

# Correction to Ring Expansion of Cyclic $\beta$ -Amino Alcohols Induced by Diethylaminosulfur Trifluoride: Synthesis of Cyclic Amines with a Tertiary Fluoride at C3

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## S Supporting Information

In the Supporting Information of this paper, the presence of solvents was visible in the spectra of **4e**, **8**, and **27**. Compounds **4e**, **8**, and **27** were resynthesized, and new spectra are provided in the revised Supporting Information submitted with this correction.

The spectra for **3c,e**, **4a–d**, **5a–c**, **7**, **11**, **12**, **15a–c**, **16a–c**, **19a–c**, **23b–d**, **24a,b,d**, **26**, **28**, **29**, and **31** were found to have been edited to remove solvents and impurities. Original products were located, and the spectra were rerecorded and have been replaced for **3c,e**, **4a,b**, **5a–c**, **7**, **11**, **15b**, **16b**, **19a–c**, **23b,c**, **24b,d**, **26**, **28**, and **29**. Compounds **4c,d**, **12**, **15a,c**, **16a,c**, **23d**, **24a**, and **31** were resynthesized, and the new spectra are provided in the revised Supporting Information submitted with this correction. The spectra editing did not affect any of the conclusions of the published paper. The yields were found to be correct.

The revised spectra  $^1\text{H}$  and  $^{13}\text{C}$  NMR data are given below:

**2d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 3.60 (s, 3H), 2.50 (m, 1H), 2.36 (m, 1H), 2.02 (m, 1H), 1.90 (m, 1H), 1.67–1.30 (m, 8H), 0.73 (t, 3H,  $J$  = 8.4 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 209.3 (s), 172.8 (s), 63.2 (s), 51.8 (q), 41.9 (t), 32.2 (t), 29.8 (t), 28.2 (t), 25.4 (t), 24.8 (t), 8.9 (q) ppm.

**3a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.07 (s br, 1H), 3.77 (s, 3H), 2.51 (m, 1H), 2.34 (m, 1H), 2.18 (m, 1H), 1.95–1.65 (m, 3H), 1.82 (q, 2H,  $J$  = 7.6 Hz), 1.56 (m, 2H), 0.89 (t, 3H,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 177.8 (s), 173.6 (s), 63.6 (s), 52.6 (q), 37.8 (t), 37.4 (t), 33.4 (t), 26.1 (t), 22.9 (t), 8.1 (q) ppm.

**3b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 5.85 (s br, 1H), 5.60 (m, 1H), 5.23 (m, 2H), 3.78 (s, 3H), 2.61 (dd, 1H,  $J$  = 14.1, 6.0, 1.2, and 1.2 Hz), 2.54 (m, 1H), 2.44–2.25 (m, 3H), 1.91 (m, 1H), 1.79–1.65 (m, 2H), 1.60–1.47 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 177.5 (s), 173.3 (s), 130.7 (d), 121.5 (t), 62.5 (s), 52.6 (q), 45.5 (t), 38.5 (t), 37.7 (t), 26.4 (t), 22.8 (t) ppm.

**3c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.36–7.27 (m, 3H), 7.07 (m, 2H), 5.88 (s br, 1H), 3.74 (s, 3H), 3.21 (d, 1H,  $J$  = 13.5 Hz), 2.96 (d, 1H,  $J$  = 13.5 Hz), 2.55 (dd, 1H,  $J$  = 15.3 and 7.5 Hz), 2.43–2.28 (m, 2H), 1.95 (m, 1H), 1.86 (m, 1H), 1.73 (m, 1H), 1.59 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 177.3 (s), 173.2 (s), 134.2 (s), 129.7 (d, 2C), 129.0 (d, 2C), 127.9 (d), 63.8 (s), 52.6 (q), 46.4 (t), 38.9 (t), 37.7 (t), 26.0 (t), 22.8 (t) ppm.

**3d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.49 (s br, 1H), 3.71 (s, 3H), 2.40–2.18 (m, 3H), 1.85–1.37 (m, 9H), 0.80 (t, 3H,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 176.0 (s), 173.7

(s), 64.2 (s), 52.5 (q), 38.5 (t), 35.1 (t), 32.4 (t), 25.8 (t), 24.9 (t), 22.5 (t), 8.2 (q) ppm.

**3e.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.34–7.24 (m, 3H), 7.10 (m, 2H), 6.15 (s br, 1H), 3.75 (s, 3H), 3.02 (m, 2H), 2.47 (dm, 1H,  $J$  = 15.7 Hz), 2.32–2.04 (m, 3H), 1.81–1.60 (m, 4H), 1.57–1.43 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 175.6 (s), 173.4 (s), 134.3 (s), 130.3 (d, 2C), 128.6 (d, 2C), 127.6 (d), 65.0 (s), 52.8 (q), 47.7 (t), 36.4 (t), 33.6 (t), 25.0 (t), 24.8 (t), 22.9 (t) ppm.

**4a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.37–7.29 (m, 4H), 7.24 (m, 1H), 3.87 (d, 1H,  $J$  = 13.7 Hz), 3.61 (dd, 1H,  $J$  = 10.3 and 1.7 Hz), 3.55 (d, 1H,  $J$  = 10.4 Hz), 3.49 (d, 1H,  $J$  = 13.9 Hz), 2.81 (ddd, 1H,  $J$  = 14.5, 9.8, and 1.7 Hz), 2.62 (ddd, 1H,  $J$  = 14.7, 7.0, and 2.3 Hz), 1.97–1.86 (m, 2H), 1.73–1.46 (m, 7H), 1.30–1.19 (m, 2H), 0.94 (t, 3H,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.6 (s), 128.7 (d, 2C), 128.4 (d, 2C), 126.8 (d), 67.9 (t), 62.0 (s), 53.7 (t), 47.1 (t), 37.6 (t), 31.3 (t), 29.8 (t), 27.1 (t), 23.3 (t), 8.7 (q) ppm.

