

## Supporting Information

### Efficient direct ester condensation between equimolar amounts of carboxylic acids and alcohols catalyzed by trifluoromethanesulfonic acid (TfOH) in Solkane365mfc

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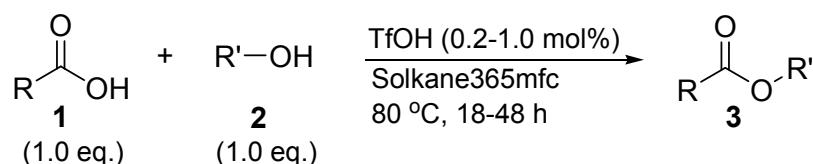
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### 1. General Methods:

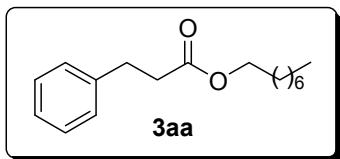
All reagents were used as received from commercial sources, unless specified otherwise, or prepared as described in the literature. Reaction mixtures were stirred magnetically. All of the reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Merck silica-gel (60-F254). The TLC plates were visualized with UV light and 7% phosphomolybdic acid or KMnO<sub>4</sub> in water/heat. Column chromatography was carried out on a column packed with silica-gel 60N spherical neutral size 63-210 µm. The <sup>1</sup>H-NMR (300 MHz) and <sup>19</sup>F-NMR (282.3 MHz) spectra was recorded on a Varian Mercury 300. The <sup>13</sup>C-NMR (150.9 MHz) was recorded on a Bruker Avance 600. Chemical shifts ( $\delta$ ) are reported in parts per million and coupling constants ( $J$ ) are in hertz. All the melting points are uncorrected. Mass spectra were recorded on a SHIMADZU GCMS-QP5050A. Infrared spectra were recorded on a JASCO FT/ IR-200 spectrometer.

### 2. General procedure for esterification of carboxylic acid 1 and alcohol 2 in Solkane365mfc

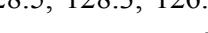


A sealed tube was charged with carboxylic acid **1** (5.0 mmol), Solkane365mc (5.0 mL), alcohol **2** (5.0 mmol) and TfOH (0.2 mol%) were added. The reaction mixture was stirred at 80 °C for 18h, then was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give compound **3**.

### **Octyl 3-Phenylpropanoate (3aa)**

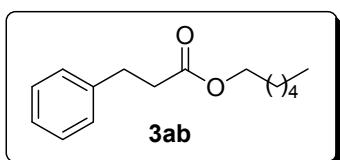


**3aa**



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.27 (m, 10H), 1.57-1.61 (m, 2H), 2.62 (t,  $J = 7.8$  Hz, 2H), 2.95 (t,  $J = 7.9$  Hz, 2H), 4.06 (t,  $J = 6.7$  Hz, 2H), 7.19-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  172.9, 140.6, 128.5, 128.3, 126.2, 64.6, 35.9, 31.8, 31.0, 29.3, 29.2, 28.7, 25.9, 22.7, 14.1; MS (ESI,  $m/z$ ) 285.2 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[1]</sup>

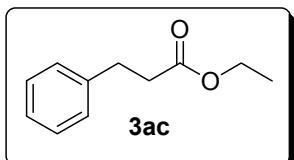
## Hexyl 3-Phenylpropanoate (3ab)



**3ab**

colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.87-0.91 (m, 3H), 1.29-1.35 (m, 6H), 1.55-1.61 (m, 2H), 2.63 (t,  $J = 7.6$  Hz, 2H), 2.95 (t,  $J = 7.8$  Hz, 2H), 4.06 (t,  $J = 6.6$  Hz, 2H), 7.20-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  173.0, 140.6, 128.5, 128.3, 126.3, 64.7, 36.0, 31.5, 31.1, 28.6, 25.6, 22.6, 14.1; MS (ESI, *m/z*) 257.1 [ $\text{M}+\text{Na}$ ] $^+$ . These assignments matched with those previously published.<sup>[2]</sup>

