SUPPORTING INFORMATION

An Efficient Palladium-Catalyzed Synthesis of Cinnamaldehydes from Acrolein Diethyl Acetal and Aryl Iodides and Bromides.

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Melting points were determined with a Büchi apparatus and are uncorrected. Reaction products were purified on axially compressed columns, packed with SiO₂ 25-40 µm (Macherey Nagel), connected to a Gilson solvent delivery system and to a Gilson refractive index detector, and eluting with *n*-hexane/ethyl acetate mixtures. ¹H NMR (200 – 400 MHz), ¹³C NMR (50.3 – 100.6 MHz) and ¹⁹F (188.2 – 376.4 MHz) spectra were recorded with a Bruker Avance 200 and a Bruker Avance 400 spectrometer. IR spectra were recorded with a Jasco FT/IR-430 spectrometer.

Typical procedure for the preparation of cinnamaldehydes: To a stirred solution of *p*-iodoanisole (0.117 g, 0.5 mmol) in 2.0 mL of DMF were added acrolein diethyl acetal (0.229 mL, 1.5 mmol), ⁿBu₄NOAc (0.302 g, 1.0 mmol), K₂CO₃ (0.104 g, 0.75 mmol), KCl (0.037 g, 0.5 mmol), and Pd(OAc)₂ (0.003 g, 0.015 mmol). The mixture was stirred for 1.5 h at 90 °C. After cooling, 2 N HCl was slowly added and the reaction mixture was stirred at room temperature for 10 min. Then, it was diluted with ether and washed with water. The organic layer was dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by chromatography (silica gel, 35 g; *n*-hexane/ethylacetate 90/10 v/v) to give 0.071 g (88% yield) of *p*-methoxycinnamaldehyde **6a**: mp: 56-7 °C; lit. mp: 57-58.5 °C;¹ IR (KBr): 1667, 1250, 1018, 977 cm⁻¹; ¹H NMR (CDCl₃) δ 9.62 (d, *J*₁ = 7.8 Hz, 1H), 7.55-7.49 (m, 2H), 7.43 (d, *J* = 15.8 Hz, 1H), 6.98-6.93 (m, 2H), 6.61 (dd, *J*₁ = 15.8 Hz, *J*₂ = 7.8 Hz 1H), 3.86 (s, 3H); ¹³C NMR (CDCl₃) δ 194.0, 162.6, 153.1, 130.7, 127.1, 126.8, 114.9, 55.8; Anal calcd for C₁₀H₁₀O₂, C, 74.06; H, 6.21. Found C, 74.11, H, 6.24.

***m*-Methoxycinnamaldehyde 6b:** oil; IR (neat): 1679, 1273, 1044, 974 cm⁻¹; ¹H NMR (CDCl₃) δ 9.71 (d, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 15.9 Hz, 1H), 7.41-7.33 (m, 1H), 7.20-7.16 (m, 1H), 7.11-7.09 (m, 1H), 7.04-6.99 (m, 1H), 6.71 (dd, *J*₁ = 15.9 Hz, *J*₂ = 7.9 Hz, 1H), 3.86 (s, 3H); ¹³C NMR (CDCl₃) δ 193.7, 160.0, 152.7, 135.4, 130.1, 128.8, 121.2, 117.1, 113.3, 55.4; Anal calcd for C₁₀H₁₀O₂, C, 74.06; H, 6.21. Found C, 74.10, H, 6.19.

***o*-Methoxycinnamaldehyde 6c:** mp 41-2 °C; lit. mp: 44-46 °C;² IR (KBr) 1674, 1252, 1024, 976 cm⁻¹; ¹H NMR (CDCl₃) δ 9.71 (d, *J* = 7.9 Hz, 1H), 7.87 (d, *J* = 16.1, 1H), 7.60-7.40 (m, 2H), 7.06-6.96. (m, 2H), 6.82 (dd, *J*₁ = 16.1 Hz, *J*₂ = 7.9 Hz, 1H), 3.94 (s, 3H); ¹³C NMR (CDCl₃) δ 194.6, 158.3, 148.3, 132.7, 129.1, 128.9, 123.0, 120.9, 111.3, 55.6; Anal calcd for C₁₀H₁₀O₂, C, 74.06; H, 6.21. Found C, 74.08; H, 6.20.