**4b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.37–7.29 (m, 4H), 7.24 (m, 1H), 5.88 (m, 1H), 5.16–5.05 (m, 2H), 3.91 (d, 1H,  $J$  = 13.5 Hz), 3.67 (dd, 1H,  $J$  = 10.6 and 1.6 Hz), 3.55 (d, 1H,  $J$  = 13.7 Hz), 3.54 (d, 1H,  $J$  = 10.8 Hz), 3.27 (s br, 1H), 2.81 (ddd, 1H,  $J$  = 14.7, 9.8, and 1.7 Hz), 2.60 (ddd, 1H,  $J$  = 15.0, 6.8, and 2.2 Hz), 2.38 (dd, 1H,  $J$  = 13.6, 7.2, 1.2, and 1.2 Hz), 2.26 (dd, 1H,  $J$  = 13.5 and 7.8 Hz), 1.94–1.83 (m, 2H), 1.76–1.58 (m, 3H), 1.50 (m, 1H), 1.32–1.17 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.6 (s), 134.5 (d), 128.7 (d, 2C), 128.4 (d, 2C), 126.9 (d), 117.9 (t), 68.2 (t), 62.0 (s), 54.1 (t), 47.3 (t), 39.4 (t), 38.4 (t), 31.4 (t), 29.8 (t), 23.3 (t) ppm.

**4c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.48–7.22 (m, 10H), 4.08 (d, 1H,  $J$  = 13.4 Hz), 3.87 (d, 1H,  $J$  = 10.5 Hz), 3.77 (d, 1H,  $J$  = 13.5 Hz), 3.55 (d, 1H,  $J$  = 10.8 Hz), 3.32 (s br, 1H), 2.96 (d, 1H,  $J$  = 13.0 Hz), 2.89 (d, 1H,  $J$  = 12.9 Hz), 2.84 (m, 1H), 2.72 (dd, 1H,  $J$  = 14.9 and 1.9 Hz), 1.88 (m, 1H), 1.78–1.50 (m, 5H), 1.40–1.24 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.7 (s), 138.2 (s), 130.5 (d, 2C), 128.7 (d, 2C), 128.4 (d, 2C), 128.1 (d, 2C), 127.0 (d), 126.2 (d), 67.7 (t), 62.9 (s), 54.3 (t), 47.1 (t), 40.2 (t), 38.1 (t), 31.3 (t), 29.8 (t), 23.2 (t) ppm.

**4d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.34–7.14 (m, 5H), 3.80 (d, 1H,  $J$  = 12.5 Hz), 3.60 (dd, 1H,  $J$  = 10.4 and 1.4 Hz), 3.51 (d, 1H,  $J$  = 10.4 Hz), 3.45 (s br, 1H), 3.39 (d, 1H,  $J$  = 12.5 Hz), 2.98 (ddd, 1H,  $J$  = 15.0, 11.7, and 3.1 Hz), 2.56 (ddd, 1H,

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*J* = 15.2, 4.1, and 4.1 Hz), 1.86–1.22 (m, 10H), 0.92 (m, 1H), 0.85 (t, 3H, *J* = 7.8 Hz), 0.67 (m, 1H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.9 (s), 129.4 (d, 2C), 128.3 (d, 2C), 127.0 (d), 67.1 (t), 62.3 (s), 55.1 (t), 47.8 (t), 34.1 (t), 30.3 (t), 27.5 (t), 26.8 (t), 25.9 (t), 25.0 (t), 8.9 (q) ppm.

**4e.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.47–7.19 (m, 10H), 4.11 (d, 1H, *J* = 12.4 Hz), 3.92 (d, 1H, *J* = 10.6 Hz), 3.62 (d, 1H, *J* = 9.8 Hz), 3.59 (d, 1H, *J* = 8.4 Hz), 3.26 (s br, 1H), 3.03 (ddd, 1H, *J* = 15.1, 11.9, and 2.8 Hz), 2.90 (d, 1H, *J* = 13.1 Hz), 2.81 (d, 1H, *J* = 13.1 Hz), 2.60 (ddd, 1H, *J* = 15.4, 4.0, and 4.0 Hz), 1.93–1.25 (m, 8H), 1.06 (m, 1H), 0.74 (m, 1H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.8 (s), 138.3 (s), 130.6 (d, 2C), 129.4 (d, 2C), 128.3 (d, 2C), 128.1 (d, 2C), 127.1 (d), 126.2 (d), 67.2 (t), 63.5 (s), 55.9 (t), 48.4 (t), 39.6 (t), 34.8 (t), 30.6 (t), 27.4 (t), 25.8 (t), 25.1 (t) ppm.

**5a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.38–7.27 (m, 4H), 7.23 (dddd, 1H, *J* = 7.1, 7.1, 1.5, and 1.5 Hz), 3.79 (d, 1H, *J* = 13.5 Hz), 3.69 (d, 1H, *J* = 13.5 Hz), 2.86 (dd, 1H, *J* = 15.3 and 15.3 Hz), 2.61 (dd, 1H, *J* = 27.3 and 15.3 Hz), 2.50 (m, 1H), 2.39 (m, 1H), 2.15 (ddm, 1H, *J* = 22.3 and 12.0 Hz), 1.88–1.74 (m, 2H), 1.69–1.36 (m, 7H), 0.94 (t, 3H, *J* = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.3 (s), 128.8 (d, 2C), 128.1 (d, 2C), 126.8 (d), 100.3 (ds, *J* = 169 Hz), 64.0 (t), 59.4 (dt, *J* = 29 Hz), 54.8 (t), 33.6 (dt, *J* = 24 Hz), 29.9 (dt, *J* = 23 Hz), 27.1 (t), 26.7 (t), 22.7 (dt, *J* = 10 Hz), 7.26 (dq, *J* = 5 Hz) ppm.

