
Supplementary material

Influence of MW irradiation on the reaction between (2*R*,7*R*,11*S*,16*S*)-1,8,10,17-tetraazapentacyclo[8.8.1.1.^{8,17}0.^{2,7}0.^{11,16}]icosane and *p*-substituted phenols

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Characterization data for synthesized compounds

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(*rac*)-2,2'-((hexahydro-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(methylene))bis(4-methylphenol) **1a**. IR-ATR: 3051, 3050, 3016, 2930, 2925, 2859, 2857, 2826, 1677, 1621, 1560, 1501, 1493, 1446, 1398, 1361, 1336, 1292, 1252, 1251, 1209, 1177, 1142, 1126, 1067, 1039, 933, 856, 836, 818, 815, 771, 766, 743, 712 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 10.30 (*bs*, 2H, ArOH), 10.05 (*bs*, 2H, ArOH), 6.94 (*d*, 2H, ³J_{H,H} = 8.4 Hz, H-5, H-5'), 6.73 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-6, H-6'), 6.73 (*s*, 2H, H-3, H-3'), 6.70 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-5, H-5'), 6.69 (*d*, 2H, ³J_{H,H} = 8.4 Hz, H-6, H-6'), 6.51 (*d*, 2H, ⁴J_{H,H} = 2.0 Hz, H-3, H-3'), 4.17 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 4.16 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.71 (*s*, 2H, ArOCH₃), 3.53 (*s*, 2H, H-2), 3.52 (*s*, 2H, H-2), 3.40 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.37 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.32 (*m*, 2H, H-3a, H-7a), 2.31 (*m*, 2H, H-3a, H-7a), 2.20 (*s*, 6H, ArCH₃), 2.07 (*m*, 2H, Heq-4, Heq-7), 2.05 (*m*, 2H, Heq-4, Heq-7), 1.85 (*m*, 2H, Heq-5, Heq-6), 1.84 (*m*, 2H, Heq-5, Heq-6), 1.30 (*m*, 4H, Hax-4, Hax-5, Hax-6, Hax-7), 1.28 (*m*, 4H, Hax-4, Hax-5, Hax-6, Hax-7); ¹³C NMR (100 MHz, CDCl₃) δ 155.0 (C1, C1'), 129.3 (C5, C5'), 129.0 (C3, C3'), 128.5 (C4, C4'), 121.2 (C2, C2'), 115.7 (C6, C6'), 75.8 (C2), 69.1 (C3a, C7a), 56.3 (NCH₂Ar), 29.0 (C4, C7), 24.0 (C5, C6), 20.2 (ArCH₃).

(*rac*)-2,2'-((hexahydro-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(methylene))bis(4-methoxyphenol) **1b**. IR-ATR: 3051, 3016, 2930, 2859, 2826, 1493, 1361, 1336, 1292, 1252, 1209, 1142, 1039, 933, 856, 836, 818, 766, 743, 712 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 10.05 (*bs*, 2H, ArOH), 6.73 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-6, H-6'), 6.70 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-5, H-5'), 6.51 (*d*, 2H, ⁴J_{H,H} = 2.0 Hz, H-3, H-3'), 4.16 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.71 (*s*, 2H, ArOCH₃), 3.53 (*s*, 2H, H-2), 3.40 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.32 (*m*, 2H, H-3a, H-7a), 2.05 (*m*, 2H, Heq-4, Heq-7), 1.84 (*m*, 2H, Heq-5, Heq-6), 1.28 (*m*, 4H, Hax-4, Hax-5, Hax-6, Hax-7); ¹³C NMR (100 MHz, CDCl₃) δ 152.9 (C1, C1'), 151.5 (C4, C4'), 122.5 (C2, C2'), 116.8 (C6, C6'), 114.2 (C5, C5'), 114.2 (C3, C3'), 76.1 (C2), 69.4 (C3a, C7a), 56.7 (ArOCH₃), 56.0 (NCH₂Ar), 29.3 (C4, C7), 24.3 (C5, C6).