***p*-Dimethylamminocinnamaldehyde 6d:** mp 138-9 °C; lit. mp: 140-141 °C;³ IR (KBr): 1661, 1370, 1190, 972 cm⁻¹; ¹H NMR (CDCl₃) δ 9.58 (d, *J* = 7.9 Hz, 1H), 7.46-7.43 (m, 2H), 7.36 (d, *J* = 15.6 Hz, 1H), 6.70-6.66 (m, 2H), 6.53 (dd, *J*₁ = 15.6 Hz, *J*₂ = 7.9 Hz, 1H), 3.04 (s, 6H); ¹³C NMR (CDCl₃) δ 194.1, 154.4, 1152.8, 130.9, 124.1, 122.1, 112.1, 40.5; Anal calcd for C₁₁H₁₃NO, C, 75.40; H, 7.48; N, 7.99. Found C, 75.32; H, 7.43; N, 8.03.

***p*-Methylcinnamaldehyde 6e:** mp 42-3 °C; lit. mp: 42 °C;⁴ IR (KBr) 1684, 980 cm⁻¹; ¹H NMR (CDCl₃) δ 9.66 (d, *J* = 7.7 Hz, 1H), 7.47-7.39 (m, 3H), 7.24-7.20 (m, 2H), 6.53 (dd, *J*₁ = 15.9 Hz, *J*₂

= 7.7 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (CDCl_3) δ 193.8, 152.9, 142.0, 131.3, 129.8, 128.5, 127.7, 21.6. Anal calcd for $\text{C}_{10}\text{H}_{10}\text{O}$, C, 82.16; H, 6.89. Found C, 82.20; H, 6.91.

***m*-Methylcinnamaldehyde 6f:** oil; IR (neat) 1680, 973 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.72 (d, J = 7.7 Hz, 1H), 7.47 (d, J = 15.9, 1H), 7.39-7.26 (m, 4H), 6.73 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR (CDCl_3) δ 193.7, 153.1, 138.8, 133.9, 132.2, 129.1, 129.0, 128.4, 125.7, 21.3. Anal calcd for $\text{C}_{10}\text{H}_{10}\text{O}$, C, 82.16; H, 6.89. Found C, 82.12; H, 6.88.

***o*-Methylcinnamaldehyde 6g:** oil; IR (neat) 1680, 972 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.74 (d, J = 7.7 Hz, 1H), 7.78 (d, J = 15.8 Hz, 1H), 7.61-7.59 (m, 1H), 7.36-7.32 (m, 1H), 7.28-7.25 (m, 2H), 6.67 (dd, J_1 = 15.8 Hz, J_2 = 7.7 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (CDCl_3) δ 193.9, 150.3, 138.0, 132.9, 131.2, 131.1, 129.7, 126.9, 126.7, 19.8. Anal calcd for $\text{C}_{10}\text{H}_{10}\text{O}$, C, 82.16; H, 6.89. Found C, 82.13; H, 6.93.

***p*-tert-Butylcinnamaldehyde 6h:** mp 61-2 $^{\circ}\text{C}$; lit. mp: 57-59 $^{\circ}\text{C}$; ^1IR (KBr) 1669, 970 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.73 (d, J = 7.7 Hz, 1H), 7.58-7.46 (m, 5H), 6.73 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H), 1.37 (s, 9H); ^{13}C NMR (CDCl_3) δ 193.8, 155.1, 152.8, 131.3, 128.4, 127.9, 126.1, 35.0, 31.1. Anal calcd for $\text{C}_{13}\text{H}_{16}\text{O}$, C, 82.94; H, 8.57. Found C, 82.98; H, 8.50.

3-(3,5-Dimethylphenyl)-propenal 6i: mp 40-41 $^{\circ}\text{C}$; IR (KBr) 1681, 968 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.69 (d, J = 7.7 Hz, 1H), 7.41 (d, J = 15.9 Hz, 1H), 7.19 (s, 2H), 7.09 (s, 1H), 6.70 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H), 2.36 (s, 1H); ^{13}C NMR (CDCl_3) δ 194.2, 153.7, 139.1, 134.4, 133.5, 128.7, 126.8, 21.6; Anal calcd for $\text{C}_{11}\text{H}_{12}\text{O}$, C, 82.46; H, 7.55. Found C, 82.51; H, 7.64.

