Enantioselective synthesis of 2-substituted pyrrolidines *via* domino cross metathesis/intramolecular aza-Michael addition

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Electronic Supplementary Information

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1. General methods.

CH₂Cl₂ and THF were dried according to *Purification of Common Laboratory Chemicals*. Other reagents were used without further purification. Thin layer chromatography (TLC) was performed on EMD precoated plates (silica gel 60 F254, Art 5715), Optical rotations were recorded on a A212000-T APIV/IW. Column chromatography was performed on Silica Gel 60 (300–400 Mesh) using a forced flow of 0.5–1.0 bar. ¹H NMR (300 MHz), ¹³C NMR (75 MHz) were measured on a Bruker AVANCE III–300 spectrometer, ¹H NMR (400 MHz), ¹³C NMR (100MHz) were measured on a Bruker AVANCE III–400 spectrometer. Chemical shifts are expressed in parts per million (ppm) with respect to the residual solvent peak. Coupling constants are reported as Hertz (Hz), signal shapes and splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; Infrared (IR) spectra were recorded on a Nicolet 6700 spectrophotometer and are reported as wavenumber (cm¹).

 4^{1} were prepared according to published literature procedures. $5a-5g^{2}$, $5h^{3}$, $5l^{2}$ were prepared according to published literature procedures. 5i, 5j, 5k were commercially available from Acros Corp. 6^{4} , 7^{4} were prepared according to published literature procedures. $3a^{5}$, $3b-3d^{6}$ were prepared according to published literature procedures. The spectroscopic data of $1j^{5}$, $2j^{5}$, $2m^{4}$, $2n^{4}$ were published in the previous literature.

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2. General procedures and data for compounds



General procedure A : Preparation of N-protected amine 1

To a solution of *N*-protected amine **4** (0.5 mmol, 1.0 equiv) in CH_2Cl_2 (0.1M) under nitrogen atmosphere, the corresponding conjugated ketone **5** (1.5 mmol, 3.0 equiv) and Hoveyda-Grubbs 2nd generation catalyst (5 mol%) were added. The resulting solution was stirred for 12 h at room temperature and then, solvents were removed and the crude mixture purified by flash chromatography with hexanes / ethyl acetate as eluents to give the desired product **1**.

Data for Compounds Afforded by General Procedure A



According to general procedure A, **1a** was obtained from **4a** and **5a** as a foam solid in 79% yield. ¹H NMR (400 MHz, CDCl₃): $\delta = 7.52$ (dd, ¹J = 2.0 Hz, ²J = 8.4 Hz, 1H), 7.48 (d, J = 2.0 Hz, 1H), 6.99-6.92 (m, 1H), 6.87 (d, J = 15.2Hz, 1H), 6.83 (d, J = 8.4 Hz, 1H), 4.56 (br s, 1H), 3.88 (s, 6H), 3.11 (q, J = 6.4 Hz, 2H), 2.28 (q, J = 7.2Hz, 2H), 1.69-1.62 (m, 2H), 1.37 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 188.80$, 155.99, 153.19, 149.17, 147.37, 130.92, 125.84, 123.11, 110.80, 109.97, 79.25, 56.06, 56.01, 40.06, 29.96, 29.67, 28.77; FTIR (film): v_{max} 3378, 2925, 2854, 1699, 1663, 1558, 1497, 1454, 1249, 1137, 1027, 980, 774 cm⁻¹; HRMS (ESI) calcd for C₁₉H₂₇NO₅Na (M+Na)⁺: 372.1787, found: 372.1788.



According to general procedure A, **1b** was obtained from **4b** and **5a** as a colorless oil in 78% yield. ¹H NMR (400 MHz, CDCl₃): δ = 7.47 (d, *J* = 8.4 Hz, 1H), 7.44 (d, *J* = 1.6 Hz, 1H), 7.21-7.15 (m, 5H), 6.94-6.89 (m, 1H), 6.85 (d, J = 15.2 Hz, 1H), 6.74 (d, J = 8.0 Hz, 1H), 5.52 (t, J = 5.6 Hz, 1H), 4.98 (s, 2H), 3.778 (s, 3H), 3.775 (s, 3H), 3.12 (q, J = 6.4 Hz, 2H), 2.20 (q, J = 6.8 Hz, 2H), 1.64-1.57 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 188.56$, 156.58, 153.15, 149.04, 147.37, 136.70, 130.72, 128.39, 127.93, 127.87, 125.65, 123.12, 110.72, 110.05, 66.37, 55.91, 55.82, 40.42, 29.77, 28.45. FTIR (film): v_{max} 3369, 3063, 2936, 2613, 2536, 2031, 1957, 1698, 1596, 1267, 1022, 890, 762 cm⁻¹; HRMS (ESI) calcd for C₂₂H₂₅NO₅ Na (M+Na)⁺: 406.1630, found: 406.1632.



According to general procedure A, **1c** was obtained from **4b** and **5c** as a white solid in 75% yield : m.p. 69-70 °C. ¹H NMR (400 MHz, CDCl₃): $\delta = 7.82$ (d, J = 7.2 Hz, 2H), 7.46-7.42 (m, 1H), 7.36-7.32 (m, 2H), 7.24-7.16 (m, 5H), 6.96-6.89 (m, 1H), 6.80 (d, J = 15.6 Hz, 1H), 5.04 (br s, 1H), 4.99 (s, 2H), 3.14 (q, J = 6.4 Hz, 2H), 2.22 (q, J = 6.8 Hz, 2H), 1.66-1.58 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 190.70$, 156.54, 148.42, 137.82, 136.63, 132.77, 128.57, 128.54, 128.12, 128.09, 126.37, 66.64, 40.54, 29.95, 28.56; FTIR (film): v_{max} 3346, 2929, 1717, 1669, 1578, 1531, 1448, 1340, 1250, 1002, 846, 773, 696 cm⁻¹; HRMS (ESI) calcd for C₂₀H₂₁NO₃Na (M+Na)⁺: 346.1419, found: 346.1425.



According to general procedure A, **1d** was obtained from **4b** and **5d** as a brown oil in 68% yield. ¹H NMR (400 MHz, CDCl₃): $\delta = 7.94$ (d, J = 8.8 Hz, 2H), 7.36-7.31 (m, 5H), 7.03-6.93 (m, 1H), 6.94 (d, J = 8.8 Hz, 2H), 6.91 (d, J = 14.8 Hz, 1H), 5.10 (s, 2H), 4.83 (br s, 1H), 3.87 (s, 3H), 3.26 (q, J = 6.8 Hz, 2H), 2.34 (q, J = 6.8 Hz, 2H), 1.78-1.71 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta =$ 188.85, 163.39, 156.42, 147.08, 136.55, 130.86, 130.72, 128.55, 128.15, 126.12, 113.79, 66.72, 55.47, 40.58, 29.91, 28.67; FTIR (film): v_{max} 3368, 2955, 1697, 1599, 1509, 1414, 1357, 1309, 1259, 1214, 1171, 1113, 1029, 990, 827, 699 cm⁻¹; HRMS (ESI) calcd for C₂₁H₂₃NO₄Na (M+Na)⁺: 376.1525, found: 376.1529.



