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Total Synthesis of the Aspercyclides

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Methyl 2-bromo-6-methylbenzoate (7):^[1] A solution of NaNO₂ (1.76 g, 25.6 mmol) in H₂O (6 mL) was added to a cold (0 °C) solution of ester **6** (1.76 g, 10.7 mmol)^[2] in HBr (40 % in H₂O, 12 mL) at such a rate as to maintain the internal temperature ≤ 5 °C. After stirring for 20 min at this temperature, the resulting mixture was transferred via a Teflon tube into a suspension of CuBr (2.75 g, 19.2 mmol) in HBr (40 % in H₂O, 12 mL) and the resulting mixture was stirred for 16 h at ambient temperature. *tert*-Butyl methyl ether (30 mL) was added, the organic phase was washed with aq. sat. NaHCO₃ and brine (10 mL each), dried over MgSO₄ and evaporated. Purification of the residue by flash chromatography (hexanes/*tert*-butyl methyl ether, 1/0 → 10/1) afforded the title compound as a yellow liquid (2.37 g, 97 %). ¹H NMR (400 MHz, CDCl₃): δ = 7.40 (t, *J* = 4.5 Hz, 1 H), 7.15-7.14 (m, 2 H), 3.95 (s, 3 H), 2.33 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ = 168.4, 137.0, 135.9, 130.4, 129.9, 128.9, 119.1, 52.5, 19.7; IR (film): $\tilde{\nu}$ = 3062, 2998, 2952, 1736, 1594, 1566, 1450, 1279, 774 cm⁻¹; MS (EI): *m/z* (%): 228 (47) [*M*⁺], 197 (100), 171 (21), 169 (21), 90 (40), 89 (40); HRMS (EI) calcd for C₉H₉BrO₂ [*M*⁺]: 227.9784, found: 227.9786; elemental analysis calcd (%) for C₉H₉BrO₂: C 47.19, H 3.96; found: C 47.06, H 3.87.

Methyl 2-(2-methoxy-6-vinylphenoxy)-6-methylbenzoate (9): A mixture of bromide **7** (0.98 g, 4.27 mmol), 2-methoxy-6-vinylphenol (**8**) (641 mg, 4.27 mmol), CuO (68.0 mg, 0.85 mmol) and K₂CO₃ (1.20 g, 8.54 mmol) in pyridine (10 mL) was stirred in a sealed tube at 130 °C for 24 h. After reaching ambient temperature, the mixture was poured into HCl (1.5 M, 50 mL), the aqueous phase was extracted with *tert*-butyl methyl ether (3 x 20 mL), the combined organic phases were washed with brine (30 mL), dried over MgSO₄, and evaporated, and the residue was purified by flash chromatography (hexanes/*tert*-butyl methyl ether gradient) to give the title compound as a pale yellow oil (701 mg, 55 %). ¹H NMR (400 MHz, CDCl₃): δ = 7.23-7.14 (m, 2 H), 7.05 (t, *J* = 8.0 Hz, 1 H), 6.91-6.81 (m, 3 H), 6.26 (d, *J* = 8.3 Hz, 1 H), 5.76 (dd, *J* = 17.7, 1.2 Hz, 1 H), 5.25 (dd, *J* = 11.1, 1.2 Hz, 1 H), 3.95 (s, 3 H), 3.74 (s, 3 H), 2.36 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ = 168.5, 155.3, 152.8, 140.4,

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136.7, 132.5, 130.5, 130.0, 125.7, 123.1, 117.9, 116.1, 112.1, 110.8, 95.6, 56.2, 52.2, 19.4; IR (film): $\tilde{\nu}$ = 3010, 2951, 2838, 1732, 1586, 1461, 1440, 1273, 996, 956, 919, 771, 737 cm^{-1} ; MS (EI): m/z (%): 298 (100) [M^+], 267(55), 251 (29), 238 (42), 223 (22), 195 (22); HRMS (ESI): m/z : calcd for $\text{C}_{18}\text{H}_{18}\text{O}_4\text{Na}$ [$M^+ + \text{Na}$]: 321.1095, found: 321.1097; elemental analysis calcd (%) for $\text{C}_{18}\text{H}_{18}\text{O}_4$: C 72.47, H 6.08; found: C 72.56, H 5.96.