Cinnamaldehyde 6j: oil; IR (neat) 1675, 973 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.73 (d, J = 7.7 Hz, 1H), 7.63-7.44 (m, 6H), 6.75 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.8, 152.9, 134.1, 131.4, 129.2, 128.6, 128.6. Anal calcd for $\text{C}_9\text{H}_8\text{O}$, C, 81.79; H, 6.10. Found C, 81.83; H, 6.03.

***p*-Phenylcinnamaldehyde 6k:** mp 121-2 $^{\circ}\text{C}$; lit. mp 110-112 $^{\circ}\text{C}$; ^5IR (KBr) 1673, 981 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.76 (d, J = 7.7 Hz, 1H), 7.69-7.43 (m, 10H), 6.79 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.8, 152.4, 144.1, 139.9, 133.0, 129.1, 129.0, 128.5, 128.2, 127.8, 127.1. Anal calcd for $\text{C}_{15}\text{H}_{12}\text{O}$, C, 86.51; H, 5.81. Found C, 86.48; H, 5.80.

3-Naphthalen-2-yl-propenal 6l: mp: 126-7 $^{\circ}\text{C}$; IR (KBr) 1664, 975 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.77 (d, J = 7.7 Hz, 1H), 7.97-7.55 (m, 8H), 6.83 (dd, J_1 = 15.9 Hz, J_2 = 7.7 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.8, 152.8, 134.7, 133.3, 131.6, 130.8, 129.0, 128.9, 128.7, 127.9, 127.1, 123.6. Anal calcd for $\text{C}_{13}\text{H}_{10}\text{O}$, C, 85.69; H, 5.53. Found C, 85.73; H, 5.51.

3-Anthracen-9-yl-propenal 6m: mp: 167-8 $^{\circ}\text{C}$; IR (KBr) 1680, 982 cm^{-1} ; ^1H NMR (CDCl_3) δ 10.03 (d, J = 7.8, 1H), 8.43 (d, J = 16.4 Hz, 1H), 8.47-7.9 (m, 5H), 7.61-7.49 (m, 4H), 6.76 (dd, J_1 = 16.4 Hz, J_2 = 7.8 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.5, 149.9, 137.4, 131.2, 129.4, 129.2, 129.1, 128.2, 126.9, 125.5, 124.7; Anal calcd for $\text{C}_{17}\text{H}_{12}\text{O}$, C, 87.90; H, 5.21. Found C, 87.93; H, 5.24.

***p*-Fluorocinnamaldehyde 6n:** oil; IR (neat) 1683, 1236, 974 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.68 (d, J = 7.6 Hz, 1H), 7.58-7.55 (m, 2H), 7.44 (d, J = 15.9 Hz, 1H), 7.14-7.10 (m, 2H), 6.63 (dd, J_1 = 15.9 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.4, 164.4 (d, J = 253.0 Hz), 151.3, 130.5 (d, J = 8.7 Hz), 130.3 (d, J = 3.5 Hz), 128.3 (d, J = 2.2 Hz), 116.3 (d, J = 22.0 Hz); ^{19}F NMR {H} δ -108.2. Anal calcd for $\text{C}_9\text{H}_7\text{FO}$, C, 71.99; H, 4.70. Found C, 71.96; H, 4.68.

***m*-Fluorocinnamaldehyde 6o:** oil; IR (neat) 1685, 1268, 974 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.72 (d, J = 7.6 Hz, 1H), , 7.44 (d, J = 15.9 Hz, 1H), 7.43-7.34 (m, 2H), 7.27-7.24 (m, 1H), 7.16-7.13 (m, 1H), 6.69 (dd, J_1 = 15.9 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.4, 163.1 (d, J = 247.5 Hz), 151.0 (d, J = 2.6 Hz), 136.3 (d, J = 7.7 Hz), 130.8 (d, J = 8.2 Hz), 129.7, 124.5 (d, J = 2.9 Hz), 118.1 (d, J = 21.4 Hz), 114.8 (d, J = 22.0 Hz); ^{19}F NMR {H} δ -112.4. Anal calcd for $\text{C}_9\text{H}_7\text{FO}$, C, 71.99; H, 4.70. Found C, 71.91; H, 4.73.

