Supporting Information For

**A KO^tBu–Promoted Three-Component Coupling of Aldehydes and Alkynes: A
Highly Efficient Synthesis of 1-En-4-yn-3-ols and 2-En-4-yn-1-ols**

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Context

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Experimental

General information:

The materials were used as purchased, and the solvent THF was dried and freshly distilled over sodium/benzophenone before use. Aldehydes were freshly distilled. All reactions under standard conditions were monitored by thin-layer chromatography (TLC) on gel F254 plates. The silica gel (200-300 meshes) is used for column chromatography, and the distillation range of petroleum is 60-90°C. ^1H and ^{13}C NMR spectra were recorded on the 300 MHz or 400 MHz instruments, and spectral data are reported in *ppm* relative to tetramethylsilane (TMS) as internal standard. MS were measured on spectrometer by direct inlet at 70 eV, and signals were given in *m/z* with relative intensity (%) in brackets. HRMS data were measured with ESI techniques.

Table 1 Optimization Studies^a

entry	base	base : 4-OMeC ₆ H ₄ CHO ^b	solvent	time (min)	yield (%) ^c
1	KOH	1.5 : 1	THF	60	0
2	CsOH	1.5 : 1	THF	60	0
3	Bu ₄ NF	1.5 : 1	THF	60	0
4	BnN(CH ₃) ₃ OH	1.5 : 1	THF	60	0
5	NaOMe	1.5 : 1	THF	60	0
6	Ph ₃ P	1.5 : 1	THF	60	0
7	KOtBu	1.5 : 1	THF	2	80
8	KOtBu	1.5 : 1	DMF	10	0
9	KOtBu	1.5 : 1	DMSO	10	0
10	KOtBu	1.5 : 1	CH ₂ Cl ₂	10	26
11	KOtBu	1.5 : 1	Et ₂ O	5	60
12	KOtBu	1.5 : 1	Toluene	10	25
13	KOtBu	1.5 : 1	Benzene	10	20
14	KOtBu	0.05 : 1	THF	20	<10
15	KOtBu	1.0 : 1	THF	2	65
16	KOtBu	1.8 : 1	THF	2	90

^a For detailed experimental operation see the **Experimental Section**. ^b Mol ratio. ^c Isolated yield of expected product.

Spectroscopic data for products

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.81-7.83 (m, 2H), 7.47-7.60 (m, 2H), 7.25-7.47 (m, 11H), 7.07 (d, J = 16 Hz, 1H), 6.52 (d, J = 16 Hz, 1H), 2.90 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 143.3, 136.1, 132.8, 131.7, 129.1, 128.6, 128.5, 128.4, 128.3, 127.93, 127.90, 126.9, 125.8, 122.3, 89.8, 87.5, 73.2; MS *m/z* (%) 310 (M^+ , 3), 129 (33), 105 (100), 77 (54); HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{18}\text{ONa}$ ($[\text{M}+\text{Na}]^+$): 333.1250, Found: 333.1252.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.70 (d, J = 8.4 Hz, 2H), 7.25-7.60 (m, 12H), 7.06 (d, J = 16 Hz, 1H), 6.52 (d, J = 16 Hz, 1H), 2.85 (brs, 1H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 140.5, 137.7, 136.2, 132.9, 131.7, 129.1, 128.9, 128.6, 128.5, 128.3, 127.8, 126.9, 125.7, 122.4, 90.0, 87.3, 73.1, 21.1; MS *m/z* (%) 324 (M^+ , 8), 195 (34), 129 (37), 119 (80), 91 (100), 77 (68); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{20}\text{ONa}$ ($[\text{M}+\text{Na}]^+$): 347.1406, Found: 347.1407.

^1H NMR (400 MHz, ACETONE, *ppm*): δ 7.61 (d, J = 8.8 Hz, 2H), 7.22-7.56 (m, 10H), 7.00 (d, J = 16 Hz, 1H), 6.75 (d, J = 8.4 Hz, 2H), 6.53 (d, J = 16 Hz, 1H), 5.28 (brs, 1H), 2.92 (s, 6H); ^{13}C NMR (75 MHz, ACETONE, *ppm*): δ 150.5, 137.1, 135.1, 132.4, 132.1, 131.7, 128.83, 128.77, 127.8, 127.4, 126.9, 123.4, 112.3, 92.2, 85.9, 72.3, 40.1; MS *m/z* (%) 353 (M^+ , 3), 148 (18), 134

(100); HRMS (ESI) calcd for $C_{25}H_{23}NONa$ ($[M+Na]^+$): 376.1672, Found: 376.1669.

