3ra').

Yield: 16%, colorless oil. TLC: R_f = 0.40 (hexane/toluene = 2/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.29–7.24 (m, 7H), 7.18–7.13 (m, 5H), 6.92 (t, \(J = 7.5\) Hz, 1H), 6.84 (d, \(J = 8.0\) Hz, 1H), 4.43 (t, \(J = 8.0\) Hz, 2H), 3.76 (s, 3H), 2.60–2.56 (m, 2H), 2.36–2.32 (m, 2H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 157.1, 144.8, 142.5, 133.3, 128.4, 128.2, 128.2, 127.6, 127.1, 125.8, 125.6, 120.6, 110.8, 55.4, 42.9, 36.7, 34.2; IR (neat): 2933, 1490, 1242, 1030, 752, 698 cm\(^{-1}\); MS m/z (%): 302/303 (12/2) [M\(^+\)], 197 (40), 91 (73), 44 (100); HRMS (APCI) found 320.2004, Calcd for C\(_{22}\)H\(_{22}\)O
1-Methoxy-4-(4-phenylbut-3-yn-2-yl)benzene (3sa).

Yield: 31%, yellow oil. TLC: R_f = 0.21 (hexane/toluene = 5/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.45–7.43 (m, 2H), 7.37 (d, \(J = 8.0\) Hz, 1H), 7.30 (m, 3H), 6.89 (d, \(J = 9.0\) Hz, 2H), q (d, \(J = 7.0\) Hz, 1H), 3.81 (s, 1H), 1.56 (d, \(J = 7.0\) Hz, 1H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 158.3, 135.5, 131.6, 128.2, 127.9, 127.7, 123.8, 113.9, 92.9, 82.2, 55.3, 31.6, 24.6; IR (neat): 2973, 1506, 1248, 1176, 1034, 831, 757, 692 cm\(^{-1}\); MS \(m/z\) (%): 236/237 (43/8) [M\(^+\)], 221/222 (100/16) [M\(^+\)–Me]; HRMS (APCI) found 237.1269, Calcd for C\(_{17}\)H\(_{16}\)O [M+H]\(^+\) 237.1274.

1-Methoxy-2-(4-phenylbut-3-yn-2-yl)benzene (3sa').

Yield: 19%, yellow oil. TLC: R_f = 0.41 (hexane/toluene = 5/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.66 (dd, \(J = 2.0, 9.5\) Hz, 1H), 7.47–7.45 (m, 2H), 7.31–7.28 (m, 3H), 7.25–7.22 (m, 1H), 6.99 (dd, \(J = 7.5, 7.5, 1.0\) Hz, 1H), 6.88 (dd, \(J = 1.0, 8.0\) Hz, 1H), 4.40 (q, \(J = 7.5\) Hz, 1H), 3.87 (s, 3H), 1.51 (d, \(J = 7.0\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 156.1, 131.6, 128.1, 127.9, 127.7, 127.6, 124.0, 120.8, 110.4, 93.2, 81.7, 55.4, 26.1, 22.9; IR (neat): 2930, 1490, 1243, 1030, 754, 691 cm\(^{-1}\); MS \(m/z\) (%): 236/237 (27/3) [M\(^+\)], 221/222 (100/17) [M\(^+\)–Me]; HRMS (APCI) found 254.1531, Calcd for C\(_{17}\)H\(_{16}\)O [M+NH\(_4\)]\(^+\) 254.1539.

1-Methoxy-4-(2-phenylpropan-2-yl)benzene (3ta).

