Supporting Information

Construction of β -Haloenamides via Direct Copper-Promoted Coupling of Lactams with 2-Chloro and 2-Bromo Vinyl iodides

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General Methods.

All non-aqueous reactions were carried out under a positive pressure of dry argon. Air and moisture sensitive liquid reagents were added via a dry syringe or cannula. ^1H and ^{13}C NMR spectra were recorded on Bruker DPX-300, CDPX-300, AMX-360 or DRX-400 MHz spectrometers. Flash chromatography was performed using EM Science silica gel 60 (230-400 mesh). Analytical and preparative thin layer chromatography (TLC) were performed on EM Science silica gel 60 PF_{254} . THF was dried over and distilled from sodium/benzophenone ketyl. CH_2Cl_2 was distilled from CaH_2 .

Experimental Details.

1-Bromo-2-iodovinylbenzene (11). To a stirred mixture of copper (II) bromide (1.3 g, 9.8 mmol) and iodine (2.5 g, 9.8 mmol) in dry acetonitrile (20 mL) at $0\text{ }^\circ\text{C}$ was added phenylacetylene (0.22 mL, 2.0 mmol) and the resulting mixture was stirred at $0\text{ }^\circ\text{C}$ for 1 h. Saturated aqueous sodium thiosulfate (75 mL) and EtOAc (100 mL) were added. The organic layer was washed with brine and dried over MgSO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography on silica gel (1:1 hexanes:EtOAc) to give (*E*)-1-bromo-2-iodovinylbenzene (**11**) as a yellow oil (600 mg, 100%). ^1H NMR (360 MHz, CDCl_3) δ 7.37-7.35 (m, 2H), 7.29-7.25 (m, 4H), 6.89 (s, 1H); ^{13}C NMR and DEPT-135 (90 MHz, CDCl_3) δ 139.8, 129.8, 129.5, 128.8, 122.5, 75.7; LRGC/MS-EI m/z (% relative intensity) 229 ($\text{MH}^+ - \text{Br}$, 40), 308 (MH^+ , 20).

1-(2-Chloro-2-phenylvinyl)-pyrrolidin-2-one (12). A resealable Schlenk tube was charged with copper (I) iodide (36 mg, 0.19 mmol), cesium carbonate (470 mg, 1.4 mmol) and 2-pyrrolidinone (**10**, 100 mg, 1.2 mmol) evacuated and filled with argon. *N,N'*-Dimethylethylenediamine (40 μL , 0.38 mmol), 1-chloro-2-iodovinylbenzene (**7**, 300 mg, 0.96 mmol) and dry THF (1 mL) were added under argon. The Schlenk tube was sealed, heated at $70\text{ }^\circ\text{C}$ for 40 h and cooled to rt. The resultant mixture was filtered through a pad of Celite eluting with EtOAc. EtOAc (25 mL) and water (25 mL) were added to the filtrate. The organic layer was washed with brine and dried over MgSO_4 . The solvent was removed under reduced pressure and the residue was purified by flash chromatography on silica gel (1:1 hexanes:EtOAc) to give 1-(2-chloro-2-phenylvinyl)-pyrrolidin-2-one (**12**) as a white solid (195 mg, 92%). IR (film) 3424, 1701 cm^{-1} ; ^1H

NMR (300 MHz, CDCl₃) δ 7.27 (bs, 5H), 7.14 (s, 1H), 2.88 (t, J = 6.9 Hz, 2H), 2.28 (t, J = 7.9 Hz, 2H), 1.79 (q, J = 7.4 Hz, 2H); ¹³C NMR and DEPT-135 (75 MHz, CDCl₃) δ 173.9, 135.7, 128.7, 128.3, 127.3, 122.3, 118.4, 46.9, 29.3, 17.8; LRMS-APCI m/z (% relative intensity) 222 (MH⁺, 100), 186 (40); HRMS-APCI: [M+H]⁺ calcd for C₁₂H₁₃ClNO, 222.0680; found, 222.0698.