1H NMR (400 MHz, $CDCl_3$, *ppm*): δ 7.75 (dd, $J = 1.6$ Hz, $J = 8$ Hz, 1H), 7.28-7.58 (m, 11H), 7.00-7.07 (m, 2H), 6.98 (d, $J = 16$ Hz, 1H), 6.69 (d, $J = 16$ Hz, 1H), 5.02 (brs, 1H), 3.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, *ppm*): δ 156.8, 136.4, 131.7, 131.0, 129.7, 129.4, 128.5, 128.4, 128.2, 127.7, 126.8, 122.7, 121.1, 112.0, 89.7, 86.8, 73.3, 55.8; MS *m/z* (%) 340 (M^+ , 1), 135 (100), 102 (55), 77 (59); HRMS (ESI) calcd for $C_{24}H_{22}O_2Na$ ($[M+Na]^+$): 363.1365, Found: 363.1366.

1H NMR (400 MHz, $CDCl_3$, *ppm*): δ 7.56-7.58 (m, 2H), 7.28-7.46 (m, 11H), 7.06 (d, $J = 16$ Hz, 1H), 6.89-6.92 (m, 1H), 6.50 (d, $J = 16$ Hz, 1H), 3.85 (s, 3H), 3.00 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, *ppm*): δ 159.6, 145.0, 136.1, 132.7, 131.7, 129.4, 129.1, 128.6, 128.5, 128.3, 127.9, 126.9, 122.3, 118.2, 113.4, 111.5, 89.8, 87.4, 73.1, 55.2; MS *m/z* (%) 340 (M^+ , 3), 135 (100), 129 (44), 77 (59); HRMS (ESI) calcd for $C_{24}H_{20}O_2Na$ ($[M+Na]^+$): 363.1356, Found: 363.1351.

1H NMR (300 MHz, $CDCl_3$, *ppm*): δ 7.67-7.70 (m, 2H), 7.02-7.56 (m, 10H), 6.92-6.97 (m, 3H), 6.48 (d, $J = 16$ Hz, 1H), 3.83 (s, 3H), 2.78 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, *ppm*): δ 159.2,

136.2, 135.6, 133.0, 131.7, 128.8, 128.6, 128.5, 128.3, 127.8, 127.2, 126.8, 122.4, 113.7, 90.1, 87.3, 72.8, 55.2; MS m/z (%) 340 (M^+ , 4), 323 (3), 135 (67), 121 (81), 105 (85), 77 (100); HRMS (ESI) calcd for $C_{24}H_{20}O_2Na$ ($[M+Na]^+$): 363.1356, Found: 363.1361.

1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.84-7.88 (m, 1H), 7.09-7.58 (m, 13H), 7.07 (d, J = 16 Hz, 1H), 6.65 (d, J = 16 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 161.4, 158.9, 136.0, 131.8, 130.7, 130.3, 130.2, 129.9, 129.8, 128.7, 128.5, 128.2, 128.0, 127.3, 126.9, 124.0, 122.2, 116.5, 116.2, 88.7, 87.1, 71.0; MS m/z (%) 328 (M^+ , 2), 205 (97), 129 (100), 95 (96), 77 (91); HRMS (ESI) calcd for $C_{23}H_{17}FONa$ ($[M+Na]^+$): 351.1156, Found: 351.1160.

1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.29-7.58 (m, 13H), 7.04-7.08 (m, 2H), 6.46 (d, J = 16 Hz, 1H), 3.07 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 161.5, 164.0, 146.0, 135.8, 132.3, 132.0, 131.8, 129.9, 129.9, 129.5, 128.8, 128.7, 128.6, 128.4, 128.2, 128.1, 126.9, 122.0, 121.5, 114.9, 114.7, 113.2, 112.9, 89.3, 87.7, 72.8; MS m/z (%) 328 (M^+ , 2), 205 (58), 129 (100), 95 (63); HRMS (ESI) calcd for $C_{23}H_{17}FONa$ ($[M+Na]^+$): 351.1156, Found: 351.1158.