2-(2-Methoxy-6-vinylphenoxy)-6-methylbenzoic acid (10): Compound **9** (183 mg, 0.61 mmol) was added to NaOH (2 M, 5 mL) in MeOH (5 mL) and the resulting mixture was stirred under reflux for 48 h. After reaching ambient temperature, the mixture was diluted with water (10 mL), the aqueous phase was extracted with *tert*-butyl methyl ether (10 mL) and the organic layer was discarded. The remaining aqueous phase was acidified to pH \sim 1 with HCl before it was extracted with *tert*-butyl methyl ether (3 x 10 mL). The combined organic phases were washed with brine (10 mL) and dried over MgSO_4 , and the solvent was evaporated to give acid **10** as a pale brown solid (173 mg, quant.). m. p. = 106-107 $^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ = 7.29-7.12 (m, 3 H), 6.94-6.87 (m, 3 H), 6.42 (d, J = 8.3 Hz, 1 H), 5.84 (d, J = 17.7 Hz, 1 H), 5.34 (d, J = 11.1 Hz, 1 H), 3.76 (s, 3 H), 2.55 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ = 167.4, 156.2, 151.4, 140.8, 140.1, 133.0, 131.2, 130.1, 126.1, 125.3, 121.6, 118.3, 117.1, 112.3, 111.8, 56.2, 20.7; IR (film): $\tilde{\nu}$ = 3073, 2839, 2658, 2564, 1699, 1585, 1461, 1414, 1272, 1068, 994, 919, 779 cm^{-1} ; MS (EI): m/z (%): 284 (100) [M^+], 266 (50), 251 (19), 238 (27), 223 (16), 195 (19), 135 (81), 77 (29); HRMS (ESI): m/z : calcd for $\text{C}_{17}\text{H}_{16}\text{O}_4\text{Na}$ [$M^+ + \text{Na}$]: 307.0940, found: 307.0941; elemental analysis calcd (%) for $\text{C}_{17}\text{H}_{16}\text{O}_4$: C 71.85, H 5.67; found: C 71.66, H 5.61.

(3R,4S)-3-(4-Methoxyphenoxy)-1-nonen-4-ol (13): *sec*-BuLi (1.3 M in cyclohexane, 1.71 mL, 2.22 mmol) was slowly added to a solution of 1-(allyloxy)-4-methoxybenzene **11** (365 mg, 2.22 mmol) in THF (12 mL) at -78 $^\circ\text{C}$ and the resulting mixture was stirred for 40 min. The cold solution was then added via canula to a suspension of *S,S*-**12** (1.57 g, 2.56 mmol)^[3] in Et_2O (5 mL) at -78 $^\circ\text{C}$ and stirring was continued for 3 h at this temperature. A solution of