1-(2-Bromo-2-phenylvinyl)-pyrrolidin-2-one (13). A resealable Schlenk tube was charged with copper (I) iodide (11 mg, 0.06 mmol), cesium carbonate (550 mg, 1.7 mmol) and 2-pyrrolidinone (**10**, 110 mg, 1.3 mmol), evacuated and filled with argon. *N,N'*-Dimethylethylenediamine (12 μ L, 0.13 mmol), 1-bromo-2-iodovinylbenzene (**11**, 340 mg, 1.1 mmol) and dry THF (1 mL) were added under argon. The Schlenk tube was sealed, heated at 70 °C overnight and cooled to rt. The resulting mixture was filtered through a pad of Celite eluting with EtOAc. EtOAc (25 mL) and water (25 mL) were added to the filtrate. The organic layer was washed with brine and dried over MgSO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography on silica gel (2:1 hexanes:EtOAc) to give 1-(2-bromo-2-phenylvinyl)-pyrrolidin-2-one (**13**, 61 mg, 21%). IR (film) 3409, 2951, 1731, 1667 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.32-7.25 (m, 5H), 7.22 (s, 1H), 2.86 (t, J = 6.9 Hz, 2H), 2.29 (t, J = 7.8 Hz, 2H), 1.79 (q, J = 7.0 Hz, 2H); ¹³C NMR and DEPT-135 (75 MHz, CDCl₃) δ 174.8, 141.7, 130.2, 129.9, 128.3, 125.9, 107.8, 48.0, 30.3, 18.9; LRMS-APCI m/z (% relative intensity) 266 (MH⁺, 100); HRMS-APCI: [M+H]⁺ calcd for C₁₂H₁₃BrNO, 266.0175; found, 266.0164.

Preparation of β -Bromoamide 15. A resealable Schlenk tube was charged with copper (I) iodide (1.7 mg, 0.009 mmol), cesium carbonate (88 mg, 0.27 mmol) and *N*-Boc- β -lactam **14** (60 mg, 0.21 mmol), evacuated and filled with argon. *N,N'*-Dimethylethylenediamine (2 μ L, 0.018 mmol), 1-bromo-2-iodovinylbenzene (**11**, 55 mg, 0.18 mmol) and dry THF (5 mL) were added under argon. The Schlenk tube was sealed,

heated at 50 °C overnight and cooled to rt. The resultant mixture was filtered through a pad of Celite eluting with EtOAc. EtOAc (25 mL) and water (25 mL) were added to the filtrate. The organic layer was washed with brine and dried over MgSO₄. The solvent was removed under reduced pressure and the residue was purified by flash chromatography on silica gel (1:1 hexanes:EtOAc) to give β -bromoamide **15** (32 mg, 39%). ¹H NMR (360 MHz, CDCl₃) δ 7.48-7.43 (m, 1H), 7.15-7.09 (m, 2H), 7.02-6.82 (m, 3H), 6.98 (s, 1H), 6.89-6.84 (m, 1H), 6.75-6.73 (m, 1H), 6.68-6.64 (m, 1H), 3.39-3.32 (m, 1H), 3.07-2.96 (m, 1H), 1.58 (s, 9H); LRMS-APCI *m/z* (% relative intensity) 369 (MH⁺ -Boc, 50).

Synthesis of Chloroamide 17. A resealable Schlenk tube charged with CuI (0.22 mg, 0.0012 mmol), Cs₂CO₃ (8 mg, 0.024 mmol), and β -lactam **16** (7 mg, 0.012 mmol) was evacuated and refilled with argon. *N,N'*-Dimethylethylenediamine (0.25 μ L, 0.0024 mmol), (1-chloro-2-iodovinyl)-benzene (**7**, 3 μ L, 0.019 mmol) and 0.5 mL of THF were added under argon. The Schlenk tube was sealed, heated at 70 °C for 1 d and cooled to rt. The reaction mixture was filtered through a short plug of silica gel eluting with EtOAc. The filtrate was concentrated and the residue was purified by preparative TLC (20% EtOAc/hexanes) to yield β -chloroamide **17** [4.1 mg, 43%, 57% based on recovered β -lactam **16** (2 mg)] as a yellow oil. IR (film) 3056, 2954, 1789, 1747 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.23-7.17 (m, 1H), 7.10-7.02 (m, 4H), 6.96 (s, 1H), 6.87 (s, 1H), 5.02 (d, *J* = 11.1 Hz, 1H), 4.38 (d, *J* = 11.1 Hz, 1H), 3.49, 3.30 (ABq, *J* = 14.9 Hz, 2H), 3.46-3.41 (m, 2H), 0.94-0.81 (m, 2H), -0.037 (s, 9H). HRMS-AP⁺: [M+H]⁺ calcd for C₂₄H₂₄ClBr₃N₂O₃SiNa, 710.8693; found, 710.8743.