***m*-Trifluoromethylcinnamaldehyde 6p:** oil; IR (neat) 1683, 1334, 1168, 1125, 974 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.76 (d, J = 7.5 Hz, 1H), 7.82-7.57 (m, 4H), 7.53 (d, J = 16.0 Hz, 1H), 6.78 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.1, 150.4, 134.8, 131.7 (q, J = 32.8 Hz), 131.2, 130.0, 129.7, 127.5 (q, J = 3.7 Hz), 125.2 (q, J = 3.8 Hz), 123.7 (q, J = 272.4 Hz); ^{19}F NMR {H} δ -63.4. Anal calcd for $\text{C}_{11}\text{H}_9\text{F}_3\text{O}$, C, 61.68; H, 4.24. Found C, 61.60; H, 4.27.

***o*-Fluorocinnamaldehyde 6q:** oil; IR (neat) 1682, 1228, 975 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.72 (d, J = 7.6 Hz, 1H), 7.68-7.64 (d, J = 16.2 Hz, 1H), 7.60-7.58 (m, 1H), 7.43-7.41 (m, 1H), 7.24-7.12 (m, 2H), 6.79 (dd, J_1 = 16.2 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.9, 161.2 (d, J = 254.8 Hz), 144.8 (d, J = 3.6 Hz), 132.9 (d, J = 8.9 Hz), 130.5 (d, J = 5.3 Hz), 128.8 (d, J = 2.4 Hz), 124.7 (d, J = 3.7 Hz), 122.1 (d, J = 11.4 Hz), 116.3 (d, J = 21.8 Hz); ^{19}F NMR {H} δ -114.7. Anal calcd for $\text{C}_9\text{H}_7\text{FO}$, C, 71.99; H, 4.70. Found C, 72.01; H, 4.65.

***p*-Cyanocinnamaldehyde 6r:** mp 128-129 $^\circ\text{C}$; lit. mp 136-138 $^\circ\text{C}$;⁶ IR (KBr) 2223, 1681, 1447, 980 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.76 (d, J = 7.5 Hz, 1H), 7.75-7.66 (m, 4H), 7.49 (d, J = 16.1 Hz, 1H), 6.77 (dd, J_1 = 16.1 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.1, 149.6, 138.2, 132.9, 131.2, 128.9, 118.2 114.3; Anal calcd for $\text{C}_{10}\text{H}_7\text{NO}$, C, 76.42; H, 4.49; N, 8.91. Found C, 76.39; H, 4.44; N, 8.87.

***p*-Ethoxycarbonylcinnamaldehyde 6s:** mp 71-72 $^\circ\text{C}$; IR (KBr) 1712, 1672, 986 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.72 (d, J_1 = 7.6 Hz, 1H), 8.09-8.04 (m, 2H), 7.62-7.59 (m, 2H), 7.49 (d, J_1 = 16.0 Hz, 1H), 6.75 (dd, J_1 = 16.0 Hz, J_2 = 7.6 Hz 1H), 4.37 (q, J_1 = 7.1 Hz, 2H); 1.39 (t, J_1 = 7.1 Hz, 3H); ^{13}C NMR (CDCl_3) δ 193.3, 165.7, 150.9, 138.0, 132.6, 130.3, 130.2, 128.3, 61.3, 14.3; Anal calcd for $\text{C}_{12}\text{H}_{12}\text{O}_3$, C, 70.57,21; H, 5.92. Found C, 70.48; H, 5.94.

***p*-Acetylcinnamaldehyde 6t:** mp 49-50 $^\circ\text{C}$; IR (KBr) 1677, 1667, 977 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.73 (d, J = 7.6 Hz, 1H), 8.01-7.98 (m, 2H), 7.66-7.63 (m, 2H), 7.50 (d, J = 16.0 Hz, 1H), 6.76 (dd, J_1 = 16.0 Hz, J_2 = 7.6 Hz, 1H), 2.62 (s, 3H); ^{13}C NMR (CDCl_3) δ 197.2, 193.3, 150.7, 138.7, 138.2, 130.4, 129.0, 128.6, 26.7; Anal calcd for $\text{C}_{11}\text{H}_{10}\text{O}_2$, C, 75.84; H, 5.79. Found C, 75.85; H, 5.83.

***p*-Chlorocinnamaldehyde 6u:** mp 59-60 $^\circ\text{C}$; lit. mp 61-63 $^\circ\text{C}$;⁷ IR (KBr) 1700, 1089, 977 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.71 (d, J = 7.6 Hz, 2H), 7.54-7.39 (m, 5H), 6.69 (dd, J_1 = 16.0 Hz, J_2 = 7.6 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.4, 151.0, 137.2, 132.5, 129.6, 129.4, 128.9. Anal calcd for $\text{C}_9\text{H}_7\text{ClO}$, C, 64.88; H, 4.23. Found C, 64.81; H, 4.18.