1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.74-7.78 (m, 2H), 7.55-7.57 (m, 2H), 7.28-7.45 (m, 8H), 7.10 (t, J = 8 Hz, 2H), 7.03 (d, J = 16 Hz, 1H), 6.47 (d, J = 16 Hz, 1H), 3.14 (brs, 1H); ^{13}C NMR

(100 MHz, CDCl₃, *ppm*): δ 163.6, 161.1, 139.2, 135.9, 132.7, 132.0, 131.7, 129.2, 128.7, 128.5, 128.3, 128.2, 128.0, 127.7, 127.6, 126.9, 122.1, 115.2, 115.0, 89.6, 87.6, 72.7; MS *m/z* (%) 328 (M⁺, 3), 129 (53), 123 (100), 77 (35); HRMS (ESI) calcd for C₂₃H₁₇FONa ([M+Na]⁺): 351.1156, Found: 351.1150.

¹H NMR (300 MHz, CDCl₃, *ppm*): δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.66 (d, *J* = 8.1 Hz, 2H), 7.53-7.56 (m, 2H), 7.26-7.45 (m, 8H), 7.06 (d, *J* = 16 Hz, 1H), 6.433. (d, *J* = 16 Hz, 1H), 2.85 (brs, 1H); ¹³C NMR (75 MHz, CDCl₃, *ppm*): δ 147.2, 135.7, 132.1, 131.8, 130.0, 129.0, 128.6, 128.4, 128.3, 127.0, 126.2, 125.5, 125.4, 125.4, 125.4, 121.9, 89.1, 88.1, 73.0; MS *m/z* (%) 378 (M⁺, 12), 309 (4), 205 (83), 173 (100).

¹H NMR (400 MHz, CDCl₃, *ppm*): δ 8.81-8.84 (m, 1H), 8.16 (d, *J* = 6.8 Hz, 1H), 7.90-7.94 (m, 2H), 7.29-7.57 (m, 12H), 7.17 (d, *J* = 16 Hz, 1H), 6.78 (d, *J* = 16 Hz, 1H), 3.06 (brs, 1H); ¹³C NMR (100 MHz, CDCl₃, *ppm*): δ 137.9, 136.1, 134.6, 132.7, 131.7, 131.0, 130.2, 129.5, 128.7, 128.5, 128.2, 128.0, 126.9, 126.6, 125.5, 125.4, 124.9, 124.0, 122.4, 90.5, 87.9, 73.0; MS *m/z* (%) 360 (M⁺, 3), 155 (100), 129 (65), 77 (69); HRMS (ESI) calcd for C₂₇H₂₀ONa ([M+Na]⁺): 383.1406, Found: 383.1411.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 8.55-8.57 (m, 1H), 7.24-7.77 (m, 13H), 7.15 (d, $J = 16$ Hz, 1H), 6.37 (d, $J = 16$ Hz, 1H), 6.14 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 160.0, 147.4, 137.6, 136.2, 132.2, 131.9, 130.4, 128.6, 128.5, 128.2, 128.0, 127.0, 123.1, 122.5, 121.4, 89.7, 86.5, 72.1; MS *m/z* (%) 311 (M^+ , 9), 294 (10), 180 (20), 79 (100); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{17}\text{NONa}$ ($[\text{M}+\text{Na}]^+$): 334.1202, Found: 334.1204.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 8.96 (s, 1H), 8.44-8.46 (m, 1H), 8.04-8.07 (m, 1H), 7.22-7.46 (m, 11H), 7.00 (d, $J = 16$ Hz, 1H), 6.39 (d, $J = 16$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 148.3, 147.3, 139.9, 135.9, 134.0, 132.5, 131.7, 129.6, 128.85, 128.5, 128.3, 128.0, 126.9, 123.2, 122.0, 89.3, 87.8, 71.3; MS *m/z* (%) 311 (M^+ , 12), 234 (14), 205 (81), 129 (88), 106 (100), 77 (88); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{17}\text{NONa}$ ($[\text{M}+\text{H}]^+$): 312.1383, Found: 312.1387.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 8.55 (d, $J = 4$ Hz, 2H), 7.64-7.65 (m, 2H), 7.25-7.47 (m, 10H), 7.03 (d, $J = 16$ Hz, 1H), 6.36 (d, $J = 16$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 153.1, 149.5, 135.8, 132.0, 131.7, 130.1, 128.9, 128.6, 128.4, 128.2, 126.9, 122.0, 120.9, 89.1, 87.8, 72.2;

