Revised Supporting Information

**Palladium Mediated Intramolecular Aryl
Amination on Furanose Derivatives : An
Expedient Approach to the Synthesis of Chiral
Benzoxazocine Derivatives and Tricyclic
Nucleosides**

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Experimental Section

General Melting points are uncorrected. ^1H (300 MHz, 600 MHz) and ^{13}C (75 MHz, 150 MHz) NMR spectra were recorded using CDCl_3 as solvent and TMS as internal standard. Mass spectra were recorded under electron impact at 70 eV. Elemental analyses were carried out with a C, H, N analyzer. Specific rotations were measured at 589 nm. TLC was performed on pre-coated plates (0.25 mm, silica gel 60 F₂₅₄). Organic extracts were dried over anhydrous sodium sulphate. Solvents were distilled and dried immediately prior to use. Column chromatography and flash chromatography were carried out using commercial-grade silica gel (60-120 mesh or 230-400 mesh). PS, EA stand for petroleum spirit (60-80 °C) and ethyl acetate.

Procedure for the synthesis of **1f**.

To a magnetically stirred solution of 1,2:5,6-di-*O*-isopropylidene glucofuranoside (260 mg, 1 mmol) and 2-iodobenzyl bromide (356 mg, 1.2 mmol) in CH_2Cl_2 (20 mL) was added Bu_4NBr (50 mg) followed by aqueous NaOH (50%, 20 mL) at 0 °C. The reaction mixture was stirred at room temperature for 12 h and extracted with CH_2Cl_2 (4×25 mL). The combined organic layer was washed with water (3×25 mL), dried, and evaporated to afford a syrup which on column chromatography over silica gel yielded the corresponding Iodobenzyl derivative **1f**.

(3a*R*,5*R*,6*S*,6a*R*)-6-(2-Bromo-benzyloxy)-5-(2,2-dimethyl-[1,3]dioxolan-4-yl)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxole (1f): Thick pale yellow oil; yield 70% (eluent PS-EA 13:1); $[\alpha]_{\text{D}}^{25} -21.2$ (*c* 1.0, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz): δ 1.33 (s, 3H), 1.35 (s, 3H), 1.43 (s, 3H), 1.51 (s, 3H), 4.01 (dd-like, 1H), 4.09-4.18 (m, 3H),

4.39 (dd, 1H, $J = 12.8, 6.9$ Hz,), 4.58-4.74 (m, 3H), 5.92 (d, 1H, $J = 3.2$ Hz), 6.97-7.83 (m, 4H, ArH); ^{13}C NMR (CDCl_3 , 75 MHz): δ 25.4, 26.2, 26.7, 26.8, 67.3, 71.5, 72.4, 81.2, 82.0, 82.4, 97.5, 105.2, 108.9, 111.8, 128.1, 128.7, 129.1, 129.3, 139.8; ESIMS, m/z : 499 (MNa^+); Anal. Calcd for $\text{C}_{19}\text{H}_{25}\text{IO}_6$: C, 47.91; H, 5.29 Found: C, 47.67; H, 5.30.

General procedure for the synthesis of amine compounds

The appropriate bromobenzyl derivative **1a-f** (2 mmol) was stirred overnight with 70% aqueous HOAc (v/v, 50 mL) at room temperature (monitored by TLC till disappearance of starting material). Removal of HOAc on a rotary evaporator under reduced pressure (temp < 40 °C) using dry toluene (4×25 mL), afforded the intermediate diol as a viscous syrup. A solution of the intermediate diol in minimum volume of methanol was cooled to 0 °C and treated with aqueous NaIO_4 (513 mg, 2.4 mmol, dissolved in 5 mL of water) slowly with stirring (45 min). The reaction mixture was evaporated under reduced pressure and the residual syrupy mass was extracted with CHCl_3 (4×25 mL). The combined organic layer was washed with water (3×25 mL), dried and evaporated to afford the crude aldehyde. This aldehyde (1 mmol) was dissolved in dry CH_2Cl_2 (25 mL), treated with activated molecular sieves (4°A) and amines (1.2 mmol) at 0 °C and then stirred at room temperature for 12 h under N_2 atmosphere. Dry MeOH (10 mL) was added to the reaction mixture and NaBH_4 (75.6 mg, 2 mmol) was added to it (in small portions) over a period of 1 h at 0 °C, and the stirring was continued for another 2 hours at room temperature. The solvent was evaporated under reduced pressure and the residue was extracted with CH_2Cl_2 (4×25 mL). The combined organic layer was washed with