***p*-Formylcinnamaldehyde 6v:** mp 97-8 $^\circ\text{C}$; lit. mp 93-95;⁸ IR (KBr) 1690, 1679, 981 cm^{-1} ; ^1H NMR (CDCl_3) δ 10.10 (s, 1H), 9.80 (d, J = 7.5 Hz, 1H), 8.02-7.97 (m, 2H), 7.81-7.76 (m, 2H), 7.59 (d, J = 16.1, 1H), 6.85 (dd, J_1 = 16.1 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.3, 191.4, 150.5, 139.6, 137.8, 131.0, 130.3, 129.0. Anal calcd for $\text{C}_{10}\text{H}_8\text{O}_2$, C, 74.99; H, 5.03. Found C, 74.94; H, 5.08.

***m*-Formylcinnamaldehyde 6w:** mp 97-8 $^\circ\text{C}$; IR (KBr) 1694, 1674, 979 cm^{-1} ; ^1H NMR (CDCl_3) δ 10.08 (s, 1H), 9.76 (d, J = 7.5 Hz, 1H), 8.08-7.83 (m, 3H), 7.66 (d, J = 7.6, 1H), 7.56 (d, J = 15.9,

1H), 6.80 (dd, $J_1 = 15.9$ Hz, $J_2 = 7.5$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.4, 191.5, 150.7, 137.1, 135.1, 133.7, 132.1, 130.0, 129.3. Anal calcd for $\text{C}_{10}\text{H}_8\text{O}_2$, C, 74.99; H, 5.03. Found C, 74.92; H, 5.09.

***o*-Formylcinnamaldehyde 6x:** mp 61-2 °C; lit. mp: 57-58;⁹ IR (KBr) 1697, 1682, 981, cm^{-1} ; ^1H NMR (CDCl_3) δ 10.24 (s, 1H), 9.81 (d, $J = 7.7$ Hz, 1H), 8.58 (d, $J = 16.0$ Hz, 1H), 7.94-7.89 (m, 1H), 7.75-7.66 (m, 3H), 6.68 (dd, $J_1 = 16.0$ Hz, $J_2 = 7.7$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.9, 192.9, 149.4, 135.4, 134.7, 134.1, 133.7, 132.3, 130.8, 128.1. Anal calcd for $\text{C}_{10}\text{H}_8\text{O}_2$, C, 74.99; H, 5.03. Found C, 74.96; H, 5.10.

***p*-Nitrocinnamaldehyde 6y:** mp 139-140 °C; lit. mp: 139-140 °C;¹⁰ IR (KBr) 1686, 1520, 1347, 980 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.76 (d, $J = 7.5$, 1H), 8.29-8.24 (m, 2H), 7.76-7.71 (m, 2H), 7.57 (d, $J = 16.1$ Hz, 1H), 6.80 (dd, $J_1 = 16.1$ Hz, $J_2 = 7.5$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 193.1, 149.1, 149.0, 140.1, 131.7, 129.2, 124.3;. Anal calcd for $\text{C}_9\text{H}_7\text{NO}_3$, C, 61.02; H, 3.98; N, 7.91. Found C, 60.97; H, 3.91; N, 7.98.

***m*-Nitrocinnamaldehyde 6z:** mp 110-1 °C; lit. mp: 112-114 °C;¹¹ IR (KBr) 1680, 1522, 1354, 981 cm^{-1} ; ^1H NMR (CDCl_3) δ 9.79 (d, $J = 7.4$ Hz, 1H), 8.43-8.42 (m, 1H), 8.34-8.28 (m, 1H), 7.95-7.91 (m, 1H), 7.71-7.63 (m, 1H), 7.58 (d, $J = 16.1$ Hz, 1H), 6.84 (dd, $J_1 = 16.1$ Hz, $J_2 = 7.4$ Hz, 1H); ^{13}C NMR (CDCl_3) δ 192.9, 149.2, 148.8, 133.7, 130.9, 130.3, 125.4, 123.1; Anal calcd for $\text{C}_9\text{H}_7\text{NO}_3$, C, 61.02; H, 3.98; N, 7.91. Found C, 60.99; H, 3.92; N, 7.99.

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