MS m/z (%) 311 (M^+ , 12), 293 (4), 234 (13), 205 (100), 129 (92), 77 (68); HRMS (ESI) calcd for $C_{24}H_{17}NONa$ ($[M+H]^+$): 312.1383, Found: 312.1388.

1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.28-7.56 (m, 11H), 7.06 (d, J = 16 Hz, 1H), 6.62 (d, J = 16 Hz, 1H), 6.54-6.55 (m, 1H), 6.38-6.39 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 154.6, 143.0, 136.0, 131.8, 131.0, 129.2, 128.8, 128.6, 128.3, 128.1, 127.0, 122.1, 110.3, 107.1, 87.2, 86.6, 68.6; MS m/z (%) 300 (M^+ , 16), 129 (100), 95 (65), 77 (61); HRMS (ESI) calcd for $C_{21}H_{16}O_2Na$ ($[M+Na]^+$): 323.1043, Found: 323.1048.

1H NMR (300 MHz, $CDCl_3$, ppm): δ 7.34-7.69 (m, 11H), 6.94 (d, J = 9 Hz, 2H), 6.38 (d, J = 15 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 159.4, 136.8, 135.5, 134.8, 134.2, 131.8, 131.7, 131.4, 128.4, 127.9, 127.7, 127.6, 127.5, 127.1, 125.8, 125.8, 125.7, 125.7, 125.6, 125.6, 125.2, 124.8, 122.8, 122.1, 120.5, 113.8, 95.1, 83.5, 72.9, 55.3; MS m/z (%) 476 (M^+ , 1), 407 (<1), 135 (33), 121 (100); HRMS (ESI) calcd for $C_{26}H_{18}O_2F_6Na$ ($[M+Na]^+$): 499.1103, Found: 499.1098.

^1H NMR (300 MHz, CDCl_3 , *ppm*): δ 7.68 (d, $J = 9$ Hz, 2H), 7.47 (d, $J = 9$ Hz, 2H), 7.36 (d, $J = 9$ Hz, 2H), 6.84-6.95 (m, 7H), 6.35 (d, $J = 16$ Hz, 1H), 3.82 (s, 6H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , *ppm*): δ 159.8, 159.4, 159.2, 136.0, 133.2, 131.2, 129.0, 128.3, 128.1, 127.2, 114.6, 113.9, 113.6, 89.0, 87.1, 72.9, 55.2; MS *m/z* (%) 400 (M^+ , 3), 293 (2), 135 (100), 121 (37), 77 (22); HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{24}\text{O}_4\text{Na}$ ($[\text{M}+\text{Na}]^+$): 423.1567, Found: 423.1565.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.64-7.67 (m, 2H), 7.48-7.52 (m, 2H), 7.36-7.39 (m, 2H), 6.89-7.06 (m, 7H), 6.37 (d, $J = 16$ Hz, 1H), 3.82 (s, 3H), 2.83 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 163.9, 163.7, 161.4, 161.2, 159.4, 135.4, 133.7, 133.6, 132.7, 132.3, 128.4, 128.37, 127.7, 127.1, 118.4, 115.7, 115.6, 115.4, 113.8, 113.4, 89.7, 86.3, 72.8, 55.3; MS *m/z* (%) 376 (M^+ , 1), 239 (5), 229 (8), 147 (28), 135 (50), 121 (100); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{18}\text{F}_2\text{O}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 399.1167, Found: 399.1172.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.25-7.51 (m, 10H), 6.94 (d, $J = 16$ Hz, 1H), 6.28 (d, $J = 16$ Hz, 1H), 6.21-6.22 (m, 1H), 2.13-2.22 (m, 4H), 1.58-1.70 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 ,