water (3×25 mL) and dried. Evaporation of the solvent followed by chromatography on silica gel afforded the amines **2a-f** as colorless oils.

(3a*R*,5*R*,6*S*,6a*R*)-Benzyl-[6-(2-bromo-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-amine (2a). Thick oil; yield 65% (eluent PS-EA, 3:1); $[\alpha]_{\text{D}}^{25}$ -26.77 (*c* 1.8, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.33 (s, 3H), 1.49 (s, 3H), 1.84 (brs, 1H), 2.94 (dd, *J* = 12.2, 5.5 Hz, 1H), 3.03 (dd, *J* = 12.2, 7.2 Hz, 1H), 3.82 (d-like, 2H), 3.98 (d, 1H, *J* = 3.0 Hz), 4.35-4.40 (m, 1H), 4.54 (d, 1H, *J* = 12.6 Hz), 4.67 (d, 1H, *J* = 3.8 Hz), 4.73 (d, 1H, *J* = 12.6 Hz), 5.94 (d, 1H, *J* = 3.7 Hz), 7.13-7.54 (m, 9H); ¹³C NMR (CDCl₃, 75 MHz): δ 26.3, 26.7, 47.5, 54.2, 71.2, 79.7, 82.1, 82.7, 104.9, 111.6, 122.7, 126.9, 127.5, 2×128.1, 2×128.3, 2×129.3, 132.6, 136.8, 140.0; IR ν_{max} (liquid film) 2930, 1571, 1450, 1376, 1213, 1079, 1024 cm⁻¹; ESIMS *m/z* 448, 450 (MH⁺ for Br⁷⁹, Br⁸¹); Anal. Calcd for C₂₂H₂₆BrNO₄: C, 58.94; H, 5.85; N, 3.12. Found: C, 58.83; H, 5.72; N, 3.05.

(3a*R*,5*R*,6*S*,6a*R*)-[6-(2-Bromo-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-isopropyl-amine (2b). Thick oil; yield 75% (eluent PS-EA, 3:1); $[\alpha]_{\text{D}}^{25}$ -34.90 (*c* 2.0, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.03 (d-like, 6H), 1.33 (s, 3H), 1.49 (s, 3H), 2.79-2.87 (m, 1H), 2.91-2.95 (m, 2H), 3.98 (d, 1H, *J* = 2.6 Hz), 4.34 (br s, 1H), 4.56 (d, 1H, *J* = 12.6 Hz), 4.69 (d, 1H, *J* = 3.6 Hz), 4.76 (d, 1H, *J* = 12.6 Hz), 5.95 (d, 1H, *J* = 3.4 Hz), 7.15-7.56 (m, 4H); ¹³C NMR (CDCl₃, 150 MHz): δ 22.6, 23.0, 26.3, 26.7, 45.7, 48.9, 71.7, 80.0, 81.8, 82.3, 104.9, 111.6, 125.9, 127.8, 128.0, 128.7, 130.4, 137.4; ESIMS *m/z* 400, 402 (MH⁺ for Br⁷⁹, Br⁸¹); Anal. Calcd for C₁₈H₂₆BrNO₄: C, 54.01; H, 6.55; N, 3.50. Found: C, 53.90; H, 6.35; N, 3.25.