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hexanal (171 mg, 0.21 mL, 1.71 mmol) in Et₂O (5 mL) was then slowly introduced and the resulting mixture stirred for 4 h at -78 °C. The reaction mixture was quenched with aq. sat. NH₄F (10 mL) and stirred for 16 h at ambient temperature, the aqueous phase was repeatedly extracted with *tert*-butyl methyl ether, and the combined organic layers were washed with brine and dried over MgSO₄. Evaporation of the solvent followed by purification of the residue by flash chromatography (CombiFlash[®], hexanes/*tert*-butyl methyl ether gradient) gave product **13** as a yellow oil (313 mg, 69 %, *anti:syn* > 95:5). $[\alpha]_D^{20} = -9.1^\circ$ ($c = 1$, CHCl₃); *ee* = 92 % (HPLC); ¹H NMR (400 MHz, CDCl₃): $\delta = 6.88\text{--}6.79$ (m, 4 H), 5.88 (ddd, $J = 17.4, 10.6, 6.8$ Hz, 1 H), 5.34 (d, $J = 9.6$ Hz, 1 H), 5.32 (d, $J = 17.3$ Hz, 1 H), 4.45–4.43 (m, 1 H), 3.86–3.82 (m, 1 H), 3.76 (s, 3 H), 1.59–1.29 (m, 8 H), 0.90 (t, $J = 6.8$ Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 154.3, 151.9, 134.1, 119.5, 117.6, 117.6, 114.6, 114.6, 83.6, 73.3, 55.7, 32.2, 31.8, 25.5, 22.6, 14.0$; IR (film): $\tilde{\nu} = 3438, 3063, 2953, 2932, 1643, 1591, 1507, 1227, 1039, 994, 928, 824$ cm⁻¹; MS (EI) m/z (rel. intensity): 264 (7) [M^+], 164 (11), 124 (100), 109 (12); HRMS (ESI) calcd for C₁₆H₂₄O₃Na [$M^+ + Na$] 287.1623, found: 287.1618; elemental analysis calcd for C₁₆H₂₄O₃: C 72.69, H 9.15; found: C 72.60, H 9.16.

Methyl 2-(2,2-dimethyl-5-vinyl-4*H*-benzo[*d*][1,3]dioxin-6-yloxy)-6-methylbenzoate (22):

A mixture of phenol **21** (1.12 g, 5.43 mmol), bromide **7** (500 mg, 2.71 mmol), 2,2,6,6-tetramethylheptane-3,5-dione (0.14 mL, 0.543 mmol), Cs₂CO₃ (1.86 g, 5.70 mmol) and CuCl (26.9 mg, 0.271 mmol) in pyridine (14 mL) was stirred at reflux temperature in a sealed Schlenk tube for 3 d. The color of the mixture gradually turned from yellow to dark-black brown within the first hour of heating. For work up, the mixture was cooled to ambient temperature before water (50 mL) was added, the resulting light-brown aqueous phase was extracted with EtOAc (3 x 100 mL), the combined extracts were washed with brine (50 mL) and dried over MgSO₄, and the solvents were evaporated. The resulting brown-black residue was purified by flash chromatography (hexanes/EtOAc, 20:1) to give product **22** as a pale yellow oil (490 mg, 51 %). ¹H NMR (300 MHz, CDCl₃): $\delta = 7.13$ (dd, $J = 8.1, 7.9$ Hz, 1 H), 6.87 (dt, $J = 7.6, 0.7$ Hz, 1 H), 6.86 (d, $J = 8.9$ Hz, 1 H), 6.73 (d, $J = 8.9$ Hz, 1 H), 6.54 (dd, $J = 17.8, 11.7$ Hz, 1 H), 6.48 (d, $J = 8.2$ Hz, 1 H), 5.59 (dd, $J = 18.0, 1.5$ Hz, 1 H), 5.49 (dd,

$J = 11.8, 1.5$ Hz, 1 H), 4.90 (s, 2 H), 3.90 (s, 3 H), 2.36 (s, 3 H), 1.57 (s, 6 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 168.3, 155.2, 147.9, 146.7, 137.0, 130.2, 128.4, 127.0, 124.6, 123.8, 121.2, 120.8, 118.3, 117.1, 113.3, 98.8, 60.4, 52.2, 24.6$; IR (film): $\tilde{\nu} = 3061, 2993, 2950, 1733, 1630, 1461, 1385, 1274, 1254, 971, 873, 827, 779$ cm^{-1} ; MS (EI): m/z (%): 354 (18) [M^+], 296 (100), 264 (94), 247 (13), 237 (77), 209 (17), 162 (37), 147 (12), 135 (24), 91 (29), 77 (27), 65 (19); HRMS (ESI): m/z : calcd for $\text{C}_{21}\text{H}_{22}\text{O}_5\text{Na}$: 377.1359, found 377.1359; elemental analysis (%) calcd for $\text{C}_{21}\text{H}_{22}\text{O}_5$: C 71.17, H 6.26; found: C 71.08, H 6.16.