ppm): δ 138.7, 136.4, 131.7, 129.8, 128.5, 128.4, 128.3, 127.3, 126.8, 123.4, 122.6, 89.7, 86.8, 74.2, 25.2, 24.1, 22.8, 22.2; MS *m/z* (%) 314 (M^+ , 6), 296 (36), 129 (100), 91 (82), 81 (53); HRMS (ESI) calcd for $C_{23}H_{22}ONa$ ($[M+Na]^+$): 337.1563, Found: 337.1568.

1H NMR (400 MHz, $CDCl_3$, *ppm*): δ 7.59-7.62 (m, 2H), 7.24-7.41 (m, 5H), 6.89-6.93 (m, 3H), 6.38 (d, $J=15.6$ Hz, 1H), 3.82 (s, 3H), 2.52 (brs, 1H), 2.35 (t, $J=7.2$ Hz, 2H), 1.58-1.64 (m, 2H), 134-1.46 (m, 4H), 0.93 (t, $J=7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, *ppm*): δ 159.2, 136.4, 136.2, 133.6, 128.5, 128.4, 127.7, 127.1, 126.8, 113.6, 88.5, 81.3, 72.6, 55.3, 31.1, 28.3, 22.2, 18.9, 14.0; MS *m/z* (%) 334 (M^+ , 1), 135 (38), 121 (100); HRMS (ESI) calcd for 357.1825, Found: 357.1831.

1H NMR (400 MHz, $CDCl_3$, *ppm*): δ 7.67-7.69 (m, 2H), 7.25-7.48 (m, 7H), 6.97 (d, $J=16$ Hz, 1H), 6.86-6.94 (m, 4H), 6.46 (d, $J=16$ Hz, 1H), 3.82 (s, 6H), 2.80 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, *ppm*): δ 159.8, 159.3, 136.3, 135.8, 133.2, 128.7, 128.5, 128.2, 127.8, 127.2, 126.9, 114.5, 113.9, 113.7, 88.8, 87.3, 72.9, 55.3; MS *m/z* (%) 370 (M^+ , 2), 293 (1), 135 (100), 77 (28); HRMS (ESI) calcd for $C_{25}H_{22}O_3Na$ ($[M+Na]^+$): 393.1461, Found: 393.1468.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.66-7.68 (m, 2H), 7.26-7.53 (m, 7H), 6.92-7.07 (m, 5H), 6.47 (d, $J = 16$ Hz, 1H), 3.82 (s, 3H), 2.89 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 163.9, 161.4, 159.3, 136.1, 135.4, 133.7, 133.6, 132.9, 128.8, 128.5, 128.3, 127.9, 127.1, 126.8, 118.4, 115.7, 115.5, 113.7, 89.8, 86.2, 72.8, 55.2; MS m/z (%) 358 (M^+ , 2), 211 (14), 147 (30), 135 (84), 121 (100), 77 (31); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{19}\text{FO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 381.1261, Found: 381.1256.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.67-7.70 (m, 2H), 7.53-7.55 (m, 2H), 7.35-7.40 (m, 5H), 6.92-7.02 (m, 5H), 6.39 (d, $J = 16$ Hz, 1H), 3.82 (s, 3H), 2.87 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 159.3, 135.6, 132.8, 132.4, 131.7, 128.7, 128.5, 128.3, 127.7, 127.2, 122.3, 115.5, 115.3, 113.8, 90, 87.4, 72.8, 55.3; MS m/z (%) 358 (M^+ , 2), 229 (18), 135 (70), 129 (31), 121 (100); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{19}\text{FO}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 381.1261, Found: 381.1258.