(3a*R*,5*R*,6*S*,6a*R*)-Benzyl-[6-(2-bromo-4,5-dimethoxy-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-amine (2c). Thick oil; yield 70% (eluent PS-EA, 2:1); $[\alpha]_{\text{D}}^{25}$ -20.35 (c 1.7, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz): δ 1.33 (s, 3H), 1.49 (s, 3H), 1.93 (brs, 1H), 2.91 (dd-like, 1H), 3.02 (dd-like, 1H), 3.79 (s, 3H), 3.81 (m, 2H), 3.85 (s, 3H), 3.96 (d, 1H, $J = 2.9$ Hz), 4.36-4.37 (m, 1H), 4.48 (d, 1H, $J = 12.1$ Hz), 4.67 (d-like, 2H), 5.94 (d, 1H, $J = 3.6$ Hz), 6.86 (s, 1H), 6.99 (s, 1H), 7.21-7.27 (m, 5H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 26.7, 27.2, 48.0, 54.6, 56.4, 56.6, 71.5, 80.1, 82.6, 82.9, 105.3, 112.0, 112.7, 113.6, 115.8, 127.3, 2 $\times$ 128.4, 2 $\times$ 128.8, 129.1, 140.4, 148.8, 149.6; IR ν_{max} (liquid film) 2933, 1505, 1457, 1262, 1213, 1162, 1077 cm^{-1} ; ESIMS m/z 508, 510 (MH^+ for Br^{79} , Br^{81}); Anal. Calcd for $\text{C}_{24}\text{H}_{30}\text{BrNO}_6$: C, 56.70; H, 5.95; N, 2.76 Found: C, 56.51; H, 5.78; N, 2.59.

(3a*R*,5*R*,6*S*,6a*R*)-Benzo[1,3]dioxol-5-ylmethyl-[6-(2-bromo-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-amine (2d). Thick pale yellow oil; yield 80% (eluent PS-EA, 3:1); $[\alpha]_{\text{D}}^{25}$ -47.99 (c 2.0, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz): δ 1.33 (s, 3H), 1.49 (s, 3H), 2.90 (dd, 1H, $J = 12.2, 5.5$ Hz), 3.02 (dd-like, 1H), 3.71 (d-like, 2H), 3.97 (d, 1H, $J = 3.0$ Hz), 4.32-4.37 (m, 1H), 4.53 (d, 1H, $J = 12.6$ Hz), 4.67 (d, 1H, $J = 3.7$ Hz), 4.74 (d, 1H, $J = 12.6$ Hz), 5.91 (s, 2H), 5.93 (d-like, 1H), 6.72-6.85 (m, 3H), 7.13-7.55 (m, 4H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 26.2, 26.7, 47.3, 53.9, 71.1, 79.7, 82.0, 82.6, 100.8, 104.8, 107.9, 108.6, 111.5, 121.2, 122.7, 127.4, 2 $\times$ 129.2, 132.5, 134.1, 136.7, 146.4, 147.6; IR ν_{max} (liquid film) 2929, 1488, 1443, 1246, 1079, 1035 cm^{-1} ; ESIMS m/z 492, 494 (MH^+ for Br^{79} , Br^{81}); Anal. Calcd for $\text{C}_{23}\text{H}_{26}\text{BrNO}_6$: C, 56.11; H, 5.32; N, 2.84. Found: C, 56.23; H, 5.05; N, 2.59.