**5b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.38–7.27 (m, 4H), 7.23 (m, 1H), 5.88 (m, 1H), 5.15–5.03 (m, 2H), 3.77 (d, 1H, *J* = 13.5 Hz), 3.68 (d, 1H, *J* = 13.5 Hz), 2.86 (dd, 1H, *J* = 15.0 and 15.0 Hz), 2.65 (dd, 1H, *J* = 26.3 and 15.3 Hz), 2.52–2.35 (m, 2H), 2.35 (dddd, 2H, *J* = 22.1, 7.0, 1.2, and 1.2 Hz), 2.11 (m, 1H), 1.90–1.75 (m, 2H), 1.66–1.38 (m, 5H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.3 (s), 133.1 (dd, *J* = 5 Hz), 128.7 (d, 2C), 128.1 (d, 2C), 126.8 (d), 118.2 (t), 99.5 (ds, *J* = 171 Hz), 64.0 (t), 59.3 (dt, *J* = 26 Hz), 54.8 (t), 42.3 (dt, *J* = 21 Hz), 34.4 (dt, *J* = 23 Hz), 27.0 (t), 26.8 (t), 22.4 (dt, *J* = 9 Hz) ppm.

**5c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.38–7.16 (m, 10H), 3.78 (d, 1H, *J* = 13.5 Hz), 3.65 (d, 1H, *J* = 13.5 Hz), 2.90 (dd, 1H, *J* = 25.5 and 14.4 Hz), 2.84 (dd, 1H, *J* = 19.4 and 14.4 Hz), 2.83 (dd, 1H, *J* = 14.8 and 14.8 Hz), 2.69 (dd, 1H, *J* = 26.8 and 15.3 Hz), 2.53–2.35 (m, 2H), 2.03 (m, 1H), 1.92–1.41 (m, 7H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.3 (s), 136.7 (s), 130.7 (d), 130.6 (d), 128.8 (d, 2C), 128.2 (d, 2C), 128.1 (d, 2C), 126.9 (d), 126.5 (d), 99.9 (ds, *J* = 173 Hz), 64.1 (t), 59.6 (dt, *J* = 25 Hz), 54.6 (t), 43.7 (dt, *J* = 21 Hz), 34.0 (dt, *J* = 23 Hz), 27.1 (t), 27.0 (t), 22.7 (dt, *J* = 9 Hz) ppm.

**5d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.32–7.12 (m, 5H), 3.86 (d, 1H, *J* = 13.3 Hz), 3.46 (d, 1H, *J* = 13.3 Hz), 2.63–2.51 (m, 2H), 2.31–2.17 (m, 3H), 1.74–1.04 (m, 11H), 0.89 (t, 3H, *J* = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.5 (s), 129.1 (d, 2C), 128.1 (d, 2C), 126.7 (d), 102.3 (ds, *J* = 170 Hz), 63.1 (dt, *J* = 3 Hz), 56.0 (dt, *J* = 22 Hz), 53.7 (t), 28.8 (dt, *J* = 22 Hz), 28.4 (dt, *J* = 23 Hz), 27.3 (t), 22.5 (t), 19.7 (t), 18.2 (dt, *J* = 11 Hz), 7.1 (dq, *J* = 5 Hz) ppm.

**5e.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.32–7.10 (m, 10H), 3.91 (d, 1H, *J* = 13.3 Hz), 3.41 (d, 1H, *J* = 13.3 Hz), 2.85–2.53 (m, 4H), 2.31–2.14 (m, 3H), 1.66–1.09 (m, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.4 (s), 136.5 (s), 130.5 (d), 130.4 (d), 129.1 (d, 2C), 128.1 (d, 2C), 128.0 (d, 2C), 126.7 (d), 126.4 (d), 102.1 (ds, *J* = 173 Hz), 63.1 (dt, *J* = 3 Hz), 56.1 (dt, *J* = 21 Hz), 53.5 (t), 42.4 (dt, *J* = 21 Hz), 28.3 (dt, *J* = 23 Hz), 27.4 (t), 22.3 (t), 19.3 (t), 18.4 (dt, *J* = 11 Hz) ppm.

**7.**  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ , 122 °C)  $\delta$ : 7.42–7.21 (m, 10H), 5.19 (d, 1H, *J* = 12.3 Hz), 5.14 (d, 1H, *J* = 12.3 Hz), 3.88 (ddd, 1H, *J* = 12.8, 4.0, and 1.0 Hz), 3.81 (d, 1H, *J* = 13.8 Hz), 3.65 (d, 1H, *J* = 13.5 Hz), 3.57 (dddm, 1H, *J* = 13.1, 4.7, and 4.7 Hz), 3.45–3.37 (m, 2H), 3.17 (ddd, 1H, *J* = 12.7, 8.9, and 3.7 Hz), 3.01 (ddd, 1H, *J* = 11.7, 8.6, and 3.3 Hz), 2.40 (ddd, 1H, *J* = 11.5, 4.3, and 4.3 Hz), 1.40 (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ , 122 °C)  $\delta$ : 171.1 (s), 154.3 (s), 138.7 (s), 136.6 (s), 129.0 (d, 2C), 128.8 (d, 2C), 128.5 (d, 2C), 128.4 (d), 128.3 (d, 2C), 127.4 (d), 79.6 (s), 66.2 (t), 61.4 (d), 59.3 (t), 47.6 (t), 46.2 (t), 43.8 (t), 28.6 (q, 3C) ppm.

**8.**  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ , 122 °C) mixture of rotamers  $\delta$ : 7.37–7.26 (m, 4H), 7.20 (m, 1H), 6.00 (m, 1H), 5.16–5.04 (m, 2H), 3.98 (s br, 1H), 3.86 (d, 1H, *J* = 14.6 Hz), 3.74 (d, 1H, *J* = 14.8 Hz), 3.64 (d, 1H, *J* = 10.5 Hz), 3.58 (d, 1H, *J* = 10.8 Hz), 3.40–3.19 (m, 4H), 2.58–2.50 (m, 2H), 2.47 (ddm, 1H, *J* = 14.0 and 7.0 Hz), 2.37 (ddm, 1H, *J* = 14.0 and 7.3 Hz), 1.44 (1.45) (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ , 122 °C)  $\delta$ : 153.6 (s), 140.0 (s), 134.1 (d), 127.4 (d, 2C), 127.3 (d, 2C), 125.7 (d), 116.3 (t), 78.1 (s), 61.9 (t), 58.5 (t), 52.4 (s), 47.8 (t), 44.4 (t), 43.0 (t), 33.4 (t), 27.5 (q, 3C) ppm.