According to general procedure A, **1e** was obtained from **4b** and **5e** as a brown oil in 75% yield. ¹H NMR (400 MHz, CDCl₃): $\delta = 8.23$ (d, J = 8.8 Hz, 2H), 7.98 (d, J = 8.8 Hz, 2H), 7.28-7.21 (m, 5H), 7.06-6.98 (m, 1H), 6.82 (d, J = 15.2 Hz, 1H), 5.03 (s, 2H), 4.81 (br s, 1H), 3.20 (q, J = 6.4 Hz, 2H), 2.32 (q, J = 7.2 Hz, 2H), 1.73-1.62 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 189.11$, 156.47, 150.56, 150.06, 142.65, 136.46, 129.50, 128.57, 128.21, 128.11, 126.04, 123.79, 66.79, 40.41, 29.98, 29.69, 28.49; FTIR (film): v_{max} 3365, 2925, 2855, 1722, 1710, 1693, 1602, 1513, 1462, 1345, 1238, 1025, 854, 818, 736 cm⁻¹; HRMS (ESI) calcd for C₂₀H₂₀N₂O₅Na (M+Na)⁺: 391.1270, found: 391.1272.



According to general procedure A, **1f** was obtained from **4b** and **5f** as a brown solid in 64% yield. mp 89-90 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.78 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.27-7.22 (m, 5H), 6.97-6.92 (m, 1H), 6.79 (d, *J* = 15.2 Hz, 1H), 5.01 (s, 2H), 4.88 (br s, 1H), 3.17 (q, *J* = 6.8 Hz, 2H), 2.26 (q, *J* = 7.2 Hz, 2H), 1.67-1.62 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ = 189.28, 156.49, 148.84, 139.18, 136.53, 136.11, 129.98, 128.88, 128.55, 128.17, 128.12, 125.94, 66.73, 40.49, 29.70, 28.56; FTIR (film): v_{max} 3348, 2924, 2854, 1714, 1620, 1506, 1456, 1377, 1245, 1091, 1009, 812, 728, 699, 619, 575 cm⁻¹; HRMS (ESI) calcd for C₂₀H₂₃ClNO₄ (M+H₃O)⁺ : 376.1316, found: 376.1528.



According to general procedure A, **1g** was obtained from **4b** and **5g** as a white solid in 78% yield. m.p. 74-75 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.46 (d, *J* = 7.2 Hz, 1H), 7.36 (d, *J* = 1.2 Hz, 1H), 7.27-7.21 (m, 5H), 6.94-6.88 (m, 1H), 6.78 (d, J = 15.2 Hz, 1H), 6.76 (d, J = 8.0 Hz, 1H), 5.95 (s, 2H), 5.01 (s, 2H), 4.89 (br s, 1H), 3.16 (q, J = 6.4 Hz, 2H), 2.24 (q, J = 7.2 Hz, 2H), 1.68-1.61 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 188.39$, 156.48, 151.66, 148.23, 147.50, 136.58, 132.54, 128.54, 128.13, 125.96, 124.77, 108.44, 107.87, 101.83, 66.69, 40.53, 29.89, 28.61; FTIR (film): v max 3345, 3065, 3033, 2929, 1712, 1666, 1622, 1519, 1488, 1445, 1323, 1172, 1135, 980, 841, 797 cm⁻¹; HRMS (ESI) calcd for C₂₁H₂₁NO₅Na (M+Na)⁺: 390.1317, found: 390.1322.



According to general procedure A, **1h** was obtained from **4b** and **5h** as a brown oil in 62% yield. 1H NMR (300 MHz, CDCl3): $\delta = 7.62$ (t, J = 0.9 Hz, 1H), 7.39-7.28 (m, 5H), 7.26 (d, J = 6.0 Hz, 1H), 7.17-7.08 (m, 1H), 6.83 (d, J = 15.6 Hz, 1H), 6.57-6.55 (m, 1H), 5.10 (s, 2H), 4.83 (br s, 1H), 3.26 (q, J = 6.6 Hz, 2H), 2.35 (q, J = 7.5 Hz, 2H), 1.80-1.70 (m, 2H); ¹³C NMR (100MHz, CDCl₃): $\delta = 177.98$, 156.43, 153.27, 147.50, 146.59, 136.53, 128.55, 128.15, 127.87, 125.45, 117.71, 112.40, 66.73, 40.53, 30.23, 28.53; FTIR (film): v_{max} 3422, 3033, 2954, 2885, 1683, 1622, 1586, 1499, 1418, 1355, 1213, 1100, 1041, 991, 882, 699 cm⁻¹; HRMS (ESI) calcd for C₁₈H₁₉NO₄Na (M+Na)⁺ : 336.1212, found: 336.1210.



According to general procedure A, **1i** was obtained from **4b** and **5i** as a brown oil in 69% yield. ¹H NMR (400 MHz, CDCl₃): $\delta = 6.76-6.69$ (m, 1H), 6.02 (d, J = 16 Hz, 1H), 4.71 (br s, 1H), 3.11-3.06 (m, 2H), 2.23-2.19 (m, 2H), 2.17 (s, 3H), 1.64-1.57 (m, 2H), 1.37 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): $\delta = 198.49$, 155.97, 147.18, 131. 59, 79.15, 39.96, 28.58, 28.46, 28.35, 26.85; FTIR (film): v_{max} 3366, 2924, 2856, 2361, 1697, 1627, 1521, 1455, 1392, 1366, 1253, 1173, 1104, 980, 777, 613 cm⁻¹; HRMS (ESI) calcd for C₁₂H₂₁NO₃Na (M+Na)⁺: 250.1419, found: 250.1421.



According to general procedure A, **1j** was obtained from **4b** and **5i** as a brown oil in 89% yield. The spectroscopic data are in agreement with those previously reported in the literature ^[5].



According to general procedure A, **1k** was obtained from **4b** and **5k** as a brown oil in 85% yield. ¹H NMR (400 MHz, CDCl₃): δ = 7.30-7.20 (m, 5H), 6.74-6.68 (m, 1H), 6.03 (d, *J* = 16 Hz, 1H), 5.02 (s, 2H), 4.91 (br s, 1H), 3.13 (q, *J* = 6.4 Hz, 2H), 2.49-2.44 (m, 2H), 2.16 (q, *J* = 7.2 Hz, 2H), 1.63-1.56 (m, 2H), 1.00(t, *J* = 7.2 Hz, 3H); ¹³C NMR (100MHz, CDCl₃): δ =201.01, 156.50, 145.60, 136.58, 130.45, 128.51, 128.10, 128.06, 66.61, 40.46, 33.29, 29.67, 29.53, 28.48, 8.05; FTIR (film): *v* max 2924, 2855, 1710, 1589, 1462, 1452, 1378, 1110, 828, 731 cm⁻¹; HRMS (ESI) calcd for C₁₆H₂₁NO₃Na (M+Na)⁺: 298.1419, found: 298.1429.