(3a*R*,5*R*,6*S*,6a*R*)-Benzo[1,3]dioxol-5-ylmethyl-[6-(2-bromo-5-methoxy-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-amine (2e). Thick pale yellow oil; yield 85% (eluent PS-EA, 3:1); $[\alpha]_{\text{D}}^{25}$ -26.57 (c 2.0, CHCl_3); ^1H NMR (CDCl_3 , 300 MHz): δ 1.33 (s, 3H), 1.49 (s, 3H), 1.64 (brs, 1H), 2.91 (dd, 1H, $J = 12.1, 5.4$ Hz), 3.01 (dd-like, 1H), 3.71 (s, 2H), 3.76 (s, 3H), 3.97 (d, 1H, $J = 2.9$ Hz), 4.32-4.37 (m, 1H), 4.49 (d, 1H, $J = 12.9$ Hz), 4.68 (d-like, 2H), 5.91 (s, 2H), 5.94 (d, 1H, $J = 3.6$ Hz), 6.69-6.71 (m, 3H), 6.80-7.42 (m, 3H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 26.7, 27.1, 47.7, 54.4, 55.8, 71.5, 80.2, 82.5, 83.1, 101.2, 105.3, 108.4, 109.1, 112.0, 113.1, 115.1, 115.2, 121.6, 133.5, 134.5, 138.2, 146.8, 148.0, 159.4; ESIMS m/z 522, 524 (MH^+ for Br^{79} , Br^{81}); Anal. Calcd for $\text{C}_{24}\text{H}_{28}\text{BrNO}_7$: C, 56.18; H, 5.40; N, 2.68. Found: C, 56.40; H, 5.25; N, 2.78.

(3a*R*,5*R*,6*S*,6a*R*)-Benzyl-[6-(2-iodo-benzyloxy)-2,2-dimethyl-tetrahydro-furo[2,3-*d*][1,3]dioxol-5-ylmethyl]-amine (2f). Thick oil; yield 67% (eluent PS-EA, 3:1); $[\alpha]_{\text{D}}^{25}$ -26.20 (c 1.7, CHCl_3); ^1H NMR (CDCl_3 , 600 MHz): δ 1.32 (s, 3H), 1.49 (s, 3H), 2.04 (brs, 1H), 2.95-2.96 (m, 1H), 3.01-3.05 (m, 1H), 3.81 (d-like, 2H), 3.98 (d, 1H, $J = 3.6$ Hz), 4.35-4.38 (m, 1H), 4.46 (d, 1H, $J = 12.0$ Hz), 4.66 (d, 1H, $J = 12.6$ Hz), 4.69 (d, 1H, $J = 3.6$ Hz), 5.95 (d, 1H, $J = 3.6$ Hz), 6.99-7.81 (m, 9H); ^{13}C NMR (CDCl_3 , 150 MHz): δ 26.3, 26.7, 47.6, 54.2, 71.2, 79.7, 82.1, 82.6, 104.9, 111.6, 122.8, 126.9, 127.9, 2 $\times$ 128.3, 128.5, 128.9, 129.3, 132.6, 139.2, 139.7, 140.0; IR, ν_{max} (KBr) 2927, 1679, 1452, 1377, 1213, 1078 cm^{-1} ; ESIMS m/z 496 (MH^+); Anal. Calcd for $\text{C}_{22}\text{H}_{26}\text{INO}_4$: C, 53.34; H, 5.29; N, 2.83. Found: C, 53.20; H, 5.30; N, 2.58.

Procedure for cycloamination of sugaramines 2b-f.

Using procedure similar to the General Procedure for Cycloamination reaction of **2a**.

(2R,3R,3aS,11aR)-10-Isopropyl-2,3-isopropylidenedioxy-3,3a,5,10,11,11a-hexahydro-2H-furo[3,2-c][1,5]benzoxazocine (3b). Pale yellow oil; yield 74% (eluent PS-EA, 5:1); $[\alpha]_{\text{D}}^{25} +62.39$ (*c* 3.0, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.02 (d, 3H, *J* = 5.9 Hz), 1.30 (brs, 9H), 2.87-2.95 (m, 1H), 3.40 (dd, 1H, *J* = 14.5, 5.3 Hz), 3.62 (dd-like, 1H), 4.48-4.55 (m, 3H), 4.61 (d, 1H, *J* = 3.5 Hz), 4.83 (d, 1H, *J* = 12.1 Hz), 5.85 (d, 1H, *J* = 3.6 Hz), 6.95-7.36 (m, 4H); ¹³C NMR (CDCl₃, 75 MHz): δ 23.0, 23.6, 26.7, 27.4, 51.6, 56.4, 77.6, 82.5, 85.8, 89.2, 105.4, 111.7, 121.9, 123.4, 129.5, 131.3, 134.9, 155.2; IR ν_{max} (liquid film) 2925, 1490, 1382, 1217, 1078, 1008 cm⁻¹; ESIMS: *m/z* 320 (MH⁺), 342 (MNa⁺); Anal. Calcd for C₁₈H₂₅NO₄: C, 67.69; H, 7.89; N, 4.39. Found: C, 67.89; H, 7.62; N, 4.21.