**10.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.35–7.24 (m, 5H), 3.67 (s, 2H), 3.50 (s, 4H), 2.78 (s br, 3H), 1.03 (s, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.1 (s), 128.6 (d, 2C), 128.4 (d, 2C), 127.2 (d), 67.0 (t, 2C), 57.0 (s), 46.1 (t), 18.3 (q) ppm.

**11.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.36–7.23 (m, 5H), 4.77 (d, 1H, *J* = 15.7 Hz), 4.60 (d, 1H, *J* = 15.7 Hz), 4.35 (d, 1H, *J* = 16.7 Hz), 4.30 (d, 1H, *J* = 16.5 Hz), 4.01 (d, 1H, *J* = 12.0 Hz), 3.69 (d, 1H, *J* = 11.8 Hz), 3.61 (d, 1H, *J* = 11.7 Hz), 3.45 (d, 1H, *J* = 11.8 Hz), 1.74 (s br, 1H), 1.15 (s, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 168.7 (s), 138.5 (s), 128.8 (d, 2C), 127.4 (d), 127. (d, 2C), 72.2 (t), 68.2 (t), 65.7 (t), 59.7 (s), 44.5 (t), 18.8 (q) ppm.

**12.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.40–7.25 (m, 5H), 3.97 (d, 1H, *J* = 13.1 Hz), 3.80 (d, 1H, *J* = 11.1 Hz), 3.79 (d, 1H, *J* = 11.1 Hz), 3.75 (m, 1H), 3.52 (m, 1H), 3.50 (d, 1H, *J* = 11.6 Hz), 3.25 (d, 1H, *J* = 11.4 Hz), 3.19 (d, 1H, *J* = 13.1 Hz), 2.63–2.53 (m, 2H), 1.14 (s, 3H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 138.3 (s), 128.9 (d, 2C), 128.5 (d, 2C), 127.3 (d), 74.0 (t), 67.6 (t), 63.8 (t), 57.1 (s), 53.3 (t), 45.8 (t), 12.9 (q) ppm.

**15a.**  $^1\text{H}$  NMR (400 MHz,  $\text{C}_6\text{D}_6$ )  $\delta$ : 7.29–7.24 (m, 2H), 7.16–7.11 (m, 2H), 7.06 (m, 1H), 6.99 (dm, 1H, *J* = 7.2 Hz), 6.92 (ddm, 1H, *J* = 7.7 and 7.7 Hz), 6.69 (ddd, 1H, *J* = 7.3, 7.3 and 1.0 Hz), 6.22 (d, 1H, *J* = 7.8 Hz), 4.47 (d, 1H, *J* = 17.1 Hz), 4.31 (d, 1H, *J* = 17.1 Hz), 3.50 (d, 1H, *J* = 16.1 Hz), 3.14 (s, 3H), 2.92 (d, 1H, *J* = 16.1 Hz), 1.89 (dq, 1H, *J* = 14.3 and 7.5 Hz), 1.71 (dq, 1H, *J* = 14.3 and 7.2 Hz), 0.74 (t, 3H, *J* = 7.4 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{C}_6\text{D}_6$ )  $\delta$ : 174.0 (s), 151.5 (s), 139.8 (s), 128.7 (d, 2C), 128.1 (d), 127.9 (d), 126.9 (d, 2C), 126.8 (s), 124.0 (d), 118.1 (d), 107.0 (d), 74.9 (s), 51.4 (q), 49.0 (t), 38.0 (t), 28.2 (t), 8.6 (q) ppm.

**15b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.36–7.27 (m, 4H), 7.23 (m, 1H), 7.03 (dd, 1H, *J* = 7.0 and 0.8 Hz), 6.93 (ddd, 1H, *J* = 7.7, 7.7, and 0.8 Hz), 6.63 (ddd, 1H, *J* = 7.5, 7.5, and 1.0 Hz), 6.12 (d, 1H, *J* = 7.8 Hz), 5.75 (m, 1H), 5.16–5.06 (m, 2H), 4.51 (d, 1H, *J* = 16.8 Hz), 4.39 (d, 1H, *J* = 16.8 Hz), 3.63 (s, 3H), 3.48 (d, 1H, *J* = 16.3 Hz), 3.24 (d, 1H, *J* = 16.3 Hz), 2.78 (dddm, 1H, *J* = 14.3, 7.5, 1.0, and 1.0 Hz), 2.68 (dddm, 1H, *J* = 14.3, 6.8, 1.3, and 1.3 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 174.1 (s), 150.9 (s), 139.0 (s), 132.6 (d), 128.5 (d,

2C), 127.5 (d), 126.7 (d), 126.6 (d, 2C), 126.4 (s), 123.7 (d), 119.3 (t), 117.7 (d), 106.6 (d), 73.8 (s), 52.2 (q), 49.0 (t), 39.6 (t), 38.2 (t) ppm.

**15c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.34–7.09 (m, 10H), 7.01 (d, 1H,  $J$  = 6.8 Hz), 6.91 (dd, 1H,  $J$  = 7.2 and 7.2 Hz), 6.62 (dd, 1H,  $J$  = 7.1 and 7.1 Hz), 6.12 (d, 1H,  $J$  = 7.8 Hz), 4.60 (d, 1H,  $J$  = 16.7 Hz), 4.51 (d, 1H,  $J$  = 16.7 Hz), 3.59 (s, 3H), 3.47 (d, 1H,  $J$  = 13.6 Hz), 3.46 (d, 1H,  $J$  = 16.0 Hz), 3.23 (d, 1H,  $J$  = 16.0 Hz), 3.09 (d, 1H,  $J$  = 13.6 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 173.5 (s), 150.4 (s), 139.1 (s), 136.0 (s), 130.4 (d, 2C), 128.5 (d, 2C), 128.3 (d, 2C), 127.5 (d), 126.9 (d), 126.7 (d), 126.6 (s), 126.6 (d, 2C), 123.8 (d), 117.9 (d), 107.1 (d), 75.4 (s), 52.1 (q), 49.0 (t), 40.6 (t), 37.7 (t) ppm.