According to general procedure A, **11** was obtained from **4b** and **51** as a brown oil in 68% yield. ¹H NMR (400 MHz, CDCl₃): δ = 7.24-7.15 (m, 7H), 7.10-7.06 (m, 3H), 6.71-6.64 (m, 1H), 6.00 (d, J = 15.6 Hz, 1H), 4.98 (s, 2H), 4.95 (br s, 1H), 3.08 (q, J = 6.4 Hz, 2H), 2.85-2.80 (m, 2H), 2.76-2.72 (m, 2H), 2.11 (q, J = 7.2 Hz, 2H), 1.57-1.50 (m, 2H); ¹³C NMR (100MHz, CDCl₃): δ = 199.44, 156.52, 146.23, 141.24, 136.60, 130.71, 128.57, 128.51, 128.41, 128.16, 128.12, 126.12, 66.68, 41.75, 40.46, 30.06, 29.59, 28.47; FTIR (film): v_{max} 3348, 3062, 3029, 2933, 1717, 1628, 1529, 1454, 1366, 1249, 1137, 1027, 980, 774, 699cm⁻¹; HRMS (ESI) calcd for C₂₂H₂₅NO₃Na (M+Na)⁺ : 374.1732, found: 374.1738.

General procedure B: Aza-Michael Addition



To a solution of *N*-protected amine **1** (0.5 mmol, 1.0 equiv) in CH_2Cl_2 (0.1M) was added the chiral Brønsted acid catalyst **3d** (10 mol %) under nitrogen atmosphere at -20°C. The reaction mixture was stirred at the same temperature. After the reaction was completed (as judged by TLC analysis), the crude reaction mixture was subjected to flash chromatography on silica gel by using mixtures of hexanes and ethyl acetate as eluents to afford the corresponding product **2**.

General procedure C: Domino Cross Metathesis/Aza-Michael Addition



To a solution of N-protected amine **4b** (0.5 mmol, 1.0 equiv) and the corresponding conjugated ketone **5** (1.5 mmol, 3.0 equiv) in CH₂Cl₂ (0.1M) under nitrogen atmosphere, the Hoveyda-Grubbs ^{2nd} generation catalyst (5mol %) and the chiral Br ønsted acid catalyst **3d** were sequentially added at 0 ^oC. The resulting solution was stirred at the same temperature. After the reaction was completed (as judged by TLC analysis), the crude reaction mixture was subjected to flash chromatography on silica gel by using mixtures of hexanes and ethyl acetate as eluents to afford the corresponding product **2**.

Data for Compounds Afforded by General Procedure B and C.



According to General Procedure B: **2a** was obtained from **1a** as a colorless oil in 93% yield and 94/6 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 90:10); $\lambda = 254$ nm, flow rate = 0.5 mL/min, t_R (minor) = 21.58 min, t_R(major) =

22.59 min; $[\alpha]^{27}{}_{D}$ +4.5 (*c* 0.4, CHCl₃, 94/6 er); ¹H NMR (400 MHz, CDCl₃): δ = 7.71 (d, *J* = 8 Hz, 1H), 7.61 (s, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 4.34-4.29 (m, 1H), 3.952 (s, 3H), 3.950 (s, 3H), 3.63 (d, *J* = 5.2Hz, 1H), 3.39-3.35 (m, 2H), 2.77-2.71 (m, 1H), 2.04-1.77(m, 4H), 1.47 (s, 9H); ¹³C NMR (100MHz, CDCl₃): δ = 197.59, 154.41, 153.34, 149.06, 130.18, 123.24, 110.33, 110.07, 79.39, 56.06, 56.02, 54.70, 46.46, 43.14, 31.11, 28.56, 23.23; IR (film): *v* max 3079, 2968, 2927, 1685, 1595, 1515, 1456, 1402, 1367, 1341, 1271, 1169, 1117, 1024, 909, 805 cm⁻¹; HRMS (ESI) calcd for C₁₉H₂₇NO₅Na (M+Na)⁺: 372.1787, found: 372.1785.



According to General Procedure B: **2b** was obtained from **1b** as a colorless oil in 90% yield and 98/2 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); $\lambda = 254$ nm, flow rate = 0.5 mL/min, t_R (minor) = 40.78 min, t_R(major) = 45.57 min; $[\alpha]^{26}_{D}$ -7.3 (*c* 4.8, CHCl₃, 98/2 er); ¹H NMR (400 MHz, CDCl₃): $\delta = 7.71$ (d, J = 7.6Hz, 1H), 7.55 (s, 1H), 7.35-7.29 (m, 5H), 6.92-6.90 (m, 1H), 5.09 (s, 2H), 4.92 (s, 1H), 4.02 (s, 1H), 3.96 (s, 3H), 3.93 (s, 3H), 3.33-3.24 (m, 2H), 3.15(s, 1H), 1.91-1.85 (m, 1H), 1.78-1.66 (m, 3H); ¹³C NMR (100MHz, CDCl₃): $\delta = 192.61$, 156.48, 154.11, 149.37, 136.54, 128.80, 128.54, 128.14, 128.09, 123.31, 110.27, 110.18, 66.69, 59.19, 56.90, 56.16, 56.07, 40.48, 29.06, 26.46; IR (film): v_{max} 3360, 3064, 2941, 1671, 1543, 1509, 1419, 1349, 1204, 1027, 1014, 889, 811 cm⁻¹; HRMS (ESI) calcd for C₂₂H₂₅NO₅K (M+K)⁺: 422.1370, found: 422.1580.



According to General Procedure B: **2c** was obtained from **1c** as a colorless oil in 91% yield and 95/5 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) = 15.93 min, t_R(major) = 12.93 min; [α]²⁵_D +13.3 (*c* 1.2, CHCl₃, 95/5 er);

According to General Procedure C: **2c** was obtained from **4b** and **5c** as a brown oil in 76% yield and 94/6 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); $\lambda = 254$ nm, flow rate = 0.5 mL/min, t_R (minor) = 32.67 min, t_R(major) = 37.36 min; ¹H NMR (400 MHz, CDCl₃): $\delta = 7.95$ (d, J = 7.2Hz, 1H), 7.74 (d, J = 7.2Hz, 1H), 7.57-7.44 (m, 2H), 7.38-7.31 (m, 6H), 5.06 (s, 2H), 4.29-4.27 (m, 1H), 3.78-3.74 (m, 1H, major rotamer) and 3.45-3.40 (m, 1H, minor rotamer), 3.39-3.32 (m, 2H), 2.75-2.68 (m, 1H), 2.01-1.94 (m, 1H), 1.85-1.67 (m, 3H); ¹³C NMR (100MHz, CDCl₃): $\delta = 198.91$ and 198.55, 153.74 and 153.58, 135.86 and 135.70, 135.60 and 135.54, 132.13, 127.58 and 127.45, 127.31 and 127.23, 127.11 and 126.90, 126.76, 65.97 and 65.59, 53.95 and 53.17, 45.75 and 45.42, 42.70 and 41.81, 30.16 and 29.32, 22.56 and 21.74; IR (film): v_{max} 3347, 3066, 2941, 1698, 1597, 1514, 1450, 1413, 1359, 1254, 1117, 1013, 913, 880 cm⁻¹; HRMS (ESI) calcd for C₂₀H₂₁NO₃Na (M+Na)⁺: 346.1419, found: 346.1414.