(2R,3R,3aS,11aR)-7,8-Dimethoxy-10-benzyl-2,3-isopropylidenedioxy-3,3a,5,10,11,11a-hexahydro-2H-furo[3,2-c][1,5]benzoxazocine (3c). Pale yellow oil; yield 68% (eluent PS-EA, 5:1); $[\alpha]_{\text{D}}^{25} +66.03$ (*c* 3.5, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.26 (s, 3H), 1.30 (s, 3H), 3.06 (dd-like, 1H), 3.26 (dd, *J* = 13.8, 5.2 Hz, 1H), 3.81 (s, 3H), 3.85 (brs, 4H), 4.20 (d, 1H, *J* = 12.9), 4.38 (d-like, 2H), 4.57 (d-like, 2H), 4.81 (d, 1H, *J* = 12.7 Hz), 5.78 (d, 1H, *J* = 3.4 Hz), 6.68 (s, 1H), 6.77 (s, 1H), 7.23-7.39 (m, 5H); ¹³C NMR (CDCl₃, 75 MHz): δ 26.9, 27.3, 56.4, 56.6, 59.5, 60.6, 78.0, 80.4, 85.8, 90.2, 104.9, 105.5, 111.7, 114.6, 126.1, 127.8, 2×128.9, 2×129.3, 139.7, 144.8, 148.9, 149.5; IR ν_{max} (liquid film) 2931, 1512, 1217, 1078, 1018 cm⁻¹; ESIMS: *m/z* 428

(MH⁺), 450 (MNa⁺); Anal. Calcd for C₂₄H₂₉NO₆: C, 67.43; H, 6.84; N, 3.28. Found: C, 67.70; H, 6.58; N, 3.22.

(2*R*,3*R*,3*aS*,11*aR*)-10-(2-Benzo[1,3]dioxol-5-yl-ethyl)-2,3-isopropylidenedioxy-3,3*a*,5,10,11,11*a*-hexahydro-2*H*-furo[3,2-*c*][1,5]benzoxazocine (3*d*). Sticky liquid; yield 77% (eluent PS-EA, 7:1); [α]_D²⁵ +121.10 (*c* 2.0, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.28 (s, 3H), 1.35 (s, 3H), 3.12 (dd-like, 1H), 3.24 (dd, *J* = 14.1, 5.2 Hz, 1H), 3.90-3.93 (m, 1H), 4.15 (d, 1H, *J* = 13.0 Hz), 4.35 (d, 1H, *J* = 13.5 Hz), 4.38 (d, 1H, *J* = 3.0 Hz), 4.57 (d-like, 2H), 4.91 (d, 1H, *J* = 12.9 Hz), 5.80 (d, 1H, *J* = 3.5 Hz), 5.91 (s, 2H), 6.73-7.26 (m, 7H); ¹³CNMR (CDCl₃, 75 MHz): δ 26.5, 26.9, 58.4, 59.2, 77.9, 79.8, 85.2, 89.5, 100.9, 105.1, 108.1, 109.1, 111.3, 119.2, 121.9, 122.7, 129.1, 130.8, 133.2, 133.3, 146.8, 147.7, 154.6; IR $\nu_{\max}$ (liquid film) 2931, 1596, 1492, 1248, 1083, 1008 cm⁻¹; ESIMS: *m/z* 412 (MH⁺), 434 (MNa⁺); Anal. Calcd for C₂₃H₂₅NO₆: C, 67.14; H, 6.12; N, 3.40. Found: C, 67.00; H, 6.01; N, 3.23.