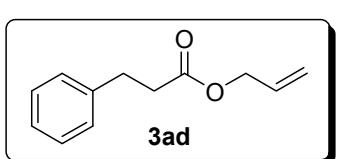
### Ethyl 3-Phenylpropanoate (3ac)



**3ac**

colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  1.23(t,  $J = 7.2$  Hz, 3H), 2.62 (t,  $J = 7.8$  Hz, 2H), 2.95 (t,  $J = 7.8$  Hz, 2H), 4.13 (q,  $J = 6.6$  Hz, 2H), 7.19-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  172.7, 140.5, 128.4, 128.2, 126.2, 60.3, 35.9, 30.9, 14.1; MS (ESI,  $m/z$ ) 201.0 [ $\text{M}+\text{Na}^+$ ]. These assignments matched with those previously published.<sup>[3]</sup>

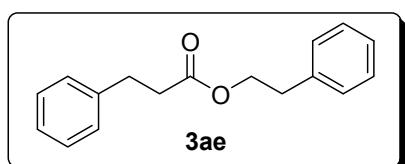
### Allyl 3-Phenylpropanoate (3ad)



**3ad**

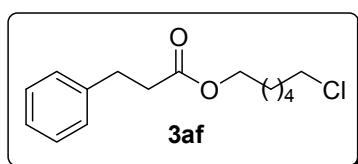
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  2.66 (t,  $J$  = 7.8 Hz, 2H), 2.97 (t,  $J$  = 7.7 Hz, 2H), 4.58 (dd,  $J$  = 4.8, 1.1 Hz, 2H), 5.21-5.31 (m, 2H), 5.90 (ddt,  $J$  = 17.4, 12.9, 5.7 Hz, 1H), 7.19-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150.9 MHz)  $\delta$  172.5, 140.5, 132.2, 128.5, 128.3, 126.3, 118.2, 65.1, 36.8, 30.9; MS (ESI,  $m/z$ ) 213.2 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[4]</sup>

### Phenylethyl 3-Phenylpropanoate (3ae)



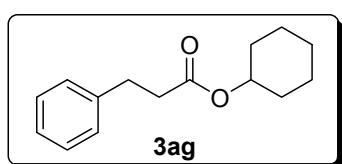
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  2.61 (t,  $J = 7.8$  Hz, 2H), 2.88-2.95 (m, 4H), 4.28 (t,  $J = 6.9$  Hz, 2H), 7.15-7.32 (m, 10H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  172.8, 140.5, 137.9, 129.0, 128.5, 128.3, 126.6, 126.3, 65.0, 35.9, 35.1, 31.0; MS (ESI,  $m/z$ ) 277.1 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[5]</sup>

### 6-Chlorohexyl 3-Phenylpropanoate (3af)



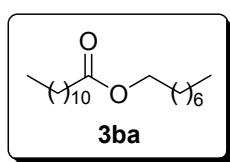
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  1.72-1.50 (m, 4H), 1.56-1.66 (m, 2H), 1.71-1.81 (m, 2H), 2.63 (t,  $J = 7.6$  Hz, 2H), 2.95 (t,  $J = 7.8$  Hz, 2H), 3.53 (t,  $J = 6.6$  Hz, 2H), 4.07 (t,  $J = 6.6$  Hz, 2H), 7.19-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  173.0, 140.6, 128.5, 128.3, 126.3, 64.4, 45.0, 35.9, 32.5, 31.0, 28.5, 26.5, 25.3; IR (neat) 3028, 2938, 2861, 2359, 1734, 1496, 1455, 1259, 1162, 1078, 750, 699, 646 cm $^{-1}$ ; MS (ESI,  $m/z$ ) 291.1 [M+Na] $^+$ .