(*rac*)-2,2'-((hexahydro-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(methylene))bis(4-chlorophenol) **1c**. IR-ATR: 3060, 2940, 2856, 1655, 1481, 1393, 1370, 1351, 1277, 1252, 1182, 1109, 1068, 1039, 994, 899, 820, 769, 679, 643 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 10.44 (*bs*, 1H, HO-Ar), 7.10 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-5, H-5'), 6.92 (*s*, 2H, H-3, H-3'), 6.74 (*d*, 2H, ³J_{H,H} = 8.8 Hz, H-6, H-6'), 4.14 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.51 (*s*, 2H, H-2), 3.42 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.34 (*dd*, 2H, ³J_{H,H} = 8.5 Hz, ³J_{H,H} = 2.8 Hz, H-3a, H-7a), 2.04 (*m*, 2H, Heq-4, Heq-7), 1.85 (*m*, 2H, Heq-5, Heq-6), 1.27 (*m*, 4H, Hax-4, Hax-5, Hax-6, Hax-7); ¹³C NMR (100 MHz, CDCl₃) δ 156.4 (C1, C1'), 129.2 (C5, C5'), 128.1 (C3, C3'), 124.3 (C4, C4'), 123.2 (C2, C2'), 117.9 (C6, C6'), 76.0 (C2), 69.4 (C3a, C7a), 56.2 (NCH₂Ar), 29.2 (C4, C7), 24.3 (C5, C6).

Dimethyl (*rac*)-3,3'-((hexahydro-1H-benzo[d]imidazole-1,3(2H)-diyl)bis(methylene))bis(4-hydroxybenzoate) **1d**. IR-ATR: 3070, 3004, 2925, 2855, 1713, 1611, 1591, 1494, 1460, 1436, 1410, 1291, 1256, 1223, 1189, 1126, 1112, 1069, 1040, 841, 826, 806, 772 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 10.79 (*bs*, 2H, ArOH), 7.86 (*dd*, 2H, ⁴J_{H,H} = 1.6 Hz, ³J_{H,H} = 8.4 Hz, H-5, H-5'), 7.68 (*s*, 2H, H-3, H-3'), 6.83 (*d*, 2H, ³J_{H,H} = 8.4 Hz, H-6, H-6'), 4.20 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.84 (*s*, 6H, CH₃-OOC-Ar), 3.54 (*d*, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.53 (*s*, CH₃-OOC-Ar), 2.38 (*dd*, 2H, ³J_{H,H} = 8.5 Hz, ³J_{H,H} = 2.8 Hz, H-3a, H-7a), 2.06 (*m*, 2H, Heq-4, Heq-7), 1.85 (*m*, 2H, Heq-5, Heq-6), 1.30 (*m*, 4H, Hax-4, Hax-5, Hax-6, Hax-7); ¹³C NMR (100 MHz, CDCl₃) δ 167.1 (Ar(C=O)O-Me), 162.2 (C1, C1'), 131.6 (C5, C5'), 130.4 (C3, C3'), 121.7 (C4, C4'), 121.4 (C2, C2'), 116.6 (C6, C6'), 75.9 (C2), 69.4 (C3a, C7a), 56.4 (NCH₂Ar), 52.18 (CH₃-OOC-Ar), 29.2 (C4, C7), 24.2 (C5, C6).