Synthesis of β -Chloroamide 21. A resealable Schlenk tube charged with CuI (0.3 mg, 0.002 mmol), Cs₂CO₃ (21 mg, 0.064 mmol), and β -lactam **18** (17 mg, 0.032 mmol) was evacuated and refilled with argon. *N,N'*-Dimethylethylenediamine (0.3 μ L, 0.003 mmol), vinyl iodide **20** (12 mg, 0.032 mmol) and 1 mL of THF were added under argon. The Schlenk tube was sealed, heated at 50 °C overnight and cooled to rt. The reaction mixture was filtered through a short plug of silica gel eluting with EtOAc. The filtrate was concentrated and the residue was purified by preparative TLC

(hexanes/EtOAc, 1:3) to yield β -chloroenamide **21** (5 mg, 23 %) as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.43 (s, 1H), 7.34-7.25 (m, 6H), 7.07 (s, 1H), 7.02 (s, 1H), 6.63 (s, 1H), 5.17 (ABq, $J = 10.6, 16.7$ Hz, 2H), 4.43 (s, 2H), 3.36 (ABq, $J = 15.0, 58.4$ Hz, 2H); HRMS-ES(+): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{16}\text{N}_4\text{O}_3\text{NaClBr}_3$, 690.8359; found, 690.8346.

Synthesis of β -Chloroenamide 22. A resealable Schlenk tube charged with CuI (0.5 mg, 0.002 mmol), Cs_2CO_3 (31 mg, 0.096 mmol), and β -lactam **19** (21 mg, 0.048 mmol) was evacuated and refilled with argon. *N,N'*-Dimethylethylenediamine (0.5 μL , 0.005 mmol), vinyl iodide **20** (20 mg, 0.053 mmol) and 0.5 mL of THF were added under argon. The Schlenk tube was sealed, heated at 50 $^\circ\text{C}$ for 21 h and cooled to rt. The reaction mixture was filtered through a short plug of silica gel eluting with EtOAc. The filtrate was concentrated and the residue was purified by flash column chromatography (50-80% EtOAc/hexanes) to yield β -chloroenamide **22** (30 mg, 91 %) as a pale yellow solid. IR (film) 3056, 2926, 1778, 1732 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.43-7.27 (m, 7H), 7.10 (s, 1H), 6.52 (s, 1H), 5.23 (s, 2H), 4.45 (s, 2H), 3.45 (ABq, $J = 14.9, 34.0$ Hz, 2H), 3.23 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 173.2, 163.4, 145.1, 136.53, 136.46, 135.8, 128.7, 128.4, 128.1, 127.3, 124.5, 122.6, 121.4, 120.0, 118.7, 117.4, 112.6, 75.1, 70.4, 64.3, 47.3, 26.8; HRMS-ES(+): $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{18}\text{N}_4\text{O}_3\text{NaClBr}_3$, 704.8515; found, 704.8499.

Synthesis of β -Chloroenamide 25. A resealable Schlenk tube charged with CuI (0.6 mg, 0.003 mmol), Cs_2CO_3 (19 mg, 0.058 mmol), and β -lactam **23** (19 mg, 0.035 mmol) was evacuated and refilled with argon. *N,N'*-Dimethylethylenediamine (0.6 μL , 0.006 mmol), vinyl iodide **24** (17 mg, 0.035 mmol) and 0.5 mL of THF were added under argon. The Schlenk tube was sealed, heated at 65 $^\circ\text{C}$ for 10 h, at 70 $^\circ\text{C}$ for 22 h, and cooled to rt. The reaction mixture was filtered through a short plug of silica gel eluting with EtOAc. The filtrate was concentrated and the residue was purified by flash column chromatography (30-40% EtOAc/hexanes) to yield β -chloroenamide **25** [14 mg, 45%, 67% based on recovered vinyl iodide **24** (6 mg)] as a pale yellow solid. IR (film) 2962,

2924, 2853, 1789, 1731 cm^{-1} ; ^1H NMR (400 MHz, CD_2Cl_2) δ 7.40-7.23 (m, 11H), 7.14 (s, 1H), 6.81 (s, 1H), 5.19 (d, $J = 11.3$ Hz, 1H), 5.04 (s, 2H), 4.89 (d, $J = 11.3$ Hz, 1H), 4.58 (d, $J = 12.2$ Hz, 1H), 4.47 (s, 2H), 4.45s, 2H), 4.45 (d, $J = 12.2$ Hz, 1H), 3.60 (s, 3H), 3.35 (d, $J = 15.0$ Hz, 1H), 3.13 (d, $J = 15.0$ Hz, 1H), 1.71 (s, 3H), 1.64 (s, 3H); HRMS (APCI+) calcd for $\text{C}_{36}\text{H}_{33}\text{Br}_3\text{N}_4\text{O}_6$ (MH^+) 888.9633, found 888.9602.