**16a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.37–7.30 (m, 4H), 7.26 (m, 1H), 7.05 (dd, 1H,  $J$  = 7.2 and 1.0 Hz), 6.96 (ddm, 1H,  $J$  = 7.3 and 7.3 Hz), 6.63 (ddd, 1H,  $J$  = 7.3, 7.3, and 1.1 Hz), 6.23 (d, 1H,  $J$  = 7.8 Hz), 4.33 (s, 2H), 3.55 (dd, 1H,  $J$  = 11.5 and 2.7 Hz), 3.48 (dd, 1H,  $J$  = 11.3 and 9.3 Hz), 3.28 (d, 1H,  $J$  = 16.1 Hz), 2.96 (d, 1H,  $J$  = 16.1 Hz), 1.75 (qd, 1H,  $J$  = 14.4 and 7.5 Hz), 1.60 (dd, 1H,  $J$  = 9.5 and 3.0 Hz), 1.49 (qd, 1H,  $J$  = 14.3 and 7.5 Hz), 0.83 (t, 3H,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 152.4 (s), 139.9 (s), 128.9 (d, 2C), 127.5 (s), 127.3 (d), 127.1 (d), 126.5 (d, 2C), 123.9 (d), 117.3 (d), 106.0 (d), 72.0 (s), 66.3 (t), 46.8 (t), 35.5 (t), 27.0 (t), 8.2 (q) ppm.

**16b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.31–7.22 (m, 4H), 7.18 (m, 1H), 6.97 (dd, 1H,  $J$  = 7.0 and 0.9 Hz), 6.89 (ddm, 1H,  $J$  = 7.8 and 7.8 Hz), 6.57 (ddd, 1H,  $J$  = 7.3, 7.3, and 1.0 Hz), 6.15 (d, 1H,  $J$  = 7.8 Hz), 5.61 (m, 1H), 5.04 (dddm, 1H,  $J$  = 16.8, 1.6, 1.6, and 1.6 Hz), 4.97 (dddm, 1H,  $J$  = 10.1, 2.3, 1.0, and 1.0 Hz), 4.33 (d, 1H,  $J$  = 16.6 Hz), 4.25 (d, 1H,  $J$  = 16.5 Hz), 3.54 (dd, 1H,  $J$  = 11.5 and 2.0 Hz), 3.46 (dd, 1H,  $J$  = 11.3 and 8.5 Hz), 3.15 (dm, 1H,  $J$  = 16.1 Hz), 2.95 (d, 1H,  $J$  = 16.1 Hz), 2.41 (dddm, 1H,  $J$  = 14.0, 7.0, 1.3, and 1.3 Hz), 2.20 (dddm, 1H,  $J$  = 14.0, 7.5, 1.0, and 1.0 Hz), 1.56 (dd, 1H,  $J$  = 8.8 and 2.9 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 151.9 (s), 139.6 (s), 132.9 (d), 128.9 (d, 2C), 127.4 (d), 127.3 (s), 127.1 (d), 126.5 (d, 2C), 124.1 (d), 118.7 (t), 117.6 (d), 106.5 (d), 71.4 (s), 66.0 (t), 47.1 (t), 38.6 (t), 35.6 (t) ppm.

**16c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.32–7.04 (m, 10H), 6.98 (dd, 1H,  $J$  = 7.1 and 0.8 Hz), 6.89 (dd, 1H,  $J$  = 7.1 and 7.1 Hz), 6.57 (ddd, 1H,  $J$  = 7.5, 7.5, and 1.1 Hz), 6.17 (d, 1H,  $J$  = 7.8 Hz), 4.45 (d, 1H,  $J$  = 16.8 Hz), 4.31 (d, 1H,  $J$  = 16.6 Hz), 3.66 (dd, 1H,  $J$  = 11.7 and 3.1 Hz), 3.49 (dd, 1H,  $J$  = 11.7 and 8.7 Hz), 2.96 (d, 1H,  $J$  = 16.1 Hz), 2.93 (d, 1H,  $J$  = 13.5 Hz), 2.88 (d, 1H,  $J$  = 15.8 Hz), 2.76 (d, 1H,  $J$  = 13.2 Hz), 1.56 (dd, 1H,  $J$  = 8.7 and 3.2 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 151.3 (s), 139.6 (s), 136.7 (s), 130.5 (d, 2C), 128.9 (d, 2C), 128.3 (d, 2C), 127.4 (d), 127.3 (s), 127.1 (d), 126.6 (d, 3C), 124.3 (d), 117.8 (d), 107.1 (d), 72.8 (s), 64.9 (t), 47.4 (t), 38.7 (t), 34.7 (t) ppm.

**17.**  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_6$ , 100 °C)  $\delta$ : 7.36–7.21 (m, 5H), 5.76 (m, 1H), 5.07 (m, 2H), 3.79–3.64 (m, 3H), 3.58–3.44 (m, 2H), 3.26 (m, 1H), 2.80 (d, 1H,  $J$  = 3.7 Hz), 2.76 (m, 1H), 2.66 (m, 2H), 2.37 (ddddd, 1H,  $J$  = 47.3, 14.6, 7.0, 1.1, and 1.1 Hz), 2.36 (ddddd, 1H,  $J$  = 21.6, 10.5, 6.8, 1.4, and 1.4 Hz), 1.42 (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ ) mixture of rotamers  $\delta$ : 154.1 (154.2) (s), 138.9 (s), 131.9 (d), 128.6 (d, 2C), 128.1 (d, 2C), 127.0 (d), 118.9 (t), 98.1 (97.8) (ds,  $J$  = 180 Hz), 78.8 (s), 62.3 (t), 62.0 (61.9) (dt,  $J$  = 28 Hz), 55.5 (55.3) (t), 53.7 (52.9) (dt,  $J$  = 30 Hz), 48.8 (49.1) (t), 40.6 (40.9) (dt,  $J$  = 22 Hz), 27.9 (q, 3C) ppm.