2-[(2,2-Dimethyl-5-vinyl-4*H*-1,3-benzodioxin-6-yl)oxy]-6-methylbenzoic acid (23): A solution of compound **22** (27.8 mg, 0.08 mmol) and KOH (25 % in MeOH, *w/w*, 6 mL) in MeOH (3 mL) was stirred under reflux for 24 h. After reaching ambient temperature, the mixture was diluted with water (10 mL) and extracted with *tert*-butyl methyl ether (15 mL), and the organic layer was discarded. The remaining aqueous phase was acidified to pH 5 with HCl (3 M) before it was extracted with *tert*-butyl methyl ether (3 x 10 mL). The combined extracts were washed with brine (10 mL), dried over MgSO_4 and evaporated to give acid **23** as a pale brown solid (25.6 mg, 96 %). m. p. = 156-157 °C; ^1H NMR (400 MHz, CDCl_3): $\delta = 7.16$ (t, $J = 8.0$ Hz, 1 H), 6.90 (d, $J = 7.6$ Hz, 1 H), 6.88 (d, $J = 8.8$ Hz, 1 H), 6.74 (d, $J = 8.8$ Hz, 1 H), 6.57-6.47 (m, 2 H), 5.56 (dd, $J = 18.0, 1.4$ Hz, 1 H), 5.47 (dd, $J = 11.8, 1.4$ Hz, 1 H), 4.88 (s, 2 H), 2.49 (s, 3 H), 1.56 (s, 6 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 170.4, 156.0, 148.3, 146.0, 138.8, 131.0, 128.3, 127.4, 124.5, 122.3, 121.5, 121.1, 118.4, 117.2, 113.2, 98.9, 60.3, 24.6, 20.3$; IR (film): $\tilde{\nu} = 3415, 3086, 1702, 1630, 1587, 1457, 1385, 1284, 1245, 832, 784$ cm^{-1} ; MS (EI): m/z (%): 340 (14) [M^+], 282 (72), 264 (31), 236 (23), 210 (8), 182 (5), 165 (9), 135 (100), 119 (7), 103 (20), 91 (14), 77 (23); HRMS (ESI): m/z : calcd for $\text{C}_{20}\text{H}_{20}\text{O}_5\text{Na}$ [$M^+ + \text{Na}$]: 363.1203, found: 363.1203; elemental analysis calcd (%) for $\text{C}_{20}\text{H}_{20}\text{O}_5$: C 70.57, H 5.92; found: C 70.50, H 6.08.

Compound 24: 2-Chloro-1-methylpyridinium iodide (91.2 mg, 0.35 mmol) was added to a solution of acid **23** (80.2 mg, 0.24 mmol), alcohol **13** (31.0 mg, 0.12 mmol) and NEt_3 (48 mg, 0.5 mmol) in MeCN (1.4 mL), and the resulting mixture was stirred under reflux for 4 d. A standard extractive work up followed by flash chromatography (hexanes/EtOAc, 15:1)