According to General Procedure B: **2d** was obtained from **1d** as a foam solid in 85% yield and 93/7 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) = 26.49 min, t_R(major) = 36.47 min; [α]²⁴_D -9.2 (*c* 0.5, CHCl₃, 93/7 er).

According to General Procedure C: **2d** was obtained from **4b** and **5d** as a colorless oil in 59% yield and 97/3 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) = 52.89 min, t_R(major) = 67.81 min. ¹H NMR (400 MHz, CDCl₃): δ = 8.06 (d, *J* = 8.4 Hz, 2H, major rotamer) and 7.81 (d, *J* = 8.4 Hz, 2H, minor rotamer), 7.38-7.31 (m, 5H), 6.93 (d, *J* = 8.4 Hz, 2H, major rotamer) and 6.75 (d, *J* = 8.8 Hz, 2H, minor rotamer), 5.16 (s, 2H), 4.36-4.34 (m, 1H), 3.85 (s, 3H, major rotamer) and 3.83(s, 3H, major rotamer), 3.80-3.78 (m, 1H, major rotamer) and 3.49-3.43 (m, 1H, minor rotamer), 3.49-3.43 (m, 2H), 2.75-2.69 (m, 1H), 2.09-2.02 (m, 1H), 1.98-1.83 (m, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 197.48 and 197.07, 163.57, 154.83 and 154.69, 136.92 and 136.67, 130.76 and 130.51, 129.91 and 129.70, 128.62 and 128.50, 128.42 and 128.09, 127.96 and 127.80, 113.78, 67.11 and 66.63, 55.47, 55.29 and 54.51, 46.81 and 46.47, 43.53 and 42.69, 30.99and 30.18, 23.59 and 22.75; IR (film): v_{max} 3589, 2924, 2851, 1703, 1597, 1560, 1508, 1458, 1259, 1102, 870 cm⁻¹; HRMS (ESI) calcd for C₂₁H₂₆NO₅ (M+H₃O)⁺: 372.1811, found: 372.1786.



According to General Procedure B: **2e** was obtained from **1e** as a colorless oil in 90% yield and 91/9 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) = 46.14 min, t_R(major) = 74.32 min; [α]²⁵_D -7.8 (*c* 1.3, CHCl₃, 91/9 er).

According to General Procedure C: 2e was obtained from 4b and 5e as a brown oil in 52% yield and 87/13 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) = 92.80 min, t_R(major) = 148.63 min. ¹H NMR (400 MHz, CDCl₃): $\delta = 8.22$ (d, J = 8.4 Hz, 2H, major rotamer) and 7.99 (d, J = 8Hz, 2H, minor rotamer), 8.14 (d, J = 8.4 Hz, 2H, major rotamer) and 7.85 (d, J = 8Hz, 2H, minor rotamer), 7.30-7.19 (m, 5H), 5.07 (s, 2H), 4.28-4.26 (m, 1H), 3.80-3.75 (m, 1H, major rotamer) and 3.50-3.47 (m, 1H, minor rotamer), 3.45-3.37 (m, 2H), 2.82-2.76 (m, 1H), 2.06-2.01 (m, 1H), 1.89-1.81 (m, 2H), 1.76-1.71 (m, 1H); ¹³C NMR (100MHz, CDCl₃): $\delta = 197.34$ (major rotamer) and 196.92 (minor rotamer), 154.92 (major rotamer) and 154.52 (minor rotamer), 150.37, 141.03 (major rotamer) and 140.65 (minor rotamer), 136.72 (major rotamer) and 136.36 (minor rotamer), 129.44 (major rotamer) and 129.18 (minor rotamer), 128.75 (major rotamer) and 128.52 (minor rotamer), 128.18 (minor rotamer) and 128.06 (major rotamer), 127.85, 123.87, 67.36 (minor rotamer) and 66.78 (major rotamer), 54.80 (major rotamer) and 54.05 (minor rotamer), 46.87 (minor rotamer) and 46.82 (major rotamer), 44.42 (minor rotamer) and 43.50 (major rotamer), 31.16 (minor rotamer) and 30.50 (major rotamer), 22.78 (major rotamer) and 22.68 (minor rotamer). IR (film): v max 3107, 2960, 2092, 1693, 1603, 1525, 1498, 1412, 1347, 1187, 1098, 856, 747cm⁻¹; HRMS (ESI) calcd for $C_{20}H_{20}N_2O_5Na (M+Na)^+$: 391.1270, found: 391.1267.



According to General Procedure B: **2f** was obtained from **1f** as a colorless oil in 87% yield and 95/5 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) = 18.44 min, t_R(major) = 13.95 min; $[\alpha]^{25}_{D}$ +23.2 (*c* 1.6, CHCl₃, 95/5 er).

According to General Procedure C: 2f was obtained from 4b and 5f as a brown oil in 60% yield and 94/6 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) = 30.19 min, t_R(major) = 40.73 min. ¹H NMR (400 MHz, CDCl₃): $\delta = 7.99$ (d, J = 8.4Hz, 2H, major rotamer) and 7.74 (d, J =8Hz, 2H, minor rotamer), 7.42 (d, J = 8.4 Hz, 2H, major rotamer) and 7.25 (d, J = 8 Hz, 2H, minor rotamer), 7.37-7.30 (m, 5H), 5.15 (s, 2H), 4.34-4.32 (m, 1H), 3.82-3.78 (m,1H, major rotamer) and 3.51-3.48 (m, 1H, minor rotamer), 3.49-3.42 (m, 2H), 2.80-2.74 (m, 1H), 2.10-2.05 (m, 1H), 1.93-1.77 (m, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 197.68 (major rotamer) and 197.28 (minor rotamer), 154.84 (major rotamer) and 154.59 (minor rotamer), 139.65, 136.83 (major rotamer) and 136.54 (minor rotamer), 135.05 (major rotamer) and 134.84 (minor rotamer), 129.85 (major rotamer) and 129.59 (minor rotamer), 128.95 (major rotamer) and 128.66 (minor rotamer), 128.51 (major rotamer) and 128.38 (minor rotamer), 128.24 (major rotamer) and 127.99 (minor rotamer), 127.82, 67.14 (major rotamer) and 66.70 (minor rotamer), 54.99 (major rotamer) and 54.19 (minor rotamer), 46.83 (major rotamer) and 46.49 (minor rotamer), 43.82 (major rotamer) and 42.92 (minor rotamer), 31.16 (minor rotamer) and 30.34 (major rotamer), 22.78 (major rotamer) and 22.69 (minor rotamer). IR (film): v max 3065, 3034, 2925, 1701, 1588, 1571, 1536, 1488, 1449, 1410, 1357, 1309, 1211, 1093, 1011, 992 cm⁻¹; HRMS (ESI) calcd for C₂₀H₂₀ClNO₃Na (M+Na)⁺: 380.1029, found: 380.1026.