**18.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.29–7.21 (m, 4H), 7.17 (m, 1H), 3.90 (dd, 1H,  $J$  = 18.3 and 13.3 Hz), 3.78 (ddd, 1H,  $J$  = 12.4, 4.9, and 3.8 Hz), 3.64 (d, 1H,  $J$  = 13.3 Hz), 3.58 (d, 1H,  $J$  = 13.2 Hz), 3.56 (dd, 1H,  $J$  = 25.8 and 13.2 Hz), 3.51 (dddd, 1H,  $J$  = 12.3, 7.6, 4.0, and 0.6 Hz), 2.90 (dd, 1H,  $J$  = 17.3 and 13.7 Hz), 2.74–2.55 (m, 3H), 1.13 (d, 3H,  $J$  = 21.3 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 139.1 (s), 129.0 (d), 128.3 (d), 127.3 (d), 98.0 (ds,  $J$  = 169 Hz), 78.7 (dt,  $J$  = 28 Hz), 74.4 (t), 64.9 (dt,  $J$  = 30 Hz), 63.7 (t), 59.5 (t), 23.1 (dq,  $J$  = 25 Hz) ppm.

**19a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.39–7.23 (m, 5H), 7.10–7.02 (m, 2H), 6.68 (dd, 1H,  $J$  = 7.3 and 7.3 Hz), 6.59 (d, 1H,  $J$  = 8.3 Hz), 4.60 (d, 1H,  $J$  = 16.9 Hz), 4.53 (d, 1H,  $J$  = 16.9 Hz), 3.48–3.31 (m, 2H), 3.15–2.88 (m, 2H), 1.81 (dq, 2H,  $J$  = 21.2 and 7.6 Hz), 1.06 (t, 3H,  $J$  = 7.6 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 144.2 (s), 138.3 (s), 129.6 (d), 128.7 (d, 2C), 127.6 (d), 126.9 (d), 126.6 (d, 2C), 119.0 (ds,  $J$  = 3 Hz), 116.8 (d), 111.0 (d), 92.0 (ds,  $J$  = 174 Hz), 56.3 (dt,  $J$  = 25 Hz), 54.8 (t), 37.3 (dt,  $J$  = 24 Hz), 30.4 (dt,  $J$  = 23 Hz), 7.1 (dq,  $J$  = 4 Hz) ppm.

**19b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.36–7.19 (m, 5H), 7.05–6.97 (m, 2H), 6.64 (ddd, 1H,  $J$  = 7.4, 7.4, and 0.8 Hz), 6.55 (d, 1H,  $J$  = 8.0 Hz), 5.90 (m, 1H), 5.20–5.09 (m, 2H), 4.51 (s, 2H), 3.43–3.28 (m, 2H), 3.10–2.87 (m, 2H), 2.51 (ddm, 2H,  $J$  = 20.8 and 7.3 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 144.1 (s), 138.3 (s), 131.6 (dd,  $J$  = 5 Hz), 129.7 (d), 128.7 (d, 2C), 127.6 (d), 127.0 (d), 126.6 (d, 2C), 119.4 (t), 118.7 (ds,  $J$  = 4 Hz), 116.9 (d), 111.1 (d), 91.4 (ds,  $J$  = 176 Hz), 56.3 (dt,  $J$  = 25 Hz), 54.9 (t), 42.1 (dt,  $J$  = 22 Hz), 37.7 (dt,  $J$  = 24 Hz) ppm.

**19c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.35–7.13 (m, 10H), 6.99 (dd, 1H,  $J$  = 7.7 and 7.7 Hz), 6.94 (d, 1H,  $J$  = 7.5 Hz), 6.62 (ddm, 1H,  $J$  = 7.5 and 7.5 Hz), 6.55 (d, 1H,  $J$  = 8.3 Hz), 4.50 (d, 1H,  $J$  = 17.1 Hz), 4.43 (d, 1H,  $J$  = 17.0 Hz), 3.40–3.25 (m, 2H), 3.01 (d, 2H,  $J$  = 23.3 Hz), 3.02–2.85 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 144.1 (s), 138.3 (s), 135.6 (s), 130.6 (d, 2C), 129.8 (d), 128.7 (d, 2C), 128.4 (d, 2C), 127.7 (d), 127.1 (d), 127.0 (d), 126.7 (d, 2C), 118.8 (ds,  $J$  = 5 Hz), 117.0 (d), 111.3 (d), 91.6 (ds,  $J$  = 177 Hz), 56.5 (dt,  $J$  = 25 Hz), 55.0 (t), 43.6 (dt,  $J$  = 22 Hz), 37.8 (dt,  $J$  = 24 Hz) ppm.

**23a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.29–7.04 (m, 10H), 3.71 (d, 1H,  $J$  = 12.7 Hz), 3.54 (d, 1H,  $J$  = 12.8 Hz), 3.21 (d, 1H,  $J$  = 13.0 Hz), 3.19–3.06 (m, 4H), 2.77 (d, 1H,  $J$  = 13.0 Hz), 2.06 (m, 1H), 1.87 (ddd, 1H,  $J$  = 11.1, 8.2, and 3.1 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 138.4 (s), 136.9 (s), 130.0 (d, 2C), 128.5 (d, 2C), 128.4 (d, 2C), 128.3 (d, 2C), 127.2 (d), 126.5 (d), 70.1 (t), 64.2 (s), 53.9 (t), 48.8 (t), 37.0 (t), 22.7 (t) ppm.