furnished ester **24** as a colorless oil (33 mg, 49 %). $[\alpha]_D^{20} = +3.1^\circ$ ($c = 1.2$, CHCl_3); ^1H NMR (400 MHz, CDCl_3): $\delta = 7.09$ (t, $J = 7.9$ Hz, 1 H), 6.83 (d, $J = 7.6$ Hz, 1 H), 6.79-6.76 (m, 3 H), 6.72-6.66 (m, 3 H), 6.52-6.39 (m, 2 H), 5.90 (ddd, $J = 17.1, 10.6, 6.2$ Hz, 1 H), 5.52 (dd, $J = 18.0, 1.4$ Hz, 1 H), 5.43-5.28 (m, 4 H), 4.87 (s, 2H), 4.74-4.72 (m, 1 H), 3.73 (s, 3 H), 2.33 (s, 3 H), 1.87-1.66 (m, 2 H), 1.56 (s, 6 H), 1.53-1.13 (m, 6 H), 0.79 (t, $J = 7.1$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 167.7, 155.3, 154.0, 152.0, 147.9, 146.6, 136.9, 134.2, 130.0, 128.5, 127.1, 125.0, 123.7, 121.1, 121.0, 119.1, 118.1, 117.2, 117.1, 114.3, 113.0, 98.8, 80.8, 76.2, 60.4, 55.6, 31.6, 29.2, 25.2, 24.7, 24.6, 22.5, 19.3, 14.0$; IR (film): $\tilde{\nu} = 2955, 1729, 1507, 1461, 1384, 1254, 1146, 1038, 992, 971, 874, 825, 778$ cm^{-1} ; MS (EI): m/z (%): 586 (14) [M^+], 528 (65), 513 (3), 463 (12), 405 (53), 323 (3), 265 (100), 237 (17), 209 (8), 173 (15), 123 (15); HRMS (ESI): m/z : calcd for $\text{C}_{36}\text{H}_{42}\text{O}_7\text{Na}$ [$M + \text{Na}^+$]: 609.2833, found: 609.2825; elemental analysis calcd (%) for $\text{C}_{36}\text{H}_{42}\text{O}_7$: C 73.70, H 7.22; found: C 73.64, H 7.18.

(S,E)-1-Oxoheptan-2-yl 2-(2-(2-iodovinyl)phenoxy)-6-methylbenzoate (54): Prepared as described for compound **43** in the main text; colorless oil (41 mg, 82 %). $[\alpha]_D^{20} = -8.1$ ($c = 0.83$, CH_2Cl_2); ^1H NMR (400 MHz, CDCl_3): $\delta = 9.52$ (s, 1 H), 7.67 (d, $J = 15.0$ Hz, 1 H), 7.40 (dd, $J = 8.0, 1.2$ Hz, 1 H), 7.18-7.25 (m, 2 H), 7.10 (t, $J = 7.5$ Hz, 1 H), 7.03 (d, $J = 7.3$ Hz, 1 H), 7.02 (d, $J = 15.0$ Hz, 1 H), 6.85 (d, $J = 8.2$ Hz, 1 H), 6.67 (d, $J = 8.0$ Hz, 1 H), 5.19 (dd, $J = 8.3, 4.6$ Hz, 1 H), 2.48 (s, 3 H), 1.67-1.84 (m, 2 H), 1.11-1.40 (m, 6 H), 0.86 (t, $J = 7.6$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 198.5, 167.3, 154.3, 153.7, 139.6, 138.4, 131.3, 131.2, 129.7, 129.1, 127.7, 125.8, 124.2, 119.3, 116.1, 79.7, 79.4, 31.6, 29.0, 24.8, 22.5, 19.8, 14.1$; IR (film): $\tilde{\nu} = 3059, 2955, 2929, 2860, 2871, 1734, 1603, 1582, 1463, 1451, 1256, 1195, 1111, 1074, 957, 755$ cm^{-1} ; MS (EI): m/z (%): 493 (9) [$M^+ + 1$], 492 (35) [M^+], 368 (39), 364 (14), 363 (82), 253 (6), 238 (14), 237 (55), 236 (30), 235 (100), 221 (11), 209 (19), 208 (18), 207 (12), 181 (21), 118 (19), 95 (10), 89 (8); HRMS (EI): m/z : calcd for $\text{C}_{23}\text{H}_{25}\text{O}_4\text{I}$: 492.0798, found 492.0795.