Coupling of 2-Bromoimidazole 26 with 2-Pyrrolidinone (10). A resealable Schlenk tube charged with CuI (0.6 mg, 0.003 mmol), Cs_2CO_3 (40 mg, 0.123 mmol), and 2-pyrrolidinone (**10**, 5 μL , 0.068 mmol) was evacuated and refilled with argon. *N, N'*-Dimethylethylenediamine (0.7 μL , 0.006 mmol), vinyl iodide **26** (34 mg, 0.061 mmol) and 1.5 mL of THF were added under argon. The Schlenk tube was sealed, heated at 50 $^\circ\text{C}$ for 45 h and cooled to rt. The reaction mixture was filtered through a short plug of silica gel eluting with EtOAc. The filtrate was concentrated and the residue was purified by flash column chromatography (50-100% EtOAc/hexanes) to yield vinyl iodide **27** (16 mg, 47 %) as a colorless oil and bis-lactam **28** (3 mg, 10%). Major Product **27**: IR (film) 2924, 2360, 1729 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.27 (m, 5H), 7.17 (s, 1H), 5.30, 5.17 (ABq, $J = 11.0$ Hz, 2H), 4.50, 4.45 (ABq, $J = 11.8$ Hz, 2H), 3.94-3.88 (m, 1H), 3.78-3.70 (m, 1H), 3.68 (s, 3H), 2.44 (t, $J = 7.7$ Hz, 2H), 2.13-2.05 (m, 2H), 1.63 (s, 3H), 1.62 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 176.0, 175.0, 141.7, 139.6, 136.7, 128.5, 128.1, 127.6, 125.6, 122.6, 86.6, 73.8, 70.9, 52.1, 49.6, 44.3, 31.0, 25.4, 25.1, 18.8; LRMS (APCI+) calcd for $\text{C}_{22}\text{H}_{26}\text{ClIN}_3\text{O}_4$ (MH^+) 558.1, found 558.1. Minor Product **28**: IR (film) 2982, 2359, 1716 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.52 (s, 1H), 7.35-7.31 (m, 4H), 5.31, 5.15 (ABq, $J = 11.0$ Hz, 2H), 4.50-4.48 (m, 2H), 4.10-3.97 (m, 1H), 3.80-3.74 (m, 1H), 3.66 (s, 3H), 3.30-3.20 (m, 1H), 3.10-3.00 (m, 1H), 2.49-2.43 (m, 3H), 2.34-2.28 (m, 3H), 2.17-2.12 (m, 4H), 1.97-1.87 (m, 4H), 1.60 (s, 6H); LRMS (APCI+) calcd for $\text{C}_{22}\text{H}_{26}\text{ClIN}_3\text{O}_4$ (MH^+) 432.2, found 432.2.

5-Oxopyrrolidinone-2-carboxylic Acid 2-Chloro-3-iodoallyl Ester (31). To a stirred solution of 2-pyrrolidinone-5-carboxylic acid (**29**, 160 mg, 1.25 mmol) in dry CH_2Cl_2 (25 mL) at rt was added DMAP (31 mg, 0.25 mmol) followed by DCC (1.9 mL, 1.9 mmol) and 2-chloro-3-iodoprop-2-en-1-ol (**30**, 110 mg, 0.51 mmol), and the resulting solution was stirred at rt for 2 d. The mixture was then filtered through a pad of silica gel (EtOAc) and the solvent was removed under reduced pressure. The residue was purified by flash column chromatography on silica gel (EtOAc) to give 5-oxopyrrolidinone-2-carboxylic acid 2-chloro-3-iodoallyl ester (**31**, 140 mg, 84%). IR (film) 3350, 1748, 1697

cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.00 (bs, 1H), 6.66 (s, 1H), 4.91 (q, *J* = 12.9, 24.3 Hz, 2H), 4.30-4.26 (m, 1H), 2.46-2.24 (m, 4H); ¹³C NMR and DEPT-135 (75 MHz, CDCl₃) δ 176.5, 169.7, 129.1, 76.5, 65.4, 53.6, 27.4, 23.1; LRMS-APCI *m/z* (% relative intensity) 330 (MH⁺, 100); HRMS-APCI: [M+H]⁺ calcd for C₈H₁₀ClINO₃, 329.9389; found, 329.9361.