Yield: 28%, colorless oil. TLC: R_f = 0.22 (hexane/ethyl acetate = 30/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.30–7.24 (m, 4H), 7.19–7.15 (m, 3H), 6.84–6.81 (m, 2H), 3.80 (s, 3H), 3.78 (s, 3H), 1.68 (s, 6H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 157.4, 151.0, 142.9, 127.9, 127.8, 126.7, 125.5, 113.2, 77.3, 77.0, 76.7, 55.2, 42.3, 30.9; IR (neat): 2967, 1507, 1496, 1252, 1182, 1036, 830, 700 cm\(^{-1}\); MS \(m/z\) (%): 226/227 (30/4) [M\(^+\)], 211/222 (100/17) [M\(^+\)–Me]; HRMS (APCI) found 244.1695, Calcd for C\(_{16}\)H\(_{18}\)O [M+NH\(_4\)]\(^+\) 244.1696.

1,4-Dimethoxy-2-(1-phenylethyl)benzene (3hb).

Yield: 70%, brown oil. TLC: R_f = 0.38 (hexane/ethyl acetate = 20/1). \(^1\)H NMR (CDCl\(_3\)) \(\delta\) 7.29–7.25 (m, 4H), 7.18–7.15 (m, 3H), 6.79 (s, 1H), 6.77 (d, \(J = 5.0\) Hz, 1H), 6.70 (dd, \(J = 3.0, 9.0\) Hz, 1H), 4.56 (q, \(J = 7.0\) Hz, 1H), 3.74 (s, 3H), 3.73 (s, 3H), 1.58 (d, \(J = 7.0\) Hz, 3H); \(^{13}\)C NMR (CDCl\(_3\)) \(\delta\) 153.5, 151.2, 146.0, 136.3, 128.1, 127.6, 125.7, 114.7, 111.5, 110.4, 56.1, 55.5, 37.5, 20.8; IR (neat): 2964, 1496, 1218, 1046, 699 cm\(^{-1}\); MS \(m/z\) (%): 242/243 (84/12) [M\(^+\)], 227/228 (42/5) [M\(^+\)–Me], 91/92 (100/9); HRMS
2,4-Dimethoxy-1-(1-phenylethyl)benzene (3hc).

Yield: 72%, colorless oil. TLC: Rf = 0.21 (hexane/toluene = 2/1). 1H NMR (CDCl3) δ 7.28–7.22 (m, 4H), 7.17–7.14 (m, 1H), 7.05–7.04 (m, 1H), 6.46–6.43 (m, 2H), 4.48 (q, J = 7.0 Hz, 1H), 3.79 (s, 3H), 3.75 (s, 3H), 1.56 (d, J = 7.0 Hz, 3H); 13C NMR (CDCl3) δ 159.0, 157.7, 146.7, 128.0, 127.9, 127.6, 127.5, 125.6, 103.9, 98.6, 55.4, 55.3, 37.0, 21.0; IR (neat): 2962, 1612, 1503, 1208, 699 cm⁻¹; MS m/z (%): 242/243 (51/11) [M⁺], 227/228 (91/16) [M⁺–Me], 91/92 (100/9); HRMS (APCI) found 243.1375, Calcd for C16H18O2 [M+H]+ 243.1380.

1,3-Dimethoxy-2-(1-phenylethyl)benzene (3hc').

Yield: 14%, colorless oil. TLC: Rf = 0.36 (hexane/toluene = 2/1). 1H NMR (CDCl3) δ 7.29–7.26 (m, 2H), 7.23–7.20 (m, 2H), 7.15–7.09 (m, 2H), 6.54 (d, J = 8.5 Hz, 2H), 4.86 (q, J = 7.5 Hz, 1H), 3.70 (s, 6H), 1.67 (d, J = 7.5 Hz, 3H); 13C NMR (CDCl3) δ 158.5, 146.3, 127.5, 127.3, 124.9, 123.3, 104.8, 55.8, 33.2, 17.4; IR (neat): 2934, 1592, 1474, 1244, 1113 cm⁻¹; MS m/z (%): 242/243 (42/8) [M⁺], 227/228 (48/8) [M⁺–Me], 91/92 (100/8); HRMS (APCI) found 243.1377, Calcd for C16H18O2 [M+H]+ 243.1380.

1,3,5-Trimethoxy-2-(1-phenylethyl)benzene (3hd).