^1H NMR (300 MHz, CDCl_3 , *ppm*): δ 7.65-7.68 (m, 2H), 7.46 (d, $J = 9.3$ Hz, 2H), 7.36-7.40 (m, 2H), 6.86-7.03 (m, 7H), 6.37 (d, $J = 16$ Hz, 1H), 3.82 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*):

δ 163.7, 161.2, 159.9, 159.3, 135.8, 133.2, 133.1, 132.5, 130.4, 128.6, 128.4, 128.4, 127.5, 127.2, 115.5, 115.3, 114.4, 114.0, 113.7, 88.7, 87.4, 72.8, 55.4, 55.3; MS m/z (%) 388 (M^+ , 3), 159 (39), 135 (100), 121 (32), 77 (14); HRMS (ESI) calcd for $C_{25}H_{21}FO_3Na$ ($[M+Na]^+$): 411.1367, Found: 411.1365.

1H NMR (300 MHz, $CDCl_3$, ppm): δ 7.66 (d, J = 9 Hz, 2H), 7.48-7.53 (m, 2H), 7.36 (d, J = 9 Hz, 2H), 6.36-7.07 (m, 7H), 6.33 (d, J = 16 Hz, 1H), 3.82 (s, 3H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 163.8, 161.4, 159.4, 159.2, 135.8, 133.7, 133.6, 130.9, 128.9, 128.5, 128.0, 127.1, 118.6, 115.6, 115.4, 114.0, 113.7, 90.1, 86.0, 72.8, 55.2, 55.2; MS m/z (%) 388 (M^+ , 2), 135 (100), 127 (85), 77 (18); HRMS (ESI) calcd for $C_{25}H_{21}FO_3Na$ ($[M+Na]^+$): 411.1367, Found: 411.1374.

1H NMR (300 MHz, $CDCl_3$, ppm): δ 7.70 (d, J = 8.4 Hz, 2H), 7.54-7.57 (m, 2H), 7.36-7.39 (m, 5H), 6.85-6.97 (m, 5H), 6.36 (d, J = 16 Hz, 1H), 3.83 (s, 3H), 3.81 (brs, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 159.4, 159.2, 135.9, 131.7, 131.0, 128.9, 128.5, 128.4, 128.3, 128.1, 127.1, 122.5, 113.9, 113.7, 90.4, 87.2, 72.9, 55.2; MS m/z (%) 370 (M^+ , 2), 293 (<1), 135 (100), 121 (80), 77 (25); HRMS (ESI) calcd for $C_{25}H_{22}O_3Na$ ($[M+Na]^+$): 393.1461, Found: 393.1454.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.58-7.61 (m, 2H), 7.48-7.53 (m, 4H), 7.03-7.10 (m, 4H), 6.89-6.90 (m, 2H), 6.43 (d, J = 9 Hz, 1H), 6.02 (d, J = 9 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 163.9, 163.4, 161.5, 161.0, 159.8, 138.5, 136.2, 133.5, 133.4, 129.4, 127.6, 127.5, 123.7, 118.8, 115.9, 115.7, 115.4, 115.2, 113.8, 95.4, 85.6, 72.4, 55.3; MS *m/z* (%) 376 (M^+ , 14), 347 (7), 281 (9), 239 (100), 123 (92); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{18}\text{F}_2\text{O}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 399.1167, Found: 399.1172.

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.64-7.68 (m, 2H), 7.55-7.58 (m, 4H), 7.31-7.41 (m, 6H), 7.03-7.07 (m, 2H), 6.53 (d, J = 8.4 Hz, 1H), 6.09 (d, J = 8.4 Hz, 1H), 2.57 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 164.0, 161.5, 142.5, 138.0, 133.1, 131.6, 128.8, 128.6, 128.5, 128.04, 127.96, 127.7, 125.9, 123.1, 122.6, 115.4, 115.2, 96.7, 85.7, 73.0; MS *m/z* (%) 328 (M^+ , 7), 251 (12), 209 (73), 105 (100), 77 (52); HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{17}\text{FONa}$ ($[\text{M}+\text{Na}]^+$): 351.1156, Found: 351.1152.