2,2'-((((2-hydroxy-5-methyl-1,3-phenylene)bis(methylene))bis(octahydro-1H-benzo[d]imidazole-3,1-diyl))bis(methylene))bis(4-methylphenol) **2a**. ^1H NMR (400 MHz, CDCl_3) For the isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3aS',7aS'): δ 6.96 (*dd*, 2H, $^3J_{\text{H,H}} = 8.0, 2.1$ Hz, H-5, H-5'), 6.82 (*s*, 2H, H-4, H-6), 6.75 (*s*, 2H, H-3, H-3'), 6.71 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-6, H-6'), 4.14 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.97 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.60 (*s*, 4H, NCH_2N), 3.51 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.41 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 2.37 (*m*, 4H, H-3a, H-7a, H-3a', H-7a'), 2.22 (*s*, 6H, $\text{CH}_3\text{-Ar}$), 2.20 (*s*, 3H, $\text{CH}_3\text{-Ar}$), 2.05 (*m*, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.83 (*m*, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.29 (*m*, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). For *meso*-(3aR,7aR,3a'S,7a'S): δ 6.96 (*dd*, 2H, $^3J_{\text{H,H}} = 8.0, 2.1$ Hz, H-5, H-5'), 6.85 (*s*, 2H, H-4, H-6), 6.77 (*s*, 2H, H-3, H-3'), 6.71 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-6, H-6'), 4.16 (*d*, 2H, $^3J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 4.07 (*d*, 2H, $^3J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.59 (*s*, 4H, NCH_2N), 3.52 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.47 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 2.37 (*m*, 4H, H-3a, H-7a, H-3a', H-7a'), 2.23 (*s*, 3H, $\text{CH}_3\text{-Ar}$), 2.22 (*s*, 6H, $\text{CH}_3\text{-Ar}$), 2.05 (*m*, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.83 (*m*, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.29 (*m*, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). ^{13}C NMR (100 MHz, CDCl_3) For the isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3aS',7aS'): δ 155.5 (C2), 155.5 (C1, C1'), 129.5 (C5, C5'), 128.9 (C3, C3'), 128.9 (C4, C6), 128.6 (C4, C4'), 128.5 (C5), 121.8 (C2, C2'), 121.8 (C1, C3), 116.1 (C6, C6'), 76.1 (C2, C2'), 69.6 (C7a, C7a'), 69.1 (C3a, C3a'), 56.7 (ArCH_2N), 56.7 (ArCH_2N), 29.4 (C4, C4'), 29.4 (C7, C7'), 24.5 (C5, C5'), 24.5 (C6, C6'), 20.8 ($\text{CH}_3\text{-Ar}$), 20.8 ($\text{CH}_3\text{-Ar}$). For *meso*-(3aR,7aR,3a'S,7a'S): δ 155.5 (C2), 155.5 (C1, C1'), 129.5 (C5, C5'), 128.9 (C3, C3'), 128.9 (C4, C6), 128.6 (C4, C4'), 128.5 (C5), 121.8 (C2, C2'), 121.8 (C1, C3), 116.1 (C6, C6'), 76.4 (C2, C2'), 69.7 (C7a, C7a'), 69.2 (C3a, C3a'), 56.7 (ArCH_2N), 56.7 (ArCH_2N), 29.4 (C4, C4'), 29.4 (C7, C7'), 24.5 (C5, C5'), 24.5 (C6, C6'), 20.8 ($\text{CH}_3\text{-Ar}$), 20.8 ($\text{CH}_3\text{-Ar}$). HRMS (ESI+) m/z (%): $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{39}\text{H}_{53}\text{N}_4\text{O}_3$: 625.4112, found: 625.4086 (100.00%); $[\text{M}+\text{K}]^+$ calculated for $\text{C}_{39}\text{H}_{52}\text{N}_4\text{O}_3\text{K}$: 663.3671, found: 663.3869 (39.71%).

2,2'-((((2-hydroxy-5-methoxy-1,3-phenylene)bis(methylene))bis(octahydro-1H-benzo[d]imidazole-3,1-diyl))bis(methylene))bis(4-methoxyphenol) **2b**. ^1H NMR (400 MHz, CDCl_3) For the isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3aS',7aS'): δ 6.72 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-6, H-6'), 6.70 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-5, H-5'), 6.59 (*s*, 2H, H-4, H-6), 6.51 (*d*, 2H, $^4J_{\text{H,H}} = 2.0$ Hz, H-3, H-3'), 4.10 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.94 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.70 (*s*, 6H, $\text{CH}_3\text{O-Ar}$), 3.69 (*s*, 3H, $\text{CH}_3\text{O-Ar}$), 3.55 (*s*, 4H, NCH_2N), 3.48 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.40 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 2.33 (*m*, 4H, H-3a, H-7a, H-3a', H-7a'), 2.02 (*m*, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.81 (*m*, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.29 (*m*, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). For *meso*-(3aR,7aR,3a'S,7a'S): δ 6.72 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-6, H-6'), 6.70 (*d*, 2H, $^3J_{\text{H,H}} = 8.0$ Hz, H-5, H-5'), 6.61 (*s*, 2H, H-4, H-6), 6.51 (*d*, 2H, $^4J_{\text{H,H}} = 2.0$ Hz, H-3, H-3'), 4.12 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 4.03 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.70 (*s*, 6H, $\text{CH}_3\text{O-Ar}$), 3.69 (*s*, 3H, $\text{CH}_3\text{O-Ar}$), 3.55 (*s*, 4H, NCH_2N), 3.51 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 3.44 (*d*, 2H, $^2J_{\text{H,H}} = 14.0$ Hz, ArCH_2N), 2.33 (*m*, 4H, H-3a, H-7a, H-3a', H-7a'), 2.02 (*m*, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.81 (*m*, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.29 (*m*, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). ^{13}C NMR (100 MHz, CDCl_3) For the isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3aS',7aS'): δ 152.8 (C1, C1'), 151.7 (C4, C4'), 149.5 (C5), 149.4 (C2), 122.8 (C2, C2'), 122.6 (C1, C3), 116.8 (C6, C6'), 114.2 (C5, C5'), 114.2 (C4, C6), 114.0 (C3, C3'), 76.3 (C2, C2'), 69.6 (C7a, C7a'), 69.1 (C3a, C3a'), 56.8 (ArCH_2N), 56.1 ($\text{CH}_3\text{O-Ar}$), 56.0 ($\text{CH}_3\text{O-Ar}$), 54.1 (ArCH_2N), 29.4 (C7, C7'), 29.3 (C4, C4'), 24.5 (C6, C6'), 24.3 (C5, C5'), 20.8 ($\text{CH}_3\text{-Ar}$), 20.8 ($\text{CH}_3\text{-Ar}$). For *meso*-(3aR,7aR,3aS',7aS'): δ