**23b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.35–7.17 (m, 8H), 7.16–7.10 (m, 2H), 3.97 (d, 1H,  $J$  = 12.9 Hz), 3.63 (d, 1H,  $J$  = 10.2 Hz), 3.44 (d, 1H,  $J$  = 12.8 Hz), 3.40 (d, 1H,  $J$  = 10.4 Hz), 3.28 (s br, 1H), 2.97 (m, 1H), 2.73 (s, 2H), 2.56 (m, 1H), 1.90–1.56 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 139.7 (s), 137.9 (s), 130.3 (d, 2C), 128.6 (d, 2C), 128.5 (d, 2C), 128.3 (d, 2C), 127.1 (d), 126.4 (d), 67.2 (s), 63.2 (t), 52.1 (t), 50.1 (t), 37.1 (t), 30.2 (t), 21.4 (t) ppm.

**23c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.42–7.22 (m, 8H), 7.21–7.16 (m, 2H), 4.19 (d, 1H,  $J$  = 13.4 Hz), 3.93 (d, 1H,  $J$  = 10.6 Hz), 3.40 (s br, 1H), 3.17 (d, 1H,  $J$  = 13.6 Hz), 3.21 (d, 1H,  $J$  = 13.4 Hz), 3.13 (d, 1H,  $J$  = 10.4 Hz), 2.82 (dm, 1H,  $J$  = 12.1 Hz), 2.73 (d, 1H,  $J$  = 13.2 Hz), 2.52 (ddd, 1H,  $J$  = 12.2, 12.2, and 2.3 Hz), 1.81–1.60 (m, 4H), 1.58–1.38 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 139.5 (s), 137.7 (s),

130.1 (d, 2C), 128.6 (d, 2C), 128.5 (d, 2C), 128.3 (d, 2C), 127.0 (d), 126.3 (d), 65.3 (t), 60.5 (s), 52.9 (t), 46.3 (t), 32.9 (t), 29.0 (t), 25.9 (t), 20.6 (t) ppm.

**23d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.48–7.22 (m, 10H), 4.08 (d, 1H,  $J$  = 13.4 Hz), 3.87 (d, 1H,  $J$  = 10.5 Hz), 3.77 (d, 1H,  $J$  = 13.5 Hz), 3.55 (d, 1H,  $J$  = 10.8 Hz), 3.32 (s br, 1H), 2.96 (d, 1H,  $J$  = 13.0 Hz), 2.89 (d, 1H,  $J$  = 12.9 Hz), 2.84 (m, 1H), 2.72 (dd, 1H,  $J$  = 14.9 and 1.9 Hz), 1.88 (m, 1H), 1.78–1.50 (m, 5H), 1.40–1.24 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.7 (s), 138.2 (s), 130.5 (d, 2C), 128.7 (d, 2C), 128.4 (d, 2C), 128.1 (d, 2C), 127.0 (d), 126.2 (d), 67.7 (t), 63.0 (s), 54.3 (t), 47.1 (t), 40.2 (t), 38.1 (t), 31.3 (t), 29.8 (t), 23.2 (t) ppm.

**24a.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.28–7.11 (m, 10H), 3.54 (s, 2H), 2.92 (d, 2H,  $J$  = 24.8 Hz), 2.72–2.64 (m, 1H), 2.65 (dd, 2H,  $J$  = 25.0 and 1.4 Hz), 2.50 (m, 1H), 1.91 (dddm, 2H,  $J$  = 26.1, 7.0, and 7.0 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 138.7 (s), 136.6 (s), 130.2 (d, 2C), 128.8 (d, 2C), 128.3 (d, 2C), 128.2 (d, 2C), 127.1 (d), 126.7 (d), 103.4 (ds,  $J$  = 178 Hz), 64.0 (dt,  $J$  = 26 Hz), 60.3 (t), 52.9 (t), 44.3 (dt,  $J$  = 25 Hz), 37.0 (dt,  $J$  = 23 Hz) ppm.

**24b.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.35–7.15 (m, 10H), 3.54 (d, 1H,  $J$  = 13.0 Hz), 3.48 (d, 1H,  $J$  = 13.3 Hz), 3.01 (dd, 1H,  $J$  = 29.2 and 14.2 Hz), 2.94 (dd, 1H,  $J$  = 29.7 and 14.2 Hz), 2.51–2.32 (m, 4H), 1.80–1.48 (m, 4H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 138.0 (s), 136.2 (s), 130.5 (d, 2C), 129.1 (d, 2C), 128.2 (d, 2C), 128.1 (d, 2C), 127.1 (d), 126.5 (d), 93.8 (ds,  $J$  = 175 Hz), 62.8 (t), 60.5 (dt,  $J$  = 24 Hz), 53.0 (t), 43.5 (dt,  $J$  = 22 Hz), 33.4 (dt,  $J$  = 21 Hz), 22.2 (dt,  $J$  = 7 Hz) ppm.

**24c.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.45–7.18 (m, 10H), 3.73 (s, 2H), 2.96 (dd, 1H,  $J$  = 32.0 and 14.4 Hz), 2.91 (dd, 1H,  $J$  = 26.1 and 14.2 Hz), 2.91–2.82 (m, 2H), 2.78 (m, 1H), 2.58 (ddd, 1H,  $J$  = 12.6, 8.4, and 4.3 Hz), 1.91 (m, 1H), 1.86 (m, 1H), 1.82–1.70 (m, 2H), 1.69–1.50 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 139.8 (s), 136.7 (s), 130.7 (d, 2C), 128.9 (d, 2C), 128.3 (d, 2C), 128.0 (d, 2C), 127.1 (d), 126.4 (d), 99.2 (ds,  $J$  = 172 Hz), 64.0 (dt,  $J$  = 30 Hz), 63.9 (t), 57.8 (t), 45.2 (dt,  $J$  = 22 Hz), 38.0 (dt,  $J$  = 23 Hz), 30.9 (t), 21.4 (dt,  $J$  = 5 Hz) ppm.