4-Chloro-9,9a-dihydro-3*H*,8*H*-pyrrolo[2,1-*c*]oxazepine-1,7-dione (32). A resealable Schlenk tube charged with copper (I) iodide (3 mg, 0.014 mmol), cesium carbonate (46 mg, 0.14 mmol) and 5-oxopyrrolidinone-2-carboxylic acid 2-chloro-3-iodoallyl ester (**31**, 31 mg, 0.09 mmol) was evacuated and filled with argon. *N,N'*-Dimethylethylenediamine (4 μL, 0.03 mmol) and dry THF (2 mL) were added under argon. The Schlenk tube was sealed, heated at 70 °C overnight and cooled to rt. The resulting bright blue mixture was filtered through a pad of silica gel (EtOAc). The solvent was removed under reduced pressure and the residue was purified by preparative TLC (EtOAc) to give 4-chloro-9,9a-dihydro-3*H*,8*H*-pyrrolo[2,1-*c*]oxazepine-1,7-dione (**32**, 8 mg, 46%). ¹H NMR (300 MHz, CDCl₃) δ 6.26 (s, 1H), 5.11 (d, *J* = 12.9 Hz, 1H), 4.45 (d, *J* = 12.9 Hz, 1H), 4.38-4.33 (m, 1H), 2.60-2.27 (m, 4H); ¹³C NMR and DEPT-135 (75 MHz, CDCl₃) δ 174.0, 170.0, 126.3, 125.9, 64.0, 61.2, 29.5, 22.4; HRMS-AP⁺: [M+H]⁺ calcd for C₈H₉ClNO₃, 202.0271; found, 202.0282.

11

11

77.911
77.558
77.205
75.700

129.863
129.518
128.991
128.834
122.546
139.856

Current Data Parameters
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PROCNO 1

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NS 48
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RG 32768
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TE 300.0 K
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D1 2.00000000 sec
CPOPRG walz16
P31 106.00 usec
SA 30 dB
D11 0.03000000 sec
S2 30 dB
P1 7.90 usec
SFO1 90.565695 MHz
NUCLEUS 13C

F2 - Processing parameters
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GB 0
PC 1.40

1D NMR plot parameters
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PFGM 4.98108 ppm/cm
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 PROCNO 1

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 DS 2
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 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
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 SF01 299.8718518 MHz

F2 - Processing parameters
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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F2 -121.96 Hz
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12

13

Current Data Parameters
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 SWH 7462.567 Hz
 FIDRES 0.113872 Hz
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 RG 2048
 DW 67.000 usec
 DE 95.71 usec
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 P1 1.0000000 sec
 SF01 360.1361620 MHz
 NUCLEUS 1H

F2 - Processing parameters
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 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 F1 2825.97 Hz
 F2P -0.475 ppm
 F2 -171.41 Hz
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CHANNEL f1 =====

Current Data Parameters
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 PROCNO 2

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 SWH 6172.839 Hz
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 DE 6.00 usec
 TE 300.0 K
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 P1 11.70 usec
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F2 - Processing parameters
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 GB 0
 PC 1.00

1D NMR plot parameters
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 F1 2399.96 Hz
 F2P 2.500 ppm
 F2 749.67 Hz
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Current Data Parameters
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 RG 80.6
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 DE 5.00 usec
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 D1 1.00000000 sec

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F2 - Processing parameters
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 LB 0.30 Hz
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1D NMR plot parameters
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MeO₂C 28

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 RG: 512
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 TE: 300.3 K
 D1: 1.00000000 sec

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 SF01: 300.1318534 MHz

F2 - Processing parameters
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 GB: 0
 PC: 1.00

1D NMR plot parameters
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 HZCM: 127.55925 Hz/cm

Current Data Parameters
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 PROCNO: 1
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 TO : 499330
 DECIB : 0.00000000
 SOLVENT : CDCl3
 NS : 11
 DS : 1
 SWH : 18795.992 Hz
 FIDRES : 0.288619 Hz
 AQ : 1.7743076 sec
 RG : 724.1
 DW : 28.000 usec
 DE : 6.00 usec
 TE : 300.0 K
 D1 : 2.00000000 sec
 D11 : 0.03000000 sec
 D12 : 0.00020000 sec
 ===== CHANNEL f1 =====
 NUC1 : 13C
 P1 : 5.40 usec
 PL1 : -8.00 dB
 SF01 : 75.4106357 MHz
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 NUC2 : 1H
 P2 : 115.00 usec
 PL2 : 0.00 dB
 PL12 : 20.00 dB
 PL13 : 20.00 dB
 SF02 : 299.8711995 MHz
 F2 - Processing parameters
 SI : 32768
 SF : 75.4025030 MHz
 WDW : EM
 SSB : 0
 LB : 1.00 Hz
 GB : 0
 PC : 1.40
 1D NMR plot parameters
 CX : 20.00 cm
 FIP : 215.000 ppm
 F1 : 16211.54 Hz
 F2 : -5.000 ppm
 F2 : -377.01 Hz
 PPRCM : 11.00000 ppm/cm
 HZCM : 829.42755 Hz/cm