152.8 (C1, C1'), 151.7 (C4, C4'), 149.5 (C5), 149.4 (C2), 122.8 (C2, C2'), 122.6 (C1, C3), 116.8 (C6, C6'), 114.2 (C5, C5'), 114.2 (C4, C6), 114.0 (C3, C3'), 76.4 (C2, C2'), 69.7 (C7a, C7a'), 69.2 (C3a, C3a'), 56.9 (ArCH₂N), 56.1 (CH₃O-Ar), 56.0 (CH₃O-Ar), 54.3 (ArCH₂N), 29.5 (C7, C7'), 29.4 (C4, C4'), 24.5 (C6, C6'), 24.4 (C5, C5').

2,2'-((((5-chloro-2-hydroxy-1,3-phenylene)bis(methylene))bis(octahydro-1H-benzo[d]imidazole-3,1-diyl))bis(methylene))bis(4-chlorophenol) 2c. ¹H NMR (400 MHz, CDCl₃) For isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3a'S,7a'S): δ 7.09 (dd, 2H, ⁴J_{H,H} = 2.2 Hz, ³J_{H,H} = 8.0 Hz, H-5, H-5'), 7.00 (s, 2H, H-4, H-6), 6.92 (s, 2H, H-3, H-3'), 6.72 (d, 2H, ³J_{H,H} = 8.6 Hz, H-6, H-6'), 4.10 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.92 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.51 (s, 4H, NCH₂N), 3.45 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.40 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.33 (m, 4H, H-3a, H-7a, H-3a', H-7a'), 2.05 (m, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.81 (m, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.27 (m, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). For *meso*-(3aR,7aR,3a'S,7a'S): δ 7.09 (dd, 2H, ⁴J_{H,H} = 2.2 Hz, ³J_{H,H} = 8.0 Hz, H-5, H-5'), 7.02 (s, 2H, H-4, H-6), 6.92 (s, 2H, H-3, H-3'), 6.72 (d, 2H, ³J_{H,H} = 8.6 Hz, H-6, H-6'), 4.12 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 4.00 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.52 (s, 4H, NCH₂N), 3.48 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.44 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.33 (m, 4H, H-3a, H-7a, H-3a', H-7a'), 2.05 (m, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.81 (m, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.27 (m, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). ¹³C NMR (100 MHz, CDCl₃) For isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3a'S,7a'S): δ 156.6 (C1, C1'), 154.2 (C2), 129.0 (C5, C5'), 128.2 (C4, C6), 128.0 (C3, C3'), 125.1 (C1, C3), 124.0 (C4, C4'), 123.7 (C5), 123.5 (C2, C2'), 117.8 (C6, C6'), 76.3 (C2, C2'), 69.6 (C7a, C7a'), 69.2 (C3a, C3a'), 56.2 (ArCH₂N), 53.6 (ArCH₂N), 29.3 (C7, C7'), 29.2 (C4, C4'), 24.4 (C6, C6'), 24.2 (C5, C5'). For *meso*-(3aR,7aR,3a'S,7a'S): 156.6 (C1, C1'), 154.2 (C2), 129.0 (C5, C5'), 128.2 (C4, C6), 128.1 (C3, C3'), 125.1 (C1, C3), 124.1 (C4, C4'), 123.7 (C5), 123.5 (C2, C2'), 117.8 (C6, C6'), 76.4 (C2, C2'), 69.6 (C7a, C7a'), 69.3 (C3a, C3a'), 56.3 (ArCH₂N), 53.8 (ArCH₂N), 29.4 (C7, C7'), 29.2 (C4, C4'), 24.4 (C6, C6'), 24.3 (C5, C5'). HRMS (ESI+) m/z (%) Calcd. for C₃₆H₄₄Cl₃N₄O₃ [M+H]⁺: 685.2474, found 685.2403 (97.22%).