According to General Procedure B: 2g was obtained from 1g as a colorless oil in 84% yield and 97/3

er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) = 18.76 min, t_R(major) = 22.02 min; [α]²⁵_D -6.4 (*c* 1.2, CHCl₃, 97/3 er).

According to General Procedure C: 2g was obtained from 4b and 5g as a pale white solid in 62% yield and 97/3 er at 0 °C and mp : 68-69°C. The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) =60.29 min, $t_R(major) = 74.32 \text{ min.}^1 \text{H NMR}$ (400 MHz, CDCl₃): $\delta = 7.63$ (d, J = 8.4 Hz, 1H, major rotamer), 7.42 (s, 1H), 7.33-7.18 (m, 5H), 6.77 (d, J = 8Hz, 1H, major rotamer) and 6.54 (d, J = 8Hz, 1H, minor rotamer), 5.94 (s, 2H), 5.074 (s, 2H, major rotamer) and 5.069 (s, 2H, minor rotamer), 4.29-4.23 (m, 1H), 3.70-3.66 (m, 1H, major rotamer) and 3.40-3.37 (m, 1H, minor rotamer), 3.40-3.33 (m, 2H), 2.66-2.60 (m, 1H), 2.00-1.93 (m, 1H), 1.85-1.72 (m, 3H). ¹³C NMR (100MHz, CDCl₃): $\delta = 195.90$ (major rotamer) and 195.51 (minor rotamers), 153.78 (major rotamer) and 153.61 (minor rotamers), 150.81, 147.19, 135.87 (major rotamer) and 135.63 (minor rotamers), 130.66 (major rotamer) and 130.54 (minor rotamers), 127.56 (major rotamer) and 127.46 (minor rotamers), 127.27 (minor rotamers) and 127.08 (major rotamer), 126.92 (minor rotamers) and 126.77 (major rotamer), 123.97 (major rotamer) and 123.67 (minor rotamers), 106.96 (minor rotamers) and 106.92 (major rotamer), 106.77 (major rotamer) and 106.72 (minor rotamers), 100.80, 65.99 (minor rotamers) and 65.61 (major rotamer), 54.23 (major rotamer) and 53.43 (minor rotamers), 45.78 (minor rotamers) and 45.43 (major rotamer), 42.53 (minor rotamers) and 41.70 (major rotamer), 30.09 (minor rotamers) and 29.21 (major rotamer), 22.56 (major rotamer) and 21.73 (minor rotamers). IR (film): v max 3506, 3066, 3032, 2924, 2785, 1671, 1604, 1505, 1489, 1437, 1355, 1282, 1258, 1187, 933, 905 cm⁻¹. HRMS (ESI) calcd for C₂₁H₂₁NO₅Na (M+Na)⁺: 390.1317, found: 390.1316.



According to General Procedure B: **2h** was obtained from **1h** as a colorless oil in 89% yield and 91/9 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 1.0 mL/min, t_R (minor) =15.05 min, t_R(major)

=21.19 min.

According to General Procedure C: **2h** was obtained from **4b** and **5h** as a brown oil in 52% yield and 92/8 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) =23.93 min, t_R(major) = 33.74 min; [α]²⁵_{D+}9.3 (*c* 0.6, CHCl₃, 92/8 er).

¹H NMR (400 MHz, CDCl₃): δ = 7.50 (s, 1H, major rotamer) and 7.41 (s, 1H, minor rotamer), , 7.32-7.19 (m, 5H), 6.90 (d, *J* = 2.4 Hz, 1H), 6.44 (s, 1H, major rotamer) and 6.33 (s, 1H, minor rotamer), 5.07 (s, 2H), 4.30-4.24 (m, 1H), 3.50 (dd, J = 2.4 & 14.4 Hz) and 3.22 (dd, J = 3.2 & 14.8 Hz), 3.41-3.34 (m, 2H), 2.68-2.62 (m, 1H), 2.02-1.95 (m,1H), 1.87-1.77 (m, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 187.40 (major rotamer) and 187.17 (minor rotamer), 154.77 (major rotamer) and 154.75 (minor rotamer), 154.65, 152.53, 146.65, 136.89 (major rotamer) and 136.61 (minor rotamer), 128.51 (major rotamer) and 128.30 (minor rotamer), 128.09 (major rotamer) and 127.96 (minor rotamer), 127.82, 118.35 (major rotamer) and 117.67 (minor rotamer), 112.25, 67.06 (minor rotamer) and 46.43 (major rotamer), 43.64 (minor rotamer) and 22.76 (minor rotamer), 31.92 (minor rotamer) and 31.15 (major rotamer), 23.58 (major rotamer) and 22.76 (minor rotamer); IR (film): ν max 3337, 3127, 3033, 2926, 1700, 1568, 1498, 1468, 1416, 1357, 1309, 1287, 1213, 1187, 1102, 1027 cm⁻¹; HRMS (ESI) calcd for C₁₈H₁₉NO₄Na (M+Na)⁺: 336.1212, found: 336.1210.



According to General Procedure B: **2i** was obtained from **1i** as a colorless oil in 90% yield and 92/8 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 16.91 min, t_R(major) = 15.38 min; $[\alpha]^{25}_{D}$ +38.4 (*c* 0.9, CHCl₃, 92/8 er) [lit. $[\alpha]^{28}_{D}$ -43.3 (c 0.1, CHCl₃) for S-enantiomer] The absolute configuration of **2i** was assigned as "R" by a comparison of its optical rotation with the literature value of the S-enantiomer. ¹H NMR (400 MHz, CDCl₃): δ = 4.07 (br s, 1H), 3.25 (m, 2H,), 3.07-3.00 (m, 1H, major rotamer) and 2.89-2.83 (m, 1H, minor rotamer), 2.37-2.24 (m, 1H), 2.08 (s,

3H), 2.03-1.95 (m, 1H), 1.80-1.71 (m, 2H), 1.60-1.54 (m, 1H), 1.39 (s, 9H); ¹³C NMR (100MHz, CDCl₃): δ = 206.57 (major rotamer) and 206.25 (minor rotamer), 153.36 (major rotamer) and 153.22 (minor rotamer), 78.53 (minor rotamer) and 78.24 (major rotamer), 52.45, 47.70 (minor rotamer) and 46.92 (major rotamer), 45.51 (major rotamer) and 45.38 (minor rotamer), 30.53 (minor rotamer) and 30.41 (major rotamer), 29.80 (major rotamer) and 29.52 (minor rotamer), 29.30 (minor rotamer) and 28.67 (major rotamer), 27.51 (minor rotamer) and 27.30 (major rotamer). IR (film) : v_{max} 3589, 2874, 2930, 2879, 1693, 1479, 1456, 1390, 1303, 1285, 1254, 1169, 1123, 1103, 1009, 951, 859 cm⁻¹; HRMS (ESI) calcd for C₁₂H₂₁NO₃Na (M+Na)⁺: 250.1419, found: 250.1414.