Yield: 83%, brown solid. Mp. 65 °C. TLC: Rf = 0.38 (hexane/ethyl acetate = 30/1). 1H NMR (CDCl3) δ 7.28–7.26 (m, 3H), 7.21 (t, J = 7.5 Hz, 2H), 7.11–7.08 (m, 1H), 6.13 (s, 1H), 4.75 (q, J = 7.0 Hz, 1H), 3.79 (s, 3H), 3.69 (s, 6H), 1.64 (d, J = 7.0 Hz, 3H); 13C NMR (CDCl3) δ 159.4, 159.0, 146.6, 127.5, 127.2, 124.8, 115.8, 91.4, 55.7, 55.2, 32.9, 17.7; IR (KBr): 2939, 2836, 1493, 1447, 1441, 1327, 1227, 1120 cm⁻¹; MS m/z (%): 272/273 (38/8) [M⁺], 257/258 (64/10) [M⁺–Me], 91/92 (100/9); HRMS (APCI) found 273.1478, Calcd for C17H20O3 [M+H]+ 273.1485.

1,3,5-Trimethyl-2-(1-phenylethyl)benzene (3he).

Yield: 92%, colorless oil. TLC: Rf = 0.48 (hexane). 1H NMR (CDCl3) δ 7.28–7.25 (m, 2H), 7.19–7.15 (m, 3H), 6.83 (s, 2H), 4.65 (d, J = 5.0 Hz, 1H), 6.70 (dd, J = 3.0, 9.0 Hz, 1H), 4.56 (q, J = 7.0 Hz, 1H), 2.27 (s, 3H), 2.12 (brs, 6H), 1.66 (d, J = 7.0 Hz, 3H); 13C NMR (CDCl3) δ 145.4, 140.0, 136.5, 135.4, 129.9, 128.1, 126.8, 125.2, 37.8, 21.0, 20.7, 16.8; IR (neat): 2965, 1494, 1457, 702 cm⁻¹; MS m/z (%): 224/225 (58/12) [M⁺], 209/210 (100/18) [M⁺–Me]; HRMS (APCI) found 242.1898, Calcd for C17H20
2-(1-Phenylethyl)benzofuran (1:0.1 regiomixture) (3hf).

Yield: 94%, colorless oil. TLC: $R_f = 0.53$ (hexane/toluene = 30/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.86–7.84 (m, 0.9H), 7.75–7.73 (m, 0.1H), 7.69–7.67 (m, 0.1H), 7.58–7.56 (m, 1H), 7.38–7.19 (m, 8.8H), 4.45 (q, $J = 7.0$ Hz, 1H), 4.42 (q, $J = 7.0$ Hz, 0.1H), 1.79 (d, $J = 7.0$ Hz, 0.3H), 1.76 (d, $J = 7.5$ Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 145.7, 140.9, 140.8, 138.8, 128.8, 128.8, 127.7, 127.6, 127.0, 126.5, 124.4, 124.3, 124.0, 123.9, 123.3, 123.0, 122.7, 122.4, 121.7, 120.3, 41.7, 39.8, 23.0, 22.6; IR (neat): 2934, 1592, 1474, 1244, 1113 cm$^{-1}$; MS m/z (%): 238/239 (52/8) [M +], 223/224 (100/16) [M +–Me]; HRMS (APCI) found 256.1153, Calcd for C$_{16}$H$_{18}$O$_2$ [M+H]$^+$ 256.1154.

2-(1-Phenylethyl)benzofuran (3hg).

Yield: 61%, yellow oil. TLC: $R_f = 0.68$ (hexane/ethyl acetate = 20/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.58–7.56 (m, 1H), 7.48–7.46 (m, 1H), 7.42–7.36 (m, 4H), 7.34–7.24 (m, 3H), 6.52–6.51 (m, 1H), 4.34 (q, $J = 7.5$ Hz, 1H), 1.78 (d, $J = 7.5$ Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 162.1, 154.9, 143.3, 128.6, 128.6, 127.5, 126.7, 123.4, 122.4, 120.4, 111.0, 102.1, 39.6, 20.3; IR (neat): 2973, 1583, 1492, 1453, 1255, 699, 750 cm$^{-1}$; MS m/z (%): 222/223 (45/9) [M +], 207/208 (100/16) [M +–Me]; HRMS (APCI) found 223.1114, Calcd for C$_{16}$H$_{14}$O [M+H]$^+$ 223.1117.