**24d.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.38–7.16 (m, 10H), 3.78 (d, 1H,  $J$  = 13.5 Hz), 3.65 (d, 1H,  $J$  = 13.5 Hz), 2.90 (dd, 1H,  $J$  = 25.5 and 14.4 Hz), 2.84 (dd, 1H,  $J$  = 19.4 and 14.4 Hz), 2.83 (dd, 1H,  $J$  = 14.8 and 14.8 Hz), 2.69 (dd, 1H,  $J$  = 26.8 and 15.3 Hz), 2.53–2.35 (m, 2H), 2.03 (m, 1H), 1.92–1.41 (m, 7H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 140.3 (s), 136.7 (s), 130.7 (d), 130.6 (d), 128.8 (d, 2C), 128.2 (d, 2C), 128.1 (d, 2C), 126.9 (d), 126.5 (d), 99.9 (ds,  $J$  = 173 Hz), 64.1 (t), 59.6 (dt,  $J$  = 25 Hz), 54.6 (t), 43.7 (dt,  $J$  = 21 Hz), 34.0 (dt,  $J$  = 23 Hz), 27.1 (t), 27.0 (t), 22.7 (dt,  $J$  = 9 Hz) ppm.

**26.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) mixture of rotamers  $\delta$ : 7.35–7.17 (m, 5H), 5.92–5.16 (m, 1H), 3.73–3.63 (m, 3H), 3.29–2.96 (m, 3H), 1.96–1.21 (m, 12H).  $^{13}\text{C}$  NMR (100 MHz,  $\text{DMSO}-d_6$ , 122 °C)  $\delta$ : 170.1 (s), 142.2 (s), 134.1 (s), 128.9 (d, 2C), 128.5 (d, 2C), 128.3 (d), 66.5 (s), 62.9 (d), 51.8 (q), 42.6 (t), 28.1 (q, 3C), 23.5 (t), 22.0 (t) ppm.

**27.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) mixture of rotamers  $\delta$ : 7.42–7.22 (m, 5H), 3.78 (3.75) (s, 3H), 3.77–3.58 (m, 2H), 2.68–2.54 (m, 1H), 2.34–2.24 (m, 1H), 1.97–1.86 (m, 1H), 1.75–1.61 (m, 1H), 1.26 (1.51) (s, 9H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) mixture of rotamers  $\delta$ : 173.3 (s), 153.9 (154.2) (s), 140.3 (139.6) (s), 127.4 (127.6) (d, 2C), 127.1 (127.2) (d, 2C), 126.9 (d), 80.3 (80.0) (s), 71.5 (71.7) (s), 52.4 (52.5) (q),

47.8 (47.9) (t), 43.4 (41.8) (t), 28.0 (28.4) (q, 3C), 22.6 (23.5) (t) ppm.

**28.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.42–7.19 (m, 10H), 4.06 (d, 1H,  $J$  = 10.3 Hz), 3.88 (d, 1H,  $J$  = 10.3 Hz), 3.57 (d, 1H,  $J$  = 13.5 Hz), 3.12 (d, 1H,  $J$  = 13.5 Hz), 3.06 (ddd, 1H,  $J$  = 9.3, 7.9, and 3.6 Hz), 2.86 (ddd, 1H,  $J$  = 9.3, 8.1, and 8.1 Hz), 2.71 (s br, 1H), 2.41 (ddd, 1H,  $J$  = 13.3, 10.1, and 5.5 Hz), 2.13 (ddd, 1H,  $J$  = 13.2, 9.0, and 7.0 Hz), 2.01–1.82 (m, 2H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 143.6 (s), 140.0 (s), 128.5 (d, 2C), 128.4 (d, 2C), 128.3 (d, 2C), 127.0 (d), 126.9 (d), 126.6 (d, 2C), 69.3 (s), 63.4 (t), 53.8 (t), 52.5 (t), 37.3 (t), 22.8 (t) ppm.

**29.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.45–7.40 (m, 2H), 7.37–7.21 (m, 8H), 3.65 (d, 1H,  $J$  = 13.3 Hz), 3.59 (d, 1H,  $J$  = 13.3 Hz), 2.99 (ddm, 1H,  $J$  = 9.8 and 9.8 Hz), 2.89 (dm, 1H,  $J$  = 11.2 Hz), 2.41 (dd, 1H,  $J$  = 30.4 and 12.5 Hz), 2.20 (ddm, 1H,  $J$  = 10.8 and 10.8 Hz), 2.12–2.00 (m, 2H), 1.84 (m, 1H), 1.64 (m, 1H) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 143.2 (ds,  $J$  = 22 Hz), 137.5 (s), 129.2 (d, 2C), 128.3 (d, 2C), 128.2 (d, 2C), 127.7 (d), 127.1 (d), 124.5 (dd,  $J$  = 9 Hz, 2C), 94.1 (ds,  $J$  = 177 Hz), 62.8 (t), 62.0 (dt,  $J$  = 22 Hz), 52.6 (t), 35.0 (dt,  $J$  = 23 Hz), 21.5 (dt,  $J$  = 2 Hz) ppm.

**31.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 3.04 (dd, 1H,  $J$  = 15.1 and 15.1 Hz), 2.93 (ddd, 1H,  $J$  = 13.4, 7.8, and 5.4 Hz), 2.82 (m, 1H), 2.79 (dd, 1H,  $J$  = 25.1 and 14.8 Hz), 1.99 (s br, 1H), 1.95 (m, 1H), 1.77–1.52 (m, 6H), 1.47 (m, 1H), 0.92 (t, 3H,  $J$  = 7.5 Hz) ppm.  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 100.2 (ds,  $J$  = 169 Hz), 57.2 (dt,  $J$  = 27 Hz), 50.6 (t), 37.3 (dt,  $J$  = 24 Hz), 32.1 (dt,  $J$  = 24 Hz), 31.8 (t), 21.4 (dt,  $J$  = 6 Hz), 7.4 (dq,  $J$  = 6 Hz) ppm.

## ■ ASSOCIATED CONTENT

### S Supporting Information

Revised file containing experimental data and spectra. This material is available free of charge via the Internet at <http://pubs.acs.org>.