### Cyclohexyl 3-Phenylpropanoate (3ag)



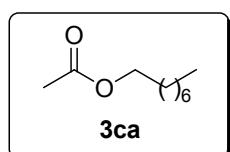
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  1.21-1.32 (m, 5H), 1.35-1.84 (m, 5H), 2.61 (t,  $J = 7.5$  Hz, 2H), 2.95 (t,  $J = 7.8$  Hz, 2H), 4.71-4.78 (m, 1H), 7.17-7.31 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  172.5, 140.7, 128.5, 128.4, 126.3, 72.7, 36.3, 31.7, 31.2, 25.5, 23.8; MS (ESI,  $m/z$ ) 255.1 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[6]</sup>

### Octyl Dodecanoate(3ba)



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.85-0.90 (m, 6H), 1.26-1.27 (m, 26H), 1.56-1.64 (m, 4H), 2.29 (t,  $J = 7.3$  Hz, 2H), 4.06 (t,  $J = 6.7$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  174.0, 64.5, 34.5, 32.0, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 29.3, 29.2, 28.8, 26.1, 25.2, 22.8, 22.7, 14.2, 14.1; MS (ESI,  $m/z$ ) 335.3 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[7]</sup>

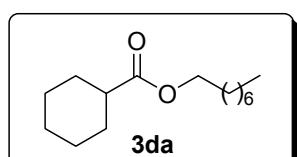
### Octyl Acetate(3ca)



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.27-1.31 (m, 10H), 1.55-1.64 (m, 2H), 2.05 (s, 3H), 4.05 (t,  $J = 6.7$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  170.9, 64.5, 31.7, 29.2, 29.1, 28.6, 25.9, 22.6, 20.8, 14.0; MS (ESI,  $m/z$ ) 195.0 [M+Na] $^+$ .

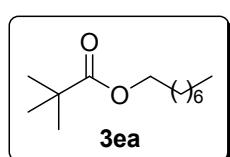
These assignments matched with those previously published.<sup>[8]</sup>

### Octyl Cyclohexanecarboxylate (3da)



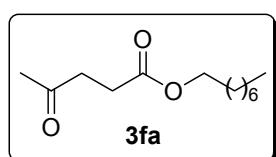
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.88 (m, 3H), 1.27 (m, 13H), 1.47-1.49 (m, 2H), 1.61-1.63 (m, 3H), 1.74-1.76 (m, 2H), 1.88-1.92 (m, 2H), 2.24-2.32 (m, 1H), 4.05 (t,  $J = 6.6$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  176.2, 64.3, 43.3, 31.8, 29.3, 29.2, 29.1, 28.7, 26.0, 25.9, 25.5, 22.7, 14.1; IR (neat) 2930, 2856, 1734, 1453, 1372, 1312, 1247, 1169, 1133, 1037, 894, 723  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 263.2 [M+Na] $^+$ .

### Octyl Pivalate (3ea)



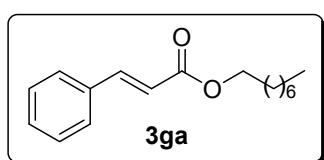
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.20 (s, 9H), 1.27-1.31 (m, 10H), 1.58-1.64 (m, 2H), 4.05 (t,  $J = 6.6$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  178.3, 64.3, 38.6, 31.8, 29.2, 28.6, 27.1, 25.9, 22.6, 14.0; IR (neat) 2929, 2857, 1732, 1458, 1397, 1364, 1284, 1156, 770  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 215.0 [M+H] $^+$ .

### Octyl 4-Oxopentanoate (3fa)



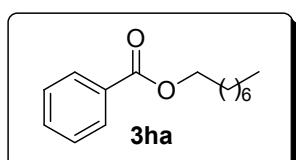
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.88 (m, 3H), 1.28 (m, 10H), 1.62 (m, 2H), 2.20 (s, 3H), 2.58 (t,  $J=6.3$  Hz, 2H), 2.75 (t,  $J = 6.5$  Hz, 2H), 4.06 (t,  $J = 6.6$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  206.5, 172.7, 64.7, 37.8, 31.7, 29.7, 29.1, 28.5, 27.9, 25.8, 22.6, 14.0; IR (neat) 2927, 2856, 1736, 1468, 1357, 1310, 1159, 1028, 952, 723  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 251.1 [M+Na] $^+$ .