(2*R*,3*R*,3*aS*,11*aR*)-7-Methoxy-10-(2-benzo[1,3]dioxol-5-yl-ethyl)-2,3-isopropylidenedioxy-3,3*a*,5,10,11,11*a*-hexahydro-2*H*-furo[3,2-*c*][1,5]benzoxazocine (3*e*). Sticky liquid; yield 70% (eluent PS-EA, 7:1); [α]_D²⁵ +82.58 (*c* 1.5, CHCl₃); ¹H NMR (CDCl₃, 300 MHz): δ 1.28 (s, 3H), 1.35 (s, 3H), 2.99 (dd-like, 1H), 3.19 (dd, 1H, *J* = 13.9, 5.5 Hz), 3.74 (s, 3H), 3.87-3.94 (m, 1H), 4.08 (d, 1H, *J* = 12.8 Hz), 4.26 (d, 1H, *J* = 12.8 Hz), 4.40 (d, 1H, *J* = 3.3 Hz), 4.56 (d-like, 2H), 4.82 (d, 1H, *J* = 12.8 Hz), 5.79 (d, 1H, *J* = 3.7 Hz), 5.90 (s, 2H), 6.71-7.25 (m, 6H); ¹³C NMR (CDCl₃, 75 MHz): δ 26.5, 26.8, 55.4, 58.7, 59.9, 78.1, 80.0, 85.2, 90.2, 100.9, 105.2, 108.0, 109.2, 111.3, 113.4, 116.4, 120.3, 122.0, 133.4, 135.3, 146.8, 147.7, 148.0, 155.2; IR $\nu_{\max}$ (liquid film) 2929,

1595, 1496, 1444, 1247, 1080, cm^{-1} ; ESIMS: m/z 442 (MH^+), 464 (MNa^+); Anal. Calcd for $\text{C}_{24}\text{H}_{27}\text{NO}_7$: C, 65.29; H, 6.16; N, 3.17. Found: C, 65.00; H, 6.32; N, 3.30.

(2*R*,3*R*,3*aS*,11*aR*)-Aceticacid-2,3-diacetoxy-10-benzyl-3,3*a*,5,10,11,11*a*-hexahydro-2*H*-1,4-dioxo-10-aza-benzo[*a*]cyclopenta[*e*]cycloocten (5). Cyclized compound **3a** (734 mg, 2 mmol) was dissolved in acetonitrile- H_2O (3:1) containing 4% H_2SO_4 (v/v) and stirred at room temperature for 12 h. The acidic solution was neutralized with solid NaHCO_3 at 0 $^\circ\text{C}$, filtered and the filtrate was evaporated in vacuum. The residue was extracted with ethylacetate (6 $\times$ 25 mL), the combined organic layer was dried and concentrated under vacuum. The colorless oil was dissolved in dry pyridine (5 mL), treated with Ac_2O (0.5 mL) and stirred at room temperature for 12 h. Pyridine was evaporated from the reaction mixture under reduced pressure and the product was extracted with CHCl_3 (3 $\times$ 25 mL). The combined organic layer was washed with cold aqueous HCl (1% , 2 $\times$ 30 mL), water (3 $\times$ 50 mL), dried (Na_2SO_4) and evaporated under reduced pressure. The crude mass was purified by silica gel flash chromatography to afford diacetate **5** (660 mg) as a colorless oil; yield 90% (eluent PS-EA, 3:2); ^1H NMR (CDCl_3 , 300 MHz): δ 2.01 (s, 3H), 2.10 (s, 3H), 3.13-3.18 (m, 1H), 3.45 (dd, 1H, J = 14.0, 11.1 Hz), 4.10-4.22 (m, 1H), 4.28 (d, 1H, J = 13.8 Hz), 4.49 (d-like, 1H), 4.56 (d, 1H, J = 12.0 Hz), 4.77 (d, 1H, J = 12.2 Hz), 4.83 (d-like, 1H), 4.95 (d, 1H, J = 12.9 Hz), 6.24 (d, 1H, J = 4.3 Hz), 7.13-7.30 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz): δ 20.4, 20.8, 58.1, 58.7, 74.5, 77.9, 78.1, 84.7, 92.9, 98.7, 119.9, 122.7, 127.0, 2 $\times$ 128.3, 128.5, 129.1, 130.7, 133.4, 138.8, 153.2, 169.3, 169.8; IR ν_{max} (liquid film) 1752, 1598, 1373, 1218, 1078, 1011 cm^{-1} ; ESIMS: m/z 434 (MNa^+); Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{NO}_6$: C, 67.14; H, 6.12; N, 3.40. Found: C, 67.00; H, 6.01; N, 3.25.