According to General Procedure B: **2j** was obtained from **1j** as a colorless oil in 97% yield and 98/2 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 30.58 min, t_R(major) = 38.02 min.

According to General Procedure C: **2j** was obtained from **4b** and **5j** as a brown oil in 74% yield and 95/5 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 28.69 min, t_R(major) = 34.62 min; $[\alpha]^{25}_{D+}37.1$ (*c* 0.18, CHCl₃, 95/5 er) [lit. ^[5] $[\alpha]^{28}_{D}$ +38.0 (*c* 0.5, CHCl₃)] The absolute configuration of **2i** was assigned as "R" by a comparison of its optical rotation with the literature value of the R-enantiomer. The spectroscopic data are in agreement with those previously reported in the literature^[5]. HRMS (ESI) calcd for C₁₅H₁₉NO₃Na (M+Na)⁺:284.1263, found: 284.1258.



According to General Procedure B: **2k** was obtained from **1k** as a colorless oil in 84% yield and 97/3 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 21.69 min, t_R(major) =

28.57 min.

According to General Procedure C: **2k** was obtained from **4b** and **5k** as a colorless oil in 76% yield and 97/3 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 21.79 min, t_R(major) = 28.41 min; $[\alpha]^{25}_{D+3}8.2$ (*c* 0.2, CHCl₃ 97/3 er). ¹H NMR (400 MHz, CDCl₃): δ = 7.32-7.27 (m, 5H), 5.11 (m, 2H), 4.24-4.19 (m, 1H), 3.48-3.42 (m, 2H), 3.14-3.10 (m, 1H, major rotamer) and 3.87-2.84 (m, 2H, minor rotamer), 2.44-2.38 (m, 2H), 2.30-2.28 (m, 1H), 2.13-2.04 (m, 1H), 1.84-1.78 (m, 2H), 1.69-1.62 (m, 1H), 1.05-0.88 (m, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 209.80 (major rotamer) and 209.59 (minor rotamer), 154.58, 136.94 (major rotamer) and 136.77 (minor rotamer), 128.44, 127.88 (major rotamer) and 127.75 (minor rotamer), 66.74 (minor rotamer) and 66.50 (major rotamer), 54.11 (major rotamer) and 53.36 (minor rotamer), 47.07 (minor rotamer) and 46.64 (major rotamer), 31.67 (minor rotamer) and 30.91 (major rotamer), 23.60 (major rotamer) and 22.81 (minor rotamer), 7.64; IR (film): ν_{max} 2935, 2087, 1701, 1498, 1450, 1414, 1358, 1283, 1213, 1185, 1101, 1028, 919 cm⁻¹; HRMS (ESI) calcd for C₁₆H₂₁NO₃Na (M+Na)⁺: 298.1419, found: 298.1424.



According to General Procedure B: **21** was obtained from **11** as a colorless oil in 87% yield and 97/3 er at -20 °C , The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 64.48 min, t_R(major) = 88.43 min.

According to General Procedure C: **21** was obtained from **4b** as a brown oil in 61% yield and 96/4 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =220 nm, flow rate = 0.5 mL/min, t_R (minor) = 65.36 min, t_R(major) = 89.77 min; [α]²⁵_D +10.7 (*c* 2.5, CHCl₃, 96/4 er). ¹H NMR (400 MHz, CDCl₃): δ = 7.25-7.01 (m, 10H), 5.01 (s, 2H), 4.14-4.08 (m, 1H), 3.30-3.28 (m, 2H), 3.06-3.02 (m, 1H, major rotamer), 2.82-2.63 (m, 4H), 2.53-2.51 (m, 1H), 2.32-2.56 (m, 1H), 1.99-1.94 (m, 2H), 1.74-1.50 (m, 1H); ¹³C NMR

(100MHz, CDCl₃): δ = 207.41 (major rotamer) and 207.22 (minor rotamer), 153.61, 139.99 (major rotamer) and 139.76 (minor rotamer), 135.89 (major rotamer) and 135.71 (minor rotamer), 127.45 (minor rotamer) and 127.30 (major rotamer), 126.90 (major rotamer) and 126.79 (minor rotamer), 125.04, 65.80 (minor rotamer) and 65.55 (major rotamer), 53.01 (major rotamer) and 52.24 (minor rotamer), 46.76 (minor rotamer) and 45.78 (major rotamer), 45.63 (minor rotamer) and 45.32 (major rotamer), 43.58, 30.56 (minor rotamer) and 29.84 (major rotamer), 28.62, 22.56 (major rotamer) and 21.75 (minor rotamer); IR (film): v_{max} 3062, 3029, 2955, 2879, 1698, 1604, 1497, 1454, 1413, 1357, 1336, 1305, 1212, 1185, 1106, 985, 769 cm⁻¹; HRMS (ESI) calcd for C₂₂H₂₅NO₃Na (M+Na)⁺: 374.1732, found: 374.1738.



According to General Procedure C: **2m** was obtained from **6** and **5j** as a brown oil in 61% yield and 94/6 er at 0 °C, The er values were determined by HPLC analysis using a Chiralpak OD-H column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) = 13.89 min, t_R(major) = 16.53 min; $[\alpha]^{25}_{D}$ +80.2 (*c* 0.4, CHCl₃, 94/6 er); [lit. ^[4] $[\alpha]^{25}_{D}$ +82.5 (*c* 1.0, CHCl₃)] The absolute configuration of **2m** was assigned as "R" by a comparison of its optical rotation with the literature value of the R-enantiomer for the same enantiomer. ¹H NMR (400 MHz, CDCl₃): 7.70-7.81 (m, 1H), 7.34-7.24 (m, 5H), 7.08-7.04 (m, 2H), 6.90-6.86 (m, 1H), 5.20 (s, 2H), 4.79-4.75 (m, 1H), 3.39-3.32 (m, 1H), 2.94 (s, 1H), 2.63-2.54 (m, 2H), 2.01 (s, 3H); The spectroscopic data are in agreement with those previously reported in the literature.^[4]