### (E)-Octyl Cinnamate (3ga)



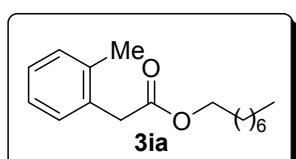
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.29-1.40 (m, 10H), 1.66-1.75 (m, 2H), 4.20 (t,  $J = 6.6$  Hz, 2H), 6.45 (d,  $J = 8.0$  Hz, 1H), 7.37-7.40 (m, 3H), 7.51-7.55 (m, 2H), 7.69 (d,  $J = 8.0$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  166.7, 144.3, 134.3, 130.0, 128.7, 127.9, 118.2, 64.5, 31.7, 29.2, 29.1, 28.6, 25.9, 22.6, 14.0; IR (neat) 3061, 3028, 2926, 2855, 1714, 1637, 1450, 1385, 1310, 1280, 1202, 1168, 1071, 979, 863, 767, 684  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 283.1 [M+Na] $^+$ .

### Octyl Benzoate (3ha)



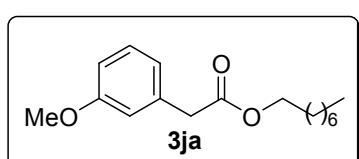
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.28-1.47 (m, 10H), 1.71-1.82 (m, 2H), 4.31 (t,  $J = 6.6$  Hz, 2H), 7.41-7.46 (m, 2H), 7.53-7.58 (m, 1H), 8.03-8.06 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  166.5, 132.7, 130.5, 129.5, 128.2, 65.0, 31.8, 29.3, 29.2, 28.7, 26.0, 22.6, 14.0; MS (ESI,  $m/z$ ) 257.1 [M+Na] $^+$ . These assignments matched with those previously published.<sup>[9]</sup>

### Octyl 2-o-Tolylacetate (3ia)



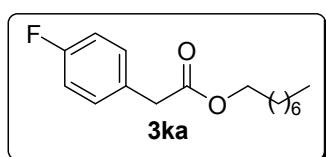
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J = 6.6$  Hz, 3H), 1.30-1.26 (m, 10H), 1.62-1.57 (m, 2H), 2.32 (s, 3H), 3.63 (s, 2H), 4.08 (t,  $J = 6.6$  Hz, 2H), 7.18-7.17 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  171.6, 136.8, 133.0, 130.3, 130.2, 127.3, 126.1, 64.9, 39.3, 31.8, 29.22, 29.20, 28.6, 25.9, 22.7, 19.6, 14.1; IR (neat) 3648, 3020, 2927, 2856, 1735, 1463, 1333, 1252, 1154, 1008, 846, 745, 727  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 285.1 [M+Na] $^+$ .

### Octyl 2-(3-Methoxyphenyl)acetate (3ja)



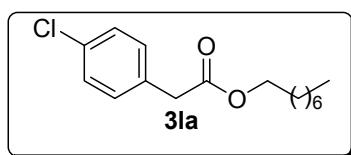
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.90 (m, 3H), 1.26-1.32 (m, 10H), 1.56-1.63 (m, 2H), 3.59 (s, 2H), 3.80 (s, 3H), 4.08 (t,  $J = 6.7$  Hz, 2H), 6.79-6.88 (m, 3H), 7.22 (d,  $J = 7.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  171.6, 159.7, 135.7, 129.5, 121.6, 114.9, 112.6, 65.1, 55.1, 41.5, 31.8, 29.2, 28.6, 25.9, 22.7, 14.1; IR (neat) 2927, 2855, 1735, 1601, 1586, 1491, 1456, 1436, 1263, 1150, 1052, 874, 769, 689  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 301.1 [M+Na] $^+$ .