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0.7843

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3.9479

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7.55278

7.52641

7.39180

7.37110

7.32313

7.32026

7.29871

7.26153

7.18956

7.18405

7.16304

7.13867

6.95521

6.80040

6.76089

6.75102

6.74502

6.72480

5.94552

5.93974

5.93304

5.91917

5.91536

4.76025

4.71809

4.68334

4.67089

4.55618

4.51394

4.37613

4.36482

4.34755

4.33462

4.32384

3.97973

3.96946

3.71915

3.71342

3.68995

3.03319

3.00873

2.99254

2.96855

2.93346

2.91498

2.89293

2.87428

1.61033

1.49527

1.46449

1.42645

1.33087

1.26910

1.25655

0.97699

0.95608

0.88322

0.86014

0.00001

APR-PA-AM

1H in CDCl3

12.9.05

0.00001

APN-MBP-A-M

1H in CDCl3

5.10.05

[illegible]

- 139.798
- 129.281
- 129.090
- 128.754
- 128.145
- 111.775
- 109.938
- 106.215
- 87.505
- 82.407
- 82.010
- 81.232
- 77.424
- 75.999
- 75.575
- 72.375
- 71.528
- 67.344
- 26.783
- 26.708
- 26.215
- 25.383

APV-18-Ar 130 11 (000)3 19.9.05

— 0.00

[illegible]

APN-10D-AM 13C CPD in CDCl3

ARN-A210-F 13C CDCl3 18.10.2005

3b

18.10.2005

27.10.2005
10/10

13C NMR
15.05

ppm

155.178
148.052
147.660
146.787

135.268
133.396

122.047
120.283
116.401
113.409
111.289
109.206
108.030
106.161
100.883

80.175
85.234
79.966
78.085
77.419
76.995
76.572

59.863
58.723
55.442

26.848
26.545

A-T-MB

13C 1H CDCl3

18.10.05

ABN-DOL-F 13C CPD in CDCl3

Chemical Shifts (ppm): 176.76, 176.10, 154.52, 138.76, 134.73, 130.60, 129.37, 126.10, 126.60, 127.59, 123.75, 120.11, 81.04, 77.32, 77.24, 77.03, 76.82, 71.99, 61.76, 59.84, 59.18, 20.90, 20.70, 0.00.

Chemical Shifts (ppm): 176.76, 176.10, 154.52, 138.76, 134.73, 130.60, 129.37, 126.10, 126.60, 127.59, 123.75, 120.11, 81.04, 77.32, 77.24, 77.03, 76.82, 71.99, 61.76, 59.84, 59.18, 20.90, 20.70, 0.00.

Chemical Shifts (ppm): 176.76, 176.10, 154.52, 138.76, 134.73, 130.60, 129.37, 126.10, 126.60, 127.59, 123.75, 120.11, 81.04, 77.32, 77.24, 77.03, 76.82, 71.99, 61.76, 59.84, 59.18, 20.90, 20.70, 0.00.

ADN - 49010 - F
H-11 6014 M. 6510
22 8.05

12. P. 2

400-MHz
 1H NMR IN CDCl₃
 25 °C

6

AN-4294.F
 4H-COSY IN CDCl3
 02.0.95