According to General Procedure C: 2n was obtained from 7 and 5j as a colorless oil in 64% yield and 97/3 er at 0 °C. The er values were determined by HPLC analysis using a Chiralpak OD-H

column (hexane/2-propanol 95:5); λ =254 nm, flow rate = 0.5 mL/min, t_R (minor) = 20.45 min, t_R(major) = 27.19 min; $[\alpha]^{25}_{D}$ +90.9 (c 0.44, CHCl_{3, 97/3} er); [lit. ^[4] $[\alpha]^{25}_{D}$ +90.6 (c 1.0, CHCl₃)], The absolute configuration of **2n** was assigned as "R" by a comparison of its optical rotation with the literature value of the R-enantiomer. ¹H NMR (400 MHz, CDCl₃): 7.32-7.14 (m, 9H), 5.43 (d, *J* = 3.2 Hz, 1H), 5.18-5.07 (m,2H), 4.77-4.59 (m, 2H), 3.24 (dd, *J* = 3.2 Hz & 17.2Hz, 1H, major rotamer) and 3.03 (dd, *J* = 2.8Hz & 16.4Hz, 1H, minor rotamer), 2.89-2.83 (m, 1H, major rotamer) and 2.77-2.70 (m, 1H, minor rotamer), 2.06 (s, 3H, major rotamer) and 1.93 (s, 3H, minor rotamer); The spectroscopic data are in agreement with those previously reported in the literature.^[4]

3. Copies of NMR Spectra

NMR Spectra for Compounds Afforded by General Procedure A.























NMR Spectra for Compounds Afforded by General Procedures B and C.

























200

150



50

0

PPM









S40







4. Copies of HPLC Traces



HPLC Spectra for Compounds Afforded by General Procedure B at -20 °C:

1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm									
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	21.341	3060859	78408	49.024	49.623				
2	22.785	3182743	79600	50.976	50.377				
Total		6243602	158008	100.000	100.000				

D:\data\yushouyun\data\-20 shuju\2a-G.lcd m٧ Det.A Ch1 22.593 500-OMe Boc 2a ÒМе 250-21.582 0 5 10 15 20 25 30 ό min

1 Det.A Ch1/254nm

Detector A	Detector A Ch1 254nm								
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	21.582	1935933	54653	6.184	7.331				
2	22.593	29367071	690887	93.816	92.669				
Total		31303004	745539	100.000	100.000				

PeakTable



1 Det.A Ch1/254nm

PeakTable

Detector A Ch1 254nm									
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	39.407	20121863	165457	49.950	56.677				
2	49.712	20162118	126474	50.050	43.323				
Total		40283981	291932	100.000	100.000				



PeakTable

Detector A Ch1 254nm									
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	40.784	1553966	20635	1.795	5.186				
2	45.573	85030989	377249	98.205	94.814				
Total		86584955	397885	100.000	100.000				



PeakTable

			-				
Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %		
1	13.002	23842327	810219	49.129	53.991		
2	15.748	24688193	690437	50.871	46.009		
Total		48530520	1500656	100.000	100.000		



		PeakTable					
Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %		
1	12.934	40358629	1243149	95.179	95.091		
2	15.930	2044261	64172	4.821	4.909		
Total	1	42402889	1307322	100.000	100.000		



PeakTable Detector A Ch1 254nm Area 57754186 Peak# Ret. Time Height Area % Height % 49.678 50.322 100.000 64.920 35.080 100.000 24.940 905041 1 58502478 489055 1394096 2 33.908 Total 116256663



	PeakTable				
Detector A	Ch1 254nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.494	592050	9588	6.893	12.234
2	36.473	7996537	68778	93.107	87.766
Total		8588587	78365	100.000	100.000



			PeakTable				
Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %		
1	45.647	58800659	619603	49.920	64.137		
2	74.132	58990006	346452	50.080	35.863		
Total		117790664	966056	100.000	100.000		



			PeakTable				
Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %		
	1 46.135	1171668	14923	9.036	14.709		
	2 74.319	11795656	86535	90.964	85.291		
Tot	al	12967324	101458	100.000	100.000		



PeakTable Detector A Ch1 254nm Ret. Time 13.215 Height % 72.839 27.161 Peak# Area Height Area % 80636141 3109491 49.165 17.328 83374822 1159472 50.835 2 Total 164010963 4268963 100.000 100.000



Detector A	Ch1 254nm		P	eakTable	
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.950	165934540	3553703	95.151	94.548
2	18.435	8455806	204902	4.849	5.452
Total		174390346	3758605	100.000	100.000



PeakTable

Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %		
1	14.992	6721299	251623	49.874	61.468		
2	21.634	6755244	157735	50.126	38.532		
Total		13476543	409359	100.000	100.000		



			Pea	akTable		
Detector A	Detector A Ch1 254nm					
Peak#	Ret. Time	Area	Height	Area %	Height %	
1	15.054	2079720	79425	8.862	14.746	
2	21.185	21386823	459199	91.138	85.254	
Total		23466543	538623	100.000	100.000	



1 Det.A Ch1/254nm

Detector A	Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %			
1	18.915	617213	17950	49.984	57.327			
2	23.873	617610	13362	50.016	42.673			
Total		1234823	31312	100.000	100.000			



PeakTable Detector A Ch1 254nm Ret. Time 18.759 22.019 Height % 6.731 93.269 Peak# Height Area % Area 1866368 54084327 56291 779982 3.336 96.664 1 2 55950694 Total 836273 100.000 100.000



1 Det.A Ch1/220nm

PeakTable

]	Detector A Ch1 220nm								
ſ	Peak#	Ret. Time	Area	Height	Area %	Height %			
	1	15.356	879459	21342	49.626	52.292			
ſ	2	16.552	892712	19471	50.374	47.708			
	Total		1772171	40813	100.000	100.000			



PeakTable Detector A Ch1 220nm Peak# Ret. Time Area Height Area % Height % 1370801 33112 92.138 7.862 15.379 91.985 16.908 2825 8.015 119447 2 35937 Total 1490248 100.000 100.000



PeakTable

Detector A Ch1 220nm								
Peak#	Ret. Time	Area	Height	Area %	Height %			
1	28.105	662507	16917	49.957	55.229			
2	36.727	663648	13714	50.043	44.771			
Total		1326155	30631	100.000	100.000			



PeakTable

Detector A	Detector A Ch1 220nm						
Peak#	Ret. Time	Area	Height	Area %	Height %		
1	30.578	770424	19752	2.063	4.441		
2	38.020	36576079	425009	97.937	95.559		
Total		37346503	444762	100.000	100.000		



PeakTable

Detector A	Detector A Ch1 220nm								
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	21.377	12302242	288060	49.530	49.794				
2	28.774	12535479	290446	50.470	50.206				
Total		24837721	578506	100.000	100.000				