### Octyl 2-(4-Fluorophenyl)acetate (3ka)



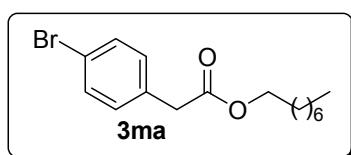
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.86-0.91 (m, 3H), 1.27 (m, 10H), 1.56-1.63 (m, 2H), 3.59 (s, 2H), 4.08 (t,  $J$  = 6.6 Hz, 2H), 6.98-7.04 (m, 2H), 7.23-7.27 (m, 2H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , **282.3 MHz**)  $\delta$  -116.4 (s, 1F);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  171.6, 162.1 (d,  $J$  = 244.5 Hz), 130.9 (d,  $J$  = 9.1 Hz), 130.0 (d,  $J$  = 3.0 Hz), 115.4 (d,  $J$  = 22.6 Hz), 65.2, 40.6, 31.8, 29.3, 29.2, 28.6, 25.9, 22.7, 14.2; IR (neat) 2927, 2856, 1735, 1607, 1510, 1467, 1337, 1224, 1155, 1093, 1016, 824, 797, 724  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 289.1 [M+Na] $^+$ .

### Octyl 2-(4-Chlorophenyl)acetate (3la)



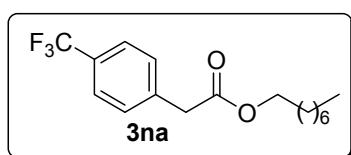
colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J$  = 6.6 Hz, 3H), 1.26 (m, 10H), 1.58-1.63 (m, 2H), 3.58 (s, 2H), 4.08 (t,  $J$  = 6.7 Hz, 2H), 7.21 (d,  $J$  = 4.2 Hz, 2H), 7.29 (d,  $J$  = 4.3 Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  171.3, 133.1, 132.7, 130.7, 128.7, 65.2, 40.8, 31.8, 29.2, 29.2, 28.6, 25.9, 22.7, 14.2; IR (neat) 2927, 2856, 1736, 1492, 1467, 1409, 1335, 1251, 1158, 1091, 1016, 806, 762  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 305.1 [M+Na] $^+$ .

### Octyl 2-(4-Bromophenyl)acetate (3ma)



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J$  = 6.6 Hz, 3H), 1.26 (m, 10H), 1.58-1.63 (m, 2H), 3.56 (s, 2H), 4.08 (t,  $J$  = 6.7 Hz, 2H), 7.16 (d,  $J$  = 4.4 Hz, 2H), 7.44 (d,  $J$  = 4.1 Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  171.2, 133.2, 131.7, 131.1, 121.2, 65.3, 40.9, 31.8, 29.3, 29.2, 28.6, 25.9, 22.7, 14.2; IR (neat) 2926, 2855, 1736, 1488, 1467, 1408, 1336, 1251, 1159, 1071, 1012, 802, 756  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 349.0 [M+Na] $^+$ .

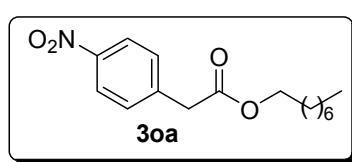
### Octyl 2-(4-Trifluoromethylphenyl)acetate (3na)



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J$  = 6.6 Hz, 3H), 1.27 (m, 10H), 1.57-1.64 (m, 2H), 3.73 (s, 2H), 4.11 (t,  $J$  = 6.7 Hz, 2H), 7.41 (d,  $J$  = 7.8 Hz, 2H), 7.59 (d,  $J$  = 8.4 Hz, 2H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , **282.3 MHz**)  $\delta$  -63.1 (s,

3F);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  170.9, 138.3, 129.8, 129.5 (d,  $J = 31.7$  Hz), 125.5 (q,  $J = 4.5$  Hz), 124.3 (d,  $J = 2471.6$  Hz), 65.4, 41.1, 31.9, 29.3, 29.2, 28.6, 25.9, 22.7, 14.1; IR (**neat**) 2927, 2856, 1736, 1606, 1522, 1467, 1347, 1221, 1163, 1110, 1016, 855, 719  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 339.0 [M+Na]<sup>+</sup>.