4-(1-Phenylethyl)phenol (1:0.8 regiomixture) (3hh).

Yield: 59%, black oil. TLC: $R_f = 0.37$ (hexane/ethyl acetate = 5/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.32–7.24 (m, 5.9H), 7.22–7.16 (m, 4.0H), 7.13–7.07 (m, 2.9H), 6.94 (t, $J = 7.5$ Hz, 0.8H), 6.75–7.72 (m, 2.8H), 5.19 (s, 1H), 4.90 (s, 0.8H), 4.39 (q, $J = 7.0$ Hz, 0.8H), 4.09 (d, $J = 7.5$ Hz, 1H), 1.64–1.59 (m, 5.4H); $^{13}$C NMR (CDCl$_3$) $\delta$ 153.4, 153.2, 146.7, 145.3, 138.6, 132.0, 128.7, 128.6, 128.3, 127.9, 127.5, 127.4, 126.4, 125.9, 120.8, 115.9, 115.1, 43.9, 38.6, 22.0, 21.0; IR (neat): 3372, 1512, 1452, 1232, 581 cm$^{-1}$; MS m/z (%): 198/199 (52/7) [M$^+$], 183/184 (100/15) [M$^+$–Me]; HRMS (APCI) found 216.1381, Calcd for C$_{14}$H$_{14}$O [M+NH$_4$]$^+$ 216.1383.

2,4-Dimethyl-6-(1-phenylethyl)phenol (3hi).

Yield: 87%, black oil. TLC: $R_f = 0.55$ (hexane/ethyl acetate = 3/1). $^1$H NMR (CDCl$_3$) $\delta$ 7.32–7.19 (m, 5H), 6.92 (s, 1H), 6.84 (s, 1H), 4.39 (s, 1H), 4.30 (q, $J = 7.5$ Hz, 1H), 2.27 (s, 3H), 2.16 (s, 3H), 1.61 (d, $J = 7.0$ Hz, 3H); $^{13}$C NMR (CDCl$_3$) $\delta$ 149.4, 145.4, 131.1, 129.6, 129.3, 128.7, 127.5, 126.5, 126.0, 125.9, 120.8, 115.9, 115.1, 43.9, 38.6, 22.0, 21.0; IR (neat): 3372, 1512, 1452, 1232, 581 cm$^{-1}$; MS m/z (%): 198/199 (52/7) [M$^+$], 183/184 (100/15) [M$^+$–Me]; HRMS (APCI) found 216.1381, Calcd for C$_{14}$H$_{14}$O [M+NH$_4$]$^+$ 216.1383.
123.9, 39.1, 21.2, 20.7, 15.9; IR (neat): 3553, 2967, 1482, 1200, 700 cm⁻¹; MS m/z (%): 226/227 (70/12) [M⁺], 211/212 (100/16) [M⁺–Me]; HRMS (APCI) found 244.1693, Calcd for C₁₆H₁₈O [M+NH₄]^+ 244.1696.
$^1\text{H}$ NMR and $^{13}\text{C}$ NMR Spectra of Products

![Chemical Structure]

- 7.260
- 7.354
- 7.311
- 7.206
- 7.281
- 7.260
- 7.220
- 7.206
- 7.191
- 7.177
- 7.173
- 7.154
- 7.159
- 6.815
- 6.822
- 6.838

- 6.458
- 6.624
- 6.378
- 6.345
- 6.352
- 6.347
- 6.334
- 6.320

- 2.050
- 2.067
- 3.590
- 3.650
- 3.502
- 3.408
- 3.401