				Pe	eakTable		
Det	Detector A Ch1 220nm						
I	Peak#	Ret. Time	Area	Height	Area %	Height %	
	1	21.689	1372557	51749	3.195	8.222	
	2	28.567	41585948	577665	96.805	91.778	
	Total		42958505	629413	100.000	100.000	

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1 Det.A Ch1/220nm

PeakTable

Detector A	Detector A Ch1 220nm								
Peak#	Ret. Time	Area	Height	Area %	Height %				
1	62.607	28838164	239493	50.533	65.138				
2	93.085	28229773	128177	49.467	34.862				
Total		57067937	367670	100.000	100.000				

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1 Det.A Ch1/220nm

PeakTable

			FC.	akiaute	
Detector A	Ch1 220nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	64.479	3303094	38766	3.196	11.823
2	88.431	100062953	289122	96.804	88.177
Total		103366047	327888	100.000	100.000

HPLC Spectra for Compounds Afforded by General Procedure C at 0 °C:



PeakTable

Detector A Ch1 220nm							
Pea	k#	Ret. Time	Area	Height	Area %	Height %	
	1	29.060	19454591	354047	49.022	55.342	
	2	37.259	20230782	285702	50.978	44.658	
,	Total		39685373	639748	100.000	100.000	



1 Det.A Ch1/220nm

			Peak	Table		
Detector A Ch1 220nm						
Peak#	Ret. Time	Area	Height	Area %	Height %	
1	28.687	2071195	59820	4.567	10.953	
2	34.618	43275656	486317	95.433	89.047	
Total		45346851	546137	100.000	100.000	



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578506

Detector A	Ch1 220nm		Peak	Fable	
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21 377	12302242	288060	49 530	49 7

12535479

24837721



49.794 50.206

100.000

50.470

100.000

1 Det.A Ch1/220nm

2 Total 28.774

			Pea	akTable	
Detector A	Ch1 220nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.794	1357741	47561	3.315	7.707
2	28.411	39605115	569550	96.685	92.293
Total		40962856	617111	100.000	100.000



Detector A	Ch1 220nm		Pe	akTable	
Peak#	Ret. Time	Area	Height	Area %	Height %
1	62.607	28838164	239493	50.533	65.138
2	93.085	28229773	128177	49.467	34.862
Total		57067937	367670	100.000	100.000



1 Det.A Ch1/220nm

				PeakTal	ble	
Detector A Ch1 220nm						
	Peak#	Ret. Time	Area	Height	Area %	Height %
	1	65.356	3470246	36909	3.894	12.324
	2	89.766	85650882	262576	96.106	87.676
	Total		89121129	299485	100.000	100.000



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			Р	eakTable	
Detector A	Ch1 254nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.221	67849025	1008073	49.575	64.721
2	36.901	69013400	549483	50.425	35.279
Total		136862425	1557556	100.000	100.000

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1 Det.A Ch1/254nm

PeakTable Detector A Ch1 254nm Area % 5.700 Peak# Height Ret. Time Area Height % 1492329 9.021 32.672 38395 24687637 26179966 387243 425638 2 37.361 94.300 90.979 Total 100.000 100.000



	Detector A	Ch1 254nm		Peak	cTable	
	Peak#	Ret. Time	Area	Height	Area %	Height %
1	1	53.310	98787670	793595	49.771	66.458
1	2	73.611	99695038	400531	50.229	33.542
	Total		198482708	1194126	100.000	100.000



PeakTable Detector A Ch1 254nm Area % 2.799 97.201 Peak# Ret. Time Height Height % Area 52.893 67.807 5133892 178310386 68951 707071 8.885 91.115 Tota 183444278 776022 100.000 100.000



PeakTable

Detector A Ch1 254nm						
Peak#	Ret. Time	Area	Height	Area %	Height %	
1	90.513	41469339	286451	49.952	63.594	
2	149.403	41548821	163984	50.048	36.406	
Total		83018159	450435	100.000	100.000	



PeakTable

			T VIII	10010	
Detector A	Ch1 254nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	92.803	14088630	104033	13.223	24.826
2	148.630	92456657	315007	86.777	75.174
Total		106545287	419040	100.000	100.000



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1 Det.A Ch1/254nm

			Pe	eakTable	
Detector A	Ch1 254nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	61.081	14707291	134684	50.225	60.018
2	78.358	14575721	89720	49.775	39.982
Tota	1	29283012	224404	100.000	100.000

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1 Det.A Ch1/254nm

Detector A Ch1 254nm Height % 5.241 94.759 Ret. Time 60.290 Peak# Area 375644 Area % Height 4733 3.009 1 74.323 85578 12106660 96.991 2 Total 12482304 90311 100.000 100.000

PeakTable



PeakTable

				-	currit	
Detector A Ch1 254nm						
	Peak#	Ret. Time	Area	Height	Area %	Height %
	1	22.492	117016865	1757034	49.485	62.111
ſ	2	32.327	119450499	1071827	50.515	37.889
	Total		236467363	2828861	100.000	100.000

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1 Det.A Ch1/254nm

 PeakTable

 Detector A Ch1 254nm
 Area
 Height
 Area %
 Height %

 1
 23.932
 2061320
 47915
 7.899
 13.166

 2
 33.741
 24034214
 316008
 92.101
 86.834

 Total
 26095533
 363923
 100.000
 100.000



т	Dataatar A	Ch1 254nm		Pea	kTable	
ſ	Peak#	Ret. Time	Area	Height	Area %	Height %
ſ	1	29.780	67687414	1595413	49.862	66.684
Γ	2	41.702	68060795	797093	50.138	33.316
	Total		135748209	2392506	100.000	100.000



			Peak	Table	
Detector A	Ch1 254nm				
Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.192	14056037	360238	5.942	14.456
2	40.733	222488238	2131708	94.058	85.544
Total		236544275	2491945	100.000	100.000

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				Pea	kTable	
]	Detector A	Ch1 254nm				
	Peak#	Ret. Time	Area	Height	Area %	Height %
ſ	1	13.148	6021802	231507	49.887	54.653
ſ	2	16.839	6049021	192086	50.113	45.347
ſ	Total		12070823	423593	100.000	100.000



		PeakTable						
Detector A	Detector A Ch1 254nm							
Peak#	Ret. Time	Area	Height	Area %	Height %			
1	13.891	5297099	245459	5.647	12.325			
2	16.534	88513008	1746090	94.353	87.675			
Total		93810107	1991549	100.000	100.000			



			PeakTable					
Detector A Ch1 254nm								
Peak#	Ret. Time	Area	Height	Area %	Height %			
1	18.282	3699986	43790	49.893	45.598			
2	27.416	3715813	52245	50.107	54.402			
Total		7415799	96035	100.000	100.000			



PeakTable Detector A Ch1 254nm Height % 5.026 94.974 Peak# Ret. Time Area 179288 Height Area % 20.445 27.188 2.619 97.381 4804 90776 95580 6667250 6846538

Total

S65

100.000

100.000