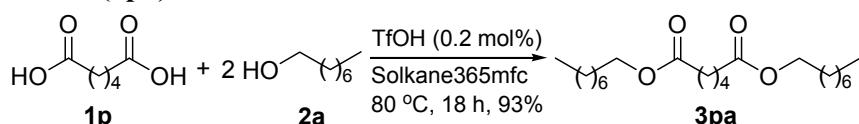
### Octyl 2-(4-Nitrophenyl)acetate (**3oa**)



colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J = 6.6$  Hz, 3H), 1.26 (m, 10H), 1.56-1.63 (m, 2H), 3.68 (s, 2H), 4.10 (t,  $J = 6.7$  Hz, 2H), 7.46 (d,  $J = 7.8$  Hz, 2H), 8.20 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  170.3, 147.2, 141.6, 130.4, 123.7, 65.6, 41.1, 31.8, 29.2, 29.2, 28.5, 25.9, 22.7, 14.1; IR (**neat**) 2929, 2858, 1738, 1621, 1467, 1421, 1326, 1256, 1165, 1126, 1067, 1020, 822, 716, 599  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 316.1 [M+Na]<sup>+</sup>.

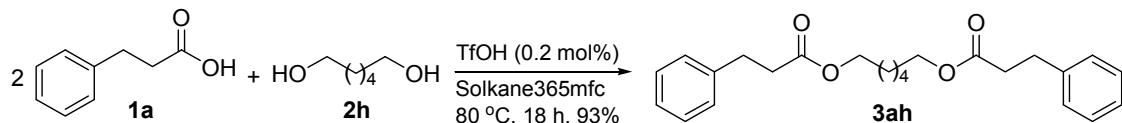
## 3. Preparation of polyaryls **3pa** and **3ah** (Scheme 2)

### Diocetyl Malonate (**3pa**)



A sealed tube was charged with adipic acid **1p** (730.7 mg, 5.00 mmol). Solkane365mc (5.0 mL), octan-1-ol **2a** (1.57 mL, 10.00 mmol) and TfOH (2.2  $\mu\text{L}$ , 0.01 mmol) were added. The reaction mixture was stirred at 80 °C for 18h, then was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give compound **3pa** (1.7298 g, 93% yield). colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  0.88 (t,  $J = 6.6$  Hz, 6H), 1.27-1.57 (m, 20H), 1.57-1.69 (m, 8H), 2.32 (t,  $J = 6.6$  Hz, 4H), 4.06 (t,  $J = 6.7$  Hz, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  173.5, 64.6, 34.0, 31.9, 29.3, 29.2, 28.7, 26.0, 24.5, 22.7, 14.2; IR (**neat**) 2928, 1733, 1457, 1170, 572  $\text{cm}^{-1}$ ; MS (ESI,  $m/z$ ) 393.1 [M+Na]<sup>+</sup>.

### Hexane-1,6-Di(3-Phenylpropanoate) (**3ah**)



A sealed tube was charged with 3-phenylpropanoic acid **1a** (1.5018 g, 10.00 mmol).

Solkane365mc (5.0 mL), hexane-1,6-diol (590.9 mg, 5.0 mmol) and TfOH (2.2  $\mu$ L, 0.01 mmol) were added. The reaction mixture was stirred at 80 °C for 18h, then was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give compound **3ah** (1.6524 g, 93% yield). colorless liquid;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , **300 MHz**)  $\delta$  1.28-1.33 (m, 4H), 1.57-1.61 (m, 4H), 2.63 (t,  $J$  = 7.6 Hz, 4H), 2.95 (t,  $J$  = 7.8 Hz, 4H), 4.05 (t,  $J$  = 6.6 Hz, 4H), 7.18-7.21 (m, 6H), 7.26-7.31 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , **150.9 MHz**)  $\delta$  173.0, 140.6, 128.5, 128.3, 126.3, 64.4, 35.9, 31.0, 28.5, 25.6; IR (**neat**) 3027, 2937, 2860, 2322, 1732, 1604, 1496, 1455, 1361, 1163, 1078, 751, 700  $\text{cm}^{-1}$ ; MS (ESI, *m/z*) 405.1 [M+Na] $^+$ .

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