Dimethyl 3,3'-((((2-hydroxy-5-(methoxycarbonyl)-1,3-phenylene)bis(methylene))bis(octahydro-1H-benzo[d]imidazole-3,1-diyl))bis(methylene))bis(4-hydroxybenzoate) 2d. ¹H NMR (400 MHz, CDCl₃) For isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3a'S,7a'S): δ 7.87 (dd, 2H, ⁴J_{H,H} = 2.1 Hz, ³J_{H,H} = 8.4 Hz, H-6, H-6'), 7.75 (s, 2H, H-4, H-6), 7.68 (s, 2H, H-2, H-2'), 6.82 (d, 2H, ³J_{H,H} = 8.4 Hz, H-5, H-5'), 4.19 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 4.14 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 4.00 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.85 (s, 9H, CH₃-OOC-Ar), 3.58 (s, 4H, NCH₂N), 3.50 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.43 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.39 (m, 4H, H-3a, H-7a, H-3a', H-7a'), 2.06 (m, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.83 (m, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.27 (m, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). For *meso*-(3aR,7aR,3a'S,7a'S): δ 7.87 (dd, 2H, ⁴J_{H,H} = 2.1 Hz, ³J_{H,H} = 8.4 Hz, H-6, H-6'), 7.75 (s, 2H, H-4, H-6), 7.68 (s, 2H, H-2, H-2'), 6.82 (d, 2H, ³J_{H,H} = 8.4 Hz, H-5, H-5'), 4.19 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 4.11 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.85 (s, 9H, CH₃-OOC-Ar), 3.58 (s, 4H, NCH₂N), 3.54 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 3.47 (d, 2H, ²J_{H,H} = 14.0 Hz, ArCH₂N), 2.39 (m, 4H, H-3a, H-7a, H-3a', H-7a'), 2.06 (m, 4H, Heq-4, Heq-7, Heq-4', Heq-7'), 1.83 (m, 4H, Heq-5, Heq-6, Heq-5', Heq-6'), 1.27 (m, 8H, Hax-4, Hax-5, Hax-6, Hax-7, Hax-4', Hax-5', Hax-6', Hax-7'). ¹³C NMR (100 MHz, CDCl₃) For isomer (3aR,7aR,3a'R,7a'R) and its enantiomer (3aS,7aS,3a'S,7a'S): 166.8 (Ar(C=O)O-Me), 166.7 (Ar(C=O)O-Me), 162.2 (C2), 161.9 (C4, C4'), 131.2 (C6, C6'), 131.2 (C4, C6), 131.0 (C2, C2'), 130.0 (C5), 129.9 (C1, C1'), 129.9 (C1, C1'), 121.3 (C1, C3), 121.0 (C3, C3'), 116.1 (C5, C5'), 75.6 (C2, C2'), 69.2 (C7a, C7a'), 69.0 (C3a, C3a'), 56.1 (ArCH₂N), 53.8

(ArCH₂N), 52.1 (CH₃-OOC-Ar), 29.0 (C7, C7'), 28.9 (C4, C4'), 24.1 (C6, C6'), 24.1 (C5, C5'). For *meso*-(3a*R*,7a*R*,3a'*S*,7a'*S*): 166.8 (Ar(C=O)O-Me), 166.7 (Ar(C=O)O-Me), 162.2 (C2), 161.9 (C4, C4'), 131.2 (C6, C6'), 131.2 (C4, C6), 131.0 (C2, C2'), 130.0 (C5), 129.9 (C1, C1'), 129.9 (C1, C1'), 121.3 (C1, C3), 121.1 (C3, C3'), 116.1 (C5, C5'), 75.7 (C2, C2'), 69.2 (C7a, C7a'), 69.0 (C3a, C3a'), 56.1 (ArCH₂N), 53.8 (ArCH₂N), 52.1 (CH₃-OOC-Ar), 29.0 (C7, C7'), 28.9 (C4, C4'), 24.1 (C6, C6'), 24.1 (C5, C5'). HRMS (ESI+) *m/z* (%) Calcd. for C₃₉H₄₈Cl₃N₄O₉ [M+H]⁺: 779.2733, found 779.2699 (100%).