2c

; ^1H NMR (300 MHz, CDCl_3 , *ppm*): δ 7.65(d, J = 9 Hz, 2H), 7.56-7.59 (m, 4H), 7.31-7.42 (m, 6H), 6.91 (d, J = 9 Hz, 2H), 6.51 (d, J = 9 Hz, 1H), 6.10 (d, J = 9 Hz, 1H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 159.8, 142.8, 136.4, 131.6, 129.7, 128.6, 128.4, 127.6, 127.6, 125.9, 123.8, 123.0, 113.8, 96.4, 86.1, 73.1, 55.3; MS *m/z* (%) 340 (M^+ , 6), 263 (6), 105 (73), 77 (100); HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{20}\text{O}_2\text{Na}$ ($[\text{M}+\text{Na}]^+$): 363.1356, Found: 363.1357.

2d

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.65-7.68 (m, 2H), 7.52-7.55 (m, 4H), 7.27-7.37 (m, 9H), 6.56 (d, J = 9 Hz, 1H), 6.07 (d, J = 9 Hz, 1H), 2.44 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3 , *ppm*): δ 142.5, 138.2, 137.0, 131.6, 128.6, 128.6, 128.4, 128.2, 127.6, 126.3, 125.9, 124.1, 122.8, 96.5, 85.9, 72.98; MS *m/z* (%) 310 (M^+ , 8), 233 (13), 205 (94), 191 (100), 77 (91); HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{18}\text{ONa}$ ($[\text{M}+\text{Na}]^+$): 333.1250, Found: 333.1253.

2e

^1H NMR (400 MHz, CDCl_3 , *ppm*): δ 7.60-7.48 (m, 5H), 7.40-7.25 (m, 7H), 6.99-6.91 (m, 2H), 6.65 (d, J = 8.8 Hz, 1H), 6.11 (d, J = 8.8 Hz, 1H), 3.85 (s, 3H), 2.67 (brs, 1H); ^{13}C NMR (100

MHz, CDCl₃, ppm): δ 156.9, 142.6, 142.6, 131.5, 130.2, 129.1, 128.5, 128.3, 127.5, 127.0, 126.0, 123.2, 121.1, 120.5, 111.5, 94.7, 87.0, 72.9, 55.6; MS m/z (%) 340 (M^+ , 4), 325 (3), 263 (3), 105 (80), 77 (100); HRMS (ESI) calcd for C₂₄H₂₀O₂Na ($[M+Na]^+$): 363.1356, Found: 36.1362.

¹H NMR (400 MHz, CDCl₃, ppm): δ 7.49-7.57 (m, 4H), 7.28-7.38 (m, 3H), 6.84-6.86 (m, 2H), 6.35 (d, J =8.4 Hz, 1H), 5.96 (d, J =8.4 Hz, 1H), 3.80 (s, 3H), 2.49 (t, J =7.2 Hz, 2H), 2.20 (brs, 1H), 1.34-1.67 (m, 6H), 0.92 (t, J =7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 159.6, 143.0, 135.1, 130.2, 128.5, 127.5, 125.8, 124.4, 113.6, 97.9, 72.9, 55.3, 31.2, 28.4, 22.2, 19.6, 14.0; MS m/z (%) 334 (M^+ , 1), 105 (56), 57 (61), 43 (100); HRMS (ESI) calcd for 357.1825, Found: 357.1832.

¹H NMR (400 MHz, CDCl₃, ppm): δ 7.80-7.83 (m, 2H), 7.47-7.60 (m, 2H), 7.28-7.47 (m, 11H), 6.51 (s, 1H), 2.88 (brs, 1H); MS m/z (%) 311 (M^+ , 4), 234 (5), 129 (48), 105 (100); HRMS (ESI) calcd for C₂₃H₁₇DONa ($[M+Na]^+$): 334.1313, Found: 334.1314.

¹H NMR (400 MHz, CDCl₃, ppm): δ 7.66-7.69 (m, 2H), 7.52-7.55 (m, 2H), 7.26-7.44 (m, 8H),

6.91-7.01 (m, 3H), 6.46 (d, $J = 16$ Hz, 0.45H), 3.82 (s, 3H), 2.62 (brs, 1H); MS m/z (%) 341 (M^+ , 2), 135 (100), 129 (51); HRMS (ESI) calcd for $C_{24}H_{19}DO_2Na$ ($[M+Na]^+$): 364.1418, Found: 364.1412.

3.070

3

13006-c-2

3.818

2.866

1w

057703-C-3