Compound 52: Prepared as described for compound **44** in the main text, using compound **51** as the substrate; colorless solid (12.2 mg, 58 %, *anti:syn* = 3:1). $[\alpha]_{\text{D}}^{20} = +65.4$ ($c = 0.43$, CH_2Cl_2); ^1H NMR (400 MHz, CDCl_3 , resolved signals of the minor isomer are marked *): $\delta = 7.24\text{-}7.34$ (m, 6 H), $7.00\text{-}7.07$ (m, 4 H), 6.80 (d, $J = 8.8$ Hz, 2 H), $5.97\text{-}6.14$ (m, 4 H), 5.43^* (ddd, $J = 8.0, 5.7, 1.5$ Hz, 1 H), 5.20 (td, $J = 9.4, 2.6$ Hz, 1 H), 4.70^* (d, $J = 15.7$ Hz, 1 H), 4.62^* (d, $J = 15.7$ Hz, 1H), 4.64 (s, 2 H), $4.08\text{-}4.12$ (m, 2 H), $1.69\text{-}1.96$ (m, 4 H), 1.53^* (s, 6 H), 1.52 (s, 6 H), $1.22\text{-}1.49$ (m, 12 H), 0.93 (t, $J = 6.9$ Hz, 3 H), 0.91^* (t, $J = 6.9$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 166.5, 166.3, 152.5, 152.4, 143.1, 142.5, 138.3, 130.5, 126.0, 125.5, 123.5, 122.2, 117.3, 116.0, 93.6, 93.2, 78.0, 76.8, 60.0, 59.4, 31.2, 31.2, 25.0, 24.8, 24.6, 24.3, 23.9, 22.2, 20.7, 13.8, 13.7$; IR (film): $\tilde{\nu} = 3440, 2962, 2932, 2850, 1740, 1705, 1601, 1465, 1448, 1373, 1279, 1214, 1128, 964, 873, 748$ cm^{-1} ; MS (EI): m/z (%): 439 (2) [$M^+ + 1$], 438 (8) [M^+], 380 (26), 280 (44), 279 (11), 263 (11), 262 (10), 251 (100), 235 (24), 160 (24), 159 (12), 121 (15); HRMS (ESI): m/z : calcd for $\text{C}_{26}\text{H}_{30}\text{O}_6\text{Na}$: 461.1935, found 461.1939.

Compound 55: Prepared as described for compound **44** in the main text; colorless oil (17.4 mg, 59 %, *anti:syn* = 10:1). $[\alpha]_{\text{D}}^{20} = +138.1$ ($c = 1.54$, CH_2Cl_2); ^1H NMR (400 MHz, CDCl_3): $\delta = 7.18\text{-}7.41$ (m, 4 H), 7.11 (t, $J = 8.0$ Hz, 1 H), 6.86 (d, $J = 7.6$ Hz, 1 H), 6.79 (d, $J = 8.5$ Hz, 1 H), 6.32 (d, $J = 15.9$ Hz, 1 H), 6.03 (dd, $J = 15.9, 9.4$ Hz, 1 H), 5.25 (td, $J = 9.8, 2.0$ Hz, 1 H), 4.09 (t, $J = 9.2$ Hz, 1 H), 2.37 (s, 3 H), 2.09 (dddd, $J = 12.1, 9.5, 7.0, 2.3$ Hz, 1 H), 1.86 (br s, 1 H), 1.72 (dddd, $J = 14.2, 9.8, 9.5, 5.0$ Hz, 1 H), $1.27\text{-}1.57$ (m, 6 H), 0.93 (t, $J = 6.8$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 168.2, 154.5, 153.2, 137.8, 135.6, 132.4, 130.4, 129.9, 128.24, 128.17, 125.3, 125.1, 124.3, 122.5, 113.7, 77.4, 77.3, 32.0, 31.8, 25.5, 22.7, 19.6, 14.2$; IR (film): $\tilde{\nu} = 3420, 3068, 3024, 2954, 2927, 2860, 1732, 1602, 1582, 1573, 1484, 1463, 1445, 1253, 1239, 1190, 1106, 1071, 1036, 959, 774, 747$ cm^{-1} ; MS (EI): m/z (%): 366 (1) [M^+], 267 (13), 266 (69), 248 (14), 238 (26), 237 (100), 220 (8), 195 (14), 194 (6), 165 (6); HRMS (ESI): m/z : calcd for $\text{C}_{23}\text{H}_{26}\text{O}_4\text{Na}$: 389.1